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INVESTIGATION OF MECHANICAL, ELECTRICAL, AND THERMAL PROPERTIES OF PARTICULATE/FIBER/POLYMER COMPOSITES

Julie Tomasi

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INVESTIGATION OF MECHANICAL, ELECTRICAL, AND THERMAL PROPERTIES OF PARTICULATE/FIBER/POLYMER COMPOSITES

By

Julie Marie Tomasi

A DISSERTATION

Submitted in partial fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

In Chemical Engineering

MICHIGAN TECHNOLOGICAL UNIVERSITY

2018

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This dissertation has been approved in partial fulfillment of the requirements for the Degree of DOCTOR OF PHILOSOPHY in Chemical Engineering.

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Table of Contents

List of Figures	vii
List of Tables	xii
Preface.....	xxiii
Acknowledgements.....	xxiv
Abstract.....	xxv
1. Introduction	1
1.1 Introduction and Motivation	1
1.1.1 Aerospace Introduction.....	2
1.1.2 Power Transmission Line Introduction.....	3
1.2 Objectives	6
1.3 References.....	7
2. Effects of Carbon Fillers on the Conductivity and Tensile Properties of Polyetheretherketone (PEEK) Composites.....	10
2.1 Abstract	10
2.2 Introduction.....	11
2.3 Materials and Experimental Methods	12
2.3.1 Materials	12
2.3.2 Test Specimen Fabrication.....	14
2.3.3 Field Emission Electron Microscope (FESEM) Test Method	16
2.3.4 Carbon Fiber Length Test Method.....	17
2.3.5 Electrical Resistivity (ER) Test Method	17
2.3.6 Thermal Conductivity (TC) Test Method	18
2.3.7 Tensile Test Method	19
2.4 Results.....	19
2.4.1 Field Emission Scanning Electron Microscopy (FESEM) Results.....	19
2.4.2 Carbon Fiber Length Results	21
2.4.3 Electrical Resistivity Results	22
2.4.4 Thermal Conductivity (TC) Results.....	29
2.4.5 Tensile Results	34
2.5 Conclusions.....	39
2.6 Future Work	40
2.7 Acknowledgements.....	41
2.8 References.....	42
3. Electrical, Thermal, and Tensile Properties of Cycloaliphatic Epoxy/Carbon Black and Cycloaliphatic Epoxy/Fumed Silica Nanocomposites.....	44
3.1 Abstract	44
3.2 Introduction.....	45
3.3 Materials	46
3.4 Experimental	48
3.4.1 Fabrication	48
3.4.2 Electrical Resistivity Testing	51
3.4.3 Thermal Conductivity Testing	52

3.4.4	Tensile Testing.....	52
3.4.5	Glass Transition Temperature Testing.....	52
3.5	Results and Discussion	54
3.5.1	Electrical Resistivity	54
3.5.2	Thermal Conductivity Results	56
3.5.3	Tensile Results	56
3.5.4	Glass Transition Temperature.....	59
3.6	Conclusions.....	60
3.7	Future Work	60
3.8	Acknowledgements.....	61
3.9	References	61
4.	Thermal, Electrical, and Mechanical Properties of Talc- and Glass Microsphere-Reinforced Cycloaliphatic Epoxy Composites.....	64
4.1	Abstract	64
4.2	Introduction.....	65
4.3	Materials and Experimental Methods	68
4.3.1	Materials	68
4.3.2	Test Specimen Fabrication.....	69
4.3.3	Scanning Electron Microscope (SEM) Test Methods	71
4.3.4	Electrical Resistivity (ER) Test Method	72
4.3.5	Thermal Conductivity (TC) Test Method	73
4.3.6	Tensile Test Method	73
4.4	Results.....	73
4.4.1	Scanning Electron Microscopy (SEM) Results	73
4.4.2	Electrical Resistivity (ER) Results.....	75
4.4.3	Thermal Conductivity (TC) Results.....	76
4.4.4	Tensile Results	77
4.5	Conclusions.....	85
4.6	Future Work	86
4.7	Acknowledgements.....	87
4.8	References	88
5.	Accelerated Hygrothermal Aging of Cycloaliphatic Epoxy/Graphene Nanoparticle Composites	91
5.1	Abstract	91
5.2	Introduction.....	92
5.3	Materials and Experimental Methods	93
5.3.1	Materials	93
5.3.2	Test Specimen Fabrication.....	95
5.3.3	Testing Procedure	97
5.4	Results and Discussion	99
5.4.1	Moisture Content Results.....	99
5.4.2	Flexural Modulus Results	100
5.4.3	Flexural Strength Results.....	102
5.4.4	Glass Transition Temperature (T_g) Results.....	103

5.5	Conclusions.....	105
5.6	Future Work.....	106
5.7	Acknowledgements.....	107
5.8	References.....	107
6.	Accelerated Hygrothermal Aging of Talc/Cycloaliphatic Epoxy Composites	110
6.1	Abstract.....	110
6.2	Introduction.....	111
6.3	Materials and Experimental Methods	112
6.3.1	Materials	112
6.3.2	Test Specimen Fabrication.....	114
6.3.3	Aging and Moisture Gain Measurement Method	115
6.3.4	Environmental Scanning Electron Microscope (ESEM) Test Method.....	117
6.3.5	Three-Point Bend Test Method.....	117
6.3.6	Dynamic Mechanical Analysis (DMA) Test Method.....	118
6.4	Results.....	118
6.4.1	Environmental Scanning Electron Microscopy (ESEM) Results	118
6.4.2	Moisture Content Results.....	119
6.4.3	Flexural Modulus Results	120
6.4.4	Flexural Strength Results.....	122
6.4.5	Glass Transition Temperature (T_g) Results.....	123
6.5	Conclusions.....	126
6.6	Future Work.....	127
6.7	Acknowledgements.....	127
6.8	References.....	128
7.	Conclusions and Future Work.....	131
7.1	Conclusions.....	131
7.1.1	PEEK Conclusions.....	131
7.1.2	CE Conclusions.....	133
7.2	Future Work	137
7.2.1	PEEK Future Work	137
7.2.2	CE Future Work.....	138
7.3	References.....	139
8.	Appendix	140
8.1	PEEK Extrusion and Injection Molding Conditions.....	140
8.2	PEEK Electrical Resistivity Data.....	166
8.3	PEEK Thermal Conductivity Data.....	181
8.4	PEEK Tensile Data	195
8.5	Carbon Black and Fumed Silica Electrical Resistivity Data	216
8.6	Carbon Black and Fumed Silica Thermal Conductivity Data	221
8.7	Carbon Black and Fumed Silica Tensile Data	225
8.8	Carbon Black and Fumed Silica DMA Data.....	235
8.9	Talc and Glass Microspheres Electrical Resistivity Data.....	242
8.10	Talc and Glass Microspheres Thermal Conductivity Data.....	247
8.11	Talc and Glass Microspheres Tensile Data.....	251

8.12	Aging TC307 Water Mass Gain Data	261
8.13	Aging TC307 Flexural Data.....	262
8.14	Aging TC307 DMA Data.....	277
8.15	Aging Talc Water Mass Gain Data	287
8.16	Aging Talc Flexural Data.....	288
8.17	Aging Talc DMA Data.....	303
8.18	Copyright Agreements and Permissions	310

List of Figures

Figure 2.1: Extruder screw design used for TC307 GNP/PEEK composites	15
Figure 2.2: FESEM micrograph of composite containing 4 wt% TC307 in PEEK	20
Figure 2.3: FESEM micrograph of composite containing 10 wt% TC307 in PEEK	20
Figure 2.4: FESEM micrograph of composite containing 30 wt% carbon fiber in PEEK	21
Figure 2.5: Log(electrical resistivity in ohm-cm) vs. filler content for composites containing varying amounts of a single filler type for GNP/PEEK, CB/PEEK, and CF/PEEK.....	23
Figure 2.6: Log(electrical resistivity in ohm-cm) contour plot for all PEEK-based formulations	29
Figure 2.7: Thermal conductivity in W/m-K vs. filler content for composites containing varying amounts of a single filler type for GNP/PEEK, CB/PEEK, and CF/PEEK	31
Figure 2.8: Tensile modulus vs. filler content for composites containing varying amounts of a single filler type for GNP/PEEK, CB/PEEK, and CF/PEEK	36
Figure 2.9: Ultimate tensile strength vs filler content for composites containing varying amounts of a single filler type for GNP/PEEK, CB/PEEK, and CF/PEEK.....	38
Figure 3.1: a) Resin Components, b) ACA Hardener Components.....	47
Figure 3.2: Electrical Resistivity of Epoxy with Different Fillers.....	55
Figure 3.3: Thermal Conductivity of Epoxy with Different Fillers.....	56
Figure 3.4: Ultimate Tensile Stress of Epoxy with Different Fillers.....	57
Figure 3.5: Tensile Modulus of Epoxy with Different Fillers	58
Figure 3.6: Glass Transition Temperature for Epoxy with Different Fillers as Determined by the Peak of the Tan Delta Curve from DMA	59
Figure 4.1: a) CE Resin Component, b) ACA Hardener Component.....	68
Figure 4.2: Scanning Electron Microscopy Image of 20 wt% Talc/Epoxy Composite...	74
Figure 4.3: Field Emission Scanning Electron Microscopy Image of 40 wt% Glass MS/Epoxy Composite.....	75
Figure 4.4: Ultimate Tensile Strength in MPa vs. Volume Fraction Talc or Glass MS ..	79
Figure 4.5: Tensile Modulus in GPa vs. Volume Fraction Talc	80
Figure 4.6: Tensile Modulus in GPa vs. Volume Fraction Glass MS.....	80
Figure 5.1: Monomer Molecules used for Solid Epoxy Matrix.....	94
Figure 5.2: Scanning Electron Microscopy Image of the GNP/Epoxy Composite	97
Figure 5.3: Water Mass Gain for GNP/Epoxy Nanocomposites	100
Figure 5.4: Flexural Modulus for GNP/Epoxy Nanocomposites.....	101
Figure 5.5: Flexural Strength for GNP/Epoxy Nanocomposites	103
Figure 5.6: Glass Transition Temperature for GNP/Epoxy Nanocomposites	104
Figure 6.1: Monomer Molecules used for Solid Epoxy Matrix: a) Cycloaliphatic Epoxy Resin, and b) Anhydride Curing Agent Hardener	113
Figure 6.2: Scanning Electron Microscopy Image of a Polished Surface of the Talc/Epoxy Composite.....	119
Figure 6.3: Water Mass Gain for Talc/Epoxy Composites.....	120

Figure 6.4: Flexural Modulus for Talc/Epoxy Composites	121
Figure 6.5: Flexural Strength for Talc/Epoxy Composites.....	123
Figure 6.6: Glass Transition Temperature determined by Tan Delta Peak for Talc/Epoxy Composites.....	125
Figure 6.7: Glass Transition Temperature determined by Storage Modulus Onset for Talc/Epoxy Composites.....	126
Figure 8.1: Extruder Screw Design named 2-5-15	140
Figure 8.2: Extruder Screw Design named 7-8-15	140
Figure 8.3: Extruder Screw Design named 8-27-15 used with Carbon Fiber and Carbon Black	141
Figure 8.4: Tensile Results for KetaSpire KT-880 NT Injection Molded 6-30-15at MTU	196
Figure 8.5 Tensile Results for VD2K: 2 wt% TC307 GNP in KetaSpire KT-880 NT: Injection Molded at Solvay 5-14-15, Extruded March 2015 at MTU	197
Figure 8.6: Tensile Results for VD4K: 4 wt% TC307 GNP in KetaSpire KT-880 NT: Injection Molded at Solvay 5-14-15, Extruded March 2015 at MTU	198
Figure 8.7: Tensile Results for VD6K: 6 wt% TC307 GNP in KetaSpire KT-880 NT: Injection Molded at Solvay 5-14-15, Extruded March 2015 at MTU	199
Figure 8.8: Tensile Results for VD8K: 8 wt% TC307 GNP in KetaSpire KT-880 NT: Injection Molded at Solvay 5-14-15, Extruded March 2015 at MTU	200
Figure 8.9: Tensile Results for VD10K: 10 wt% TC307 GNP in KetaSpire KT-880 NT: Injection Molded at Solvay 5-14-15, Extruded March 2015 at MTU	201
Figure 8.10: Tensile Results for VD12.5K: 12.5 wt% TC307 GNP in KetaSpire KT-880 NT: Extruded April 13 2016 at MTU, IM at MTU April 20 2016	202
Figure 8.11: Tensile Results for VD15K: 15 wt% TC307 GNP in KetaSpire KT-880 NT: Extruded 8-25-15 at MTU screw 7-8-15, IM at MTU Sept 10 2015.....	203
Figure 8.12: Tensile Results for VF10K: 10 wt% Carbon Fiber from KT-880 CF30 in KetaSpire KT-880 NT: Extruded 3-2-16 at MTU screw 8-27-15, IM at MTU 3-23-16	204
Figure 8.13: Tensile Results for VF20K: 20 wt% Carbon Fiber from KT-880 CF30 in KetaSpire KT-880 NT: Extruded 4-13-16 at MTU screw 8-27-15, IM at MTU April 20 2016	205
Figure 8.14: Tensile Results for VF30K: 30 wt% Carbon Fiber from KT-880 CF30, IM at Solvay May 2015	206
Figure 8.15: Tensile Results for VA2.5K: 2.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black in Solvay KetaSpire KT-880 NT: Extruded 2-16-16 at MTU screw 8-27-15, IM at MTU 2-24-16.....	207
Figure 8.16: Tensile Results for VA5K: 5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black in Solvay KetaSpire KT-880 NT: Extruded 2-16-16 at MTU screw 8-27-15, IM at MTU 2-24-16.....	208
Figure 8.17: Tensile Results for VA7.5K: 7.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black in Solvay KetaSpire KT-880 NT: Extruded 3-2-16 at MTU screw 8-27-15, IM at MTU 5-10-16.....	209

Figure 8.18: Tensile Results for VA10K: 10 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black in Solvay KetaSpire KT-880 NT: Extruded 3-2-16 at MTU screw 8-27-15, IM at MTU 5-10-16.....	210
Figure 8.19: Tensile Results for VA2.5F30K: 2.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black and 30 wt% Carbon Fiber from Solvay KT-880 CF30 in Solvay KetaSpire KT-880 NT: Extruded 6-19-16 at MTU screw 8-27-15, IM at MTU 6-23-16	211
Figure 8.20: Tensile Results for VA5F30K: 5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black and 30 wt% Carbon Fiber from Solvay KT-880 CF30 in Solvay KetaSpire KT-880 NT: Extruded 5-30-16 at MTU screw 8-27-15, IM at MTU 6-7-16	212
Figure 8.21: Tensile Results for VA7.5F30K: 7.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black and 30 wt% Carbon Fiber from Solvay KT-880 CF30 in Solvay KetaSpire KT-880 NT: Extruded 5-30-16 at MTU screw 8-27-15, IM at MTU 6-7-16	213
Figure 8.22: Tensile Results for VA2.5F10K: 2.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black and 10 wt% Carbon Fiber from Solvay KT-880 CF30 in Solvay KetaSpire KT-880 NT: Extruded 3-2-16 at MTU screw 8-27-15, IM at MTU 3-23-16	214
Figure 8.23: Tensile Results for VA2.5F20K: 2.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black and 20 wt% Carbon Fiber from Solvay KT-880 CF30 in Solvay KetaSpire KT-880 NT: Extruded 4-13-16 at MTU screw 8-27-15, IM at MTU 4-20-16	215
Figure 8.24 Tensile Results 1 wt% Cabot Carbon Black 2 in Cycloaliphatic Epoxy....	226
Figure 8.25 Tensile Results 1 wt% Cabot Carbon Black 3 in Cycloaliphatic Epoxy....	227
Figure 8.26 Tensile Results 0.5 wt% Cabot Carbon Black 1 in Cycloaliphatic Epoxy.....	228
Figure 8.27 Tensile Results 1 wt% Cabot Carbon Black 1 in Cycloaliphatic Epoxy....	229
Figure 8.28 Tensile Results 2 wt% Cabot Carbon Black 1 in Cycloaliphatic Epoxy....	230
Figure 8.29 Tensile Results 1 wt% Silica1 in Cycloaliphatic Epoxy	231
Figure 8.30 Tensile Results 2 wt% Silica1 in Cycloaliphatic Epoxy	232
Figure 8.31 Tensile Results 3 wt% Silica1 in Cycloaliphatic Epoxy	233
Figure 8.32 Tensile Results 4 wt% Silica1 in Cycloaliphatic Epoxy	234
Figure 8.33: Loss Modulus for Carbon Black 1 in Cycloaliphatic Epoxy.....	237
Figure 8.34: Storage Modulus for Carbon Black 1 in Cycloaliphatic Epoxy.....	237
Figure 8.35: Tan Delta for Carbon Black 1 in Cycloaliphatic Epoxy	238
Figure 8.36: Loss Modulus for Silica1 in Cycloaliphatic Epoxy.....	240
Figure 8.37: Storage Modulus for Silica1 in Cycloaliphatic Epoxy.....	240
Figure 8.38: Tan Delta for Silica 1 in Cycloaliphatic Epoxy	241
Figure 8.39: Tensile Results for Neat Cycloaliphatic Epoxy	251
Figure 8.40: Tensile Results for 5 wt% Talc in Cycloaliphatic Epoxy	252
Figure 8.41: Tensile Results for 10 wt% Talc in Cycloaliphatic Epoxy	253
Figure 8.42: Tensile Results for 15 wt% Talc in Cycloaliphatic Epoxy	254
Figure 8.43: Tensile Results for 20 wt% Talc in Cycloaliphatic Epoxy	255
Figure 8.44: Tensile Results for 5 wt% Glass Microspheres in Cycloaliphatic Epoxy.....	256

Figure 8.45: Tensile Results for 10 wt% Glass Microspheres in Cycloaliphatic Epoxy	257
Figure 8.46: Tensile Results for 20 wt% Glass Microspheres in Cycloaliphatic Epoxy	258
Figure 8.47: Tensile Results for 30 wt% Glass Microspheres in Cycloaliphatic Epoxy	259
Figure 8.48: Tensile Results for 40 wt% Glass Microspheres in Cycloaliphatic Epoxy	260
Figure 8.49: Flexural Results for Neat Cycloaliphatic Epoxy Unaged	262
Figure 8.50: Flexural Results for Neat Cycloaliphatic Epoxy aged 50 hours	263
Figure 8.51: Flexural Results for Neat Cycloaliphatic Epoxy aged 100 hours	264
Figure 8.52: Flexural Results for Neat Cycloaliphatic Epoxy aged 200 hours	265
Figure 8.53: Flexural Results for Neat Cycloaliphatic Epoxy aged 400 hours	266
Figure 8.54: Flexural Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Unaged	267
Figure 8.55: Flexural Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 50 hours.....	268
Figure 8.56: Flexural Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 100 hours.....	269
Figure 8.57: Flexural Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 200 hours.....	270
Figure 8.58: Flexural Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 400 hours.....	271
Figure 8.59: Flexural Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Unaged	272
Figure 8.60: Flexural Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 50 hours.....	273
Figure 8.61: Flexural Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Age 100 hour	274
Figure 8.62: Flexural Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Age 200 hour	275
Figure 8.63: Flexural Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Age 400 hour	276
Figure 8.64: Storage Modulus for Neat Cycloaliphatic Epoxy.....	279
Figure 8.65: Loss Modulus for Neat Cycloaliphatic Epoxy	279
Figure 8.66: Tan Delta for Neat Cycloaliphatic Epoxy	280
Figure 8.67: Storage Modulus for 2 wt% TC307 GNP in Cycloaliphatic Epoxy.....	282
Figure 8.68: Loss Modulus for 2 wt% TC307 GNP in Cycloaliphatic Epoxy	283
Figure 8.69: Tan Delta for 2 wt% TC307 GNP in Cycloaliphatic Epoxy	283
Figure 8.70: Storage Modulus for 4 wt% TC307 GNP in Cycloaliphatic Epoxy.....	285
Figure 8.71: Loss Modulus for 4 wt% TC307 GNP in Cycloaliphatic Epoxy	286
Figure 8.72: Tan Delta for 4 wt% TC307 GNP in Cycloaliphatic Epoxy	286
Figure 8.73: Flexural Results for Neat Cycloaliphatic Epoxy Unaged	288
Figure 8.74: Flexural Results for Neat Cycloaliphatic Epoxy aged 50 hours	289
Figure 8.75: Flexural Results for Neat Cycloaliphatic Epoxy aged 100 hours	290
Figure 8.76: Flexural Results for Neat Cycloaliphatic Epoxy aged 200 hours	291
Figure 8.77: Flexural Results for Neat Cycloaliphatic Epoxy aged 400 hours	292
Figure 8.78: Flexural Results for 10% Talc in Cycloaliphatic Epoxy Unaged	293

Figure 8.79: Flexural Results for 10% Talc in Cycloaliphatic Epoxy Aged 50 hours ..	294
Figure 8.80: Flexural Results for 10% Talc in Cycloaliphatic Epoxy Aged 100 hours	295
Figure 8.81: Flexural Results for 10% Talc in Cycloaliphatic Epoxy Aged 200 hours	296
Figure 8.82: Flexural Results for 10% Talc in Cycloaliphatic Epoxy Aged 400 hours	297
Figure 8.83: Flexural Results for 20% Talc in Cycloaliphatic Epoxy Unaged	298
Figure 8.84: Flexural Results for 20% Talc in Cycloaliphatic Epoxy Aged 50 hours ..	299
Figure 8.85: Flexural Results for 20% Talc in Cycloaliphatic Epoxy Age 100 hour	300
Figure 8.86: Flexural Results 20% Talc in Cycloaliphatic Epoxy Age 200 hour.....	301
Figure 8.87: Flexural Results for 20% Talc in Cycloaliphatic Epoxy Age 400 hour	302
Figure 8.88: Tan Delta Peak for Aged Talc/CE composites.....	308
Figure 8.89: Storage Modulus Onset for Aged Talc/CE composites.....	308
Figure 8.90: Loss Modulus Peak for Aged Talc/CE composites.....	309

List of Tables

Table 2.1: Filler loading levels in PEEK along with electrical resistivity and thermal conductivity results	14
Table 2.2: Electrical resistivity and thermal conductivity results for factorial design formulations	25
Table 2.3: 2 ² factorial results for Log(electrical resistivity, ohm-cm).....	27
Table 2.4: 2 ² factorial results for thermal conductivity in W/m-K.....	33
Table 2.5: Tensile properties of all PEEK based formulations.....	35
Table 3.1: Electrical Resistivity and Thermal Conductivity at 55 °C for Carbon Black/Epoxy and Fumed Silica/Epoxy Composites	55
Table 3.2: Ultimate Tensile Stress, Strain at Ultimate Tensile Stress, and Tensile Modulus for Carbon Black/Epoxy and Fumed Silica/Epoxy Composites.....	58
Table 3.3: Storage Modulus Onset, Loss Modulus Peak, and Tan Delta Peak for Carbon Black/Epoxy and Fumed Silica/Epoxy Composites	60
Table 4.1: Filler Loading Levels and Electrical Resistivity and Thermal Conductivity Results for Talc/Epoxy Composites and Glass MS/Epoxy Composites.....	76
Table 4.2: Tensile Properties of Talc/Epoxy Composites and Glass MS/Epoxy Composites.....	78
Table 5.1: Flexural Modulus for GNP/Epoxy Nanocomposites.....	102
Table 5.2: Flexural Strength for GNP/Epoxy Nanocomposites.....	103
Table 5.3: Glass Transition Temperature for GNP/Epoxy Nanocomposites.....	105
Table 6.1: Flexural Modulus for Talc/Epoxy Composites.....	122
Table 6.2: Flexural Strength for Talc/Epoxy Composites	123
Table 6.3: Glass Transition Temperature determined by Tan Delta Peak for Talc/Epoxy Composites.....	125
Table 8.1: Extrusion Conditions for Solvay KetaSpire KT-880 NT with Asbury Carbons TC307 GNP	141
Table 8.2: Extrusion Conditions for Solvay KetaSpire KT-880 NT with Asbury Carbons T307 GNP (continued from Table 8.1).....	142
Table 8.3: Extrusion Conditions for Solvay KetaSpire KT-880 NT with Akzo Nobel Ketjenblack EC-600JD Carbon Black	144
Table 8.4: Extrusion Conditions for Solvay KetaSpire KT-880 NT with Akzo Nobel Ketjenblack EC-600JD Carbon Black and/or Carbon Fiber from Solvay KT-880 CF30.....	146
Table 8.5: Extrusion Conditions for Solvay KetaSpire KT-880 NT with Akzo Nobel Ketjenblack EC-600JD Carbon Black and Carbon Fiber from Solvay KT-880 CF30.....	148
Table 8.6: Extrusion Conditions for Solvay KetaSpire KT-880 NT with Akzo Nobel Ketjenblack EC-600JD Carbon Black or Carbon Fiber from Solvay KT-880 CF30.....	150
Table 8.7: Extrusion Conditions for Solvay KetaSpire KT-880 NT with Akzo Nobel Ketjenblack EC-600 JD Carbon Black and Carbon Fiber from Solvay KT-880 CF30.....	152

Table 8.8: Injection Molding Conditions for Asbury TC307 GNP in KT-880 NT	154
Table 8.9: Injection Molding Conditions for Asbury TC307 GNP in KT-880 NT (continued from Table 8.8)	155
Table 8.10: Injection Molding Conditions for Asbury TC307 GNP in KT-880 NT	156
Table 8.11: Injection Molding Conditions for Akzo Nobel Ketjenblack EC-600 JD Carbon Black and/or Carbon Fiber from KT-880 CF30 in KT-880 NT.....	158
Table 8.12: Injection Molding Conditions for Akzo Nobel Ketjenblack EC-600 JD Carbon Black and/or Carbon Fiber from KT-880 CF30 in KT-880 NT.....	160
Table 8.13: Injection Molding Conditions for Akzo Nobel Ketjenblack EC-600 JD Carbon Black in KT-880 NT	162
Table 8.14: Injection Molding Conditions for Akzo Nobel Ketjenblack EC-600 JD Carbon Black and Carbon Fiber from KT-880 CF30 in KT-880 NT	164
Table 8.15: ASTM D257 Through-Plane Electrical Resistivity Results for KetaSpire KT 880 NT injection molded at MTU 6-30-15	166
Table 8.16: ASTM D257 Through-Plane Electrical Resistivity Results for VD2K: 2 wt% TC307 GNP in KetaSpire KT880 NT: Extruded 3-17-15 at MTU, 2-5-15 extruder screw: Injection Molded at Solvay	167
Table 8.17: ASTM D257 Through-Plane Electrical Resistivity Results for VD4K: 4 wt% TC307 GNP in KetaSpire KT880 NT: Extruded 3-17-15 at MTU, 2-5-15 extruder screw: Injection Molded at Solvay	167
Table 8.18: ASTM D257 Through-Plane Electrical Resistivity Results for VD6K: 6 wt% TC307 GNP in KetaSpire KT880 NT: Extruded 3-17-15 at MTU, 2-5-15 extruder screw: Injection Molded at Solvay	168
Table 8.19: ASTM D257 Through-Plane Electrical Resistivity Results for VD8K: 8 wt% TC307 GNP in KetaSpire KT880 NT: Extruded 3-19-15 at MTU, 2-5-15 extruder screw: Injection Molded at Solvay	168
Table 8.20: ASTM D257 Through-Plane Electrical Resistivity Results for VD10K: 10 wt% TC307 GNP in KetaSpire KT880 NT: Extruded 3-19-15 at MTU, 2-5-15 extruder screw: Injection Molded at Solvay	169
Table 8.21: ASTM D257 Through-Plane Electrical Resistivity Results for VD12.5K: 12.5 wt% TC307 GNP in KetaSpire KT880 NT: Extruded 4-13-16 at MTU, 78- 27-15 extruder screw: Injection Molded at MTU 4-20-16	169
Table 8.22: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for VD15K: 15 wt% TC307 in KT880 NT: extruded 8-25-15 at MTU, 7-8-15 screw, Injection Molded at MTU 9-10-15	170
Table 8.23: ASTM D257 Through-Plane Electrical Resistivity Results for 10 wt% Carbon Fiber from KT-880 CF30 in KetaSpire KT 880 NT Extruded 3-2-2016, 8-27-15 screw, Injection Molded at MTU 3-23-16	170
Table 8.24: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for VF20K: 20 wt% Carbon Fiber from KT 880 CF30 with KT880 NT: Extruded 4-13-16 at MTU, 8-27-15 screw, Injection Molded at MTU 4-20-16.....	171
Table 8.25: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for KT880 CF30: 30 wt% Carbon Fiber in KetaSpire KT880 NT: From Solvay: Injection Molded at Solvay	172

Table 8.26: ASTM D257 Through-Plane Electrical Resistivity Results for 2.5 wt% Ketjenblack EC-600 JD Carbon Black in KetaSpire KT 880 NT Extruded 2-16-16, 8-27-15 screw, Injection Molded at MTU 2-24-16	173
Table 8.27: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for VA5K: 5 wt% Ketjenblack EC-600 JD in KT880 NT: Extruded 2-16-16 at MTU, 8-27-15 screw, Injection Molded at MTU 2-24-16	174
Table 8.28: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for VA7.5K: 7.5 wt% Ketjenblack EC-600 JD in KetaSpire KT 880 NT, Extruded 3-2-16 with 8-27-15 screw, Injection Molded at MTU 5-10-16	175
Table 8.29: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for VA10K: 10 wt% Ketjenblack EC-600 JD in KetaSpire KT 880 NT, Extruded 4-13-16 with 8-27-15 screw, Injection Molded at MTU 5-10-16	176
Table 8.30: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for VA2.5F30K: 2.5 wt% Ketjenblack EC-600 JD Carbon Black, 30 wt% Carbon Fiber from KT-880 CF30 with KT-880 NT: Extruded 6-19-16 at MTU, 8-27-15 screw, Injection Molded at MTU 6-23-16	177
Table 8.31: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for VA5F30K: 5 wt% Ketjenblack EC-600 JD Carbon Black, 30 wt% Carbon Fiber from KT-880 CF30 with KT-880 NT: Extruded 5-30-16 at MTU, 8-27-15 screw, Injection Molded at MTU 6-7-16	178
Table 8.32: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for VA7.5F30K: 7.5 wt% Ketjenblack EC-600 JD Carbon Black, 30 wt% Carbon Fiber from KT-880 CF30 with KT-880 NT: Extruded 5-30-16 at MTU, 8-27-15 screw, Injection Molded at MTU 6-7-16	179
Table 8.33: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for VA2.5F10K: 2.5 wt% Ketjenblack EC-600 JD Carbon Black, 10 wt% Carbon Fiber from KT-880 CF30 with KT-880 NT: Extruded 3-2-16 at MTU, 8-27-15 screw, Injection Molded at MTU 3-23-16	180
Table 8.34: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for VA2.5F20K: 2.5 wt% Ketjenblack EC-600 JD Carbon Black, 20 wt% Carbon Fiber from KT-880 CF30 with KT-880 NT: Extruded 4-13-16 at MTU, 8-27-15 screw, Injection Molded at MTU 4-20-16	180
Table 8.35: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300 Guarded Heat Flow Meter Method for KetaSpire KT-880 NT: Injection Molded at MTU June 30, 2015	181
Table 8.36: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300 Guarded Heat Flow Meter Method for 2 wt% TC307 GNP in KetaSpire KT-880 NT (VD2K): Extruded at MTU 3-17-15, 2-5-15 extruder screw, Injection Molded May 14, 2015 by Solvay.....	182
Table 8.37: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300 Guarded Heat Flow Meter Method for 4 wt% TC307 GNP in KetaSpire KT-880 NT (VD4K): Extruded at MTU 3-17-15, 2-5-15 extruder screw: Injection Molded May 14, 2015 by Solvay.....	182

Table 8.38: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300 Guarded Heat Flow Meter Method for 6 wt% TC307 GNP in KetaSpire KT-880 NT (VD6K): Extruded at MTU 3-17-15, 2-5-15 extruder screw: Injection Molded May 14, 2015 by Solvay.....	183
Table 8.39: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300 Guarded Heat Flow Meter Method for 8 wt% TC307 GNP in KetaSpire KT-880 NT (VD8K): Extruded at MTU 3-19-15, 2-5-15 extruder screw: Injection Molded May 14, 2015 by Solvay.....	183
Table 8.40: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300 Guarded Heat Flow Meter Method for 10 wt% TC307 GNP in KetaSpire KT-880 NT (VD10K): Extruded at MTU 3-19-15, 2-5-15 extruder screw: Injection Molded May 14, 2015 by Solvay.....	184
Table 8.41: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300 Guarded Heat Flow Meter Method for 12.5 wt% TC307 GNP in KetaSpire KT-880 NT (VD12.5K): Extruded at MTU 4-13-16 all in zone 1, 8-27-15 extruder screw: Injection Molded 4-20-16 at MTU	184
Table 8.42: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300 Guarded Heat Flow Meter Method for 15 wt% TC307 GNP in KetaSpire KT-880 NT (VD10K): Extruded at MTU 8-25-15, 7-8-15 extruder screw: Injection Molded Sept 10, 2015 at MTU	185
Table 8.43: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300 Guarded Heat Flow Meter Method for 2.5 wt% Ketjenblack EC-600 JD in KetaSpire KT-880 NT (VA2.5K): Extruded at MTU 2-16-16, 8-27-15 extruder screw: Injection Molded at MTU 2-24-16	186
Table 8.44: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300 Guarded Heat Flow Meter Method for 5 wt% Ketjenblack EC-600 JD in KetaSpire KT-880 NT (VA5K): Extruded at MTU 2-16-16, 8-27-15 extruder screw: Injection Molded at MTU 2-24-16	187
Table 8.45: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300 Guarded Heat Flow Meter Method for 7.5 wt% Ketjenblack EC-600 JD in KetaSpire KT-880 NT (VA7.5K): Extruded at MTU 3-2-16, 8-27-15 extruder screw: Injection Molded at MTU 5-10-16	188
Table 8.46: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300 Guarded Heat Flow Meter Method for 10 wt% Ketjenblack EC-600 JD in KetaSpire KT-880 NT (VA10K): Extruded at MTU 4-13-16, 8-27-15 extruder screw: Injection Molded at MTU 5-10-16	189
Table 8.47: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300 Guarded Heat Flow Meter Method for 10 wt% carbon fiber from KT-880 CF30 in KetaSpire KT-880 NT (VF10K): Extruded at MTU 3-2-16, 8-27-15 extruder screw: Injection Molded at MTU 3-23-16	189
Table 8.48: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300 Guarded Heat Flow Meter Method for 20 wt% carbon fiber from KT-880 CF30 in KetaSpire KT-880 NT (VF10K): Extruded at MTU 4-13-16, 8-27-15 extruder screw: Injection Molded at MTU 4-20-16	190

Table 8.49: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300 Guarded Heat Flow Meter Method for KetaSpire KT-880 CF30 (30 wt% Carbon Fiber): From Solvay: Injection Molded May 14, 2015 by Solvay.....	191
Table 8.50: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300 Guarded Heat Flow Meter Method for 2.5 wt% Ketjenblack EC-600 JD, 30 wt% carbon fiber from KT-880 CF30 in KetaSpire KT-880 NT (VA2.5F30K): Extruded at MTU 6-19-16, 8-27-15 extruder screw: Injection Molded at MTU 6-23-16	192
Table 8.51: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300 Guarded Heat Flow Meter Method for 5 wt% Ketjenblack EC-600 JD, 30 wt% carbon fiber from KT-880 CF30 in KetaSpire KT-880 NT (VA2.5F20K): Extruded at MTU 5-30-16, 8-27-15 extruder screw: Injection Molded at MTU 6-7-16	193
Table 8.52: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300 Guarded Heat Flow Meter Method for 7.5 wt% Ketjenblack EC-600 JD, 30 wt% carbon fiber from KT-880 CF30 in KetaSpire KT-880 NT (VA2.5F20K): Extruded at MTU 5-30-16, 8-27-15 extruder screw: Injection Molded at MTU 6-7-16	194
Table 8.53: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300 Guarded Heat Flow Meter Method for 2.5 wt% Ketjenblack EC-600 JD, 10 wt% carbon fiber from KT-880 CF30 in KetaSpire KT-880 NT (VA2.5F10K): Extruded at MTU 3-2-16, 8-27-15 extruder screw: Injection Molded at MTU 3-23-16	195
Table 8.54: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300 Guarded Heat Flow Meter Method for 2.5 wt% Ketjenblack EC-600 JD, 20 wt% carbon fiber from KT-880 CF30 in KetaSpire KT-880 NT (VA2.5F20K): Extruded at MTU 4-13-16, 8-27-15 extruder screw: Injection Molded at MTU 4-20-16	195
Table 8.55: Tensile Results for KetaSpire KT-880 NT Injection Molded 6-30-15 at MTU	196
Table 8.56: Tensile Results for VD2K: 2 wt% TC307 GNP in KetaSpire KT-880 NT: Injection Molded at Solvay 5-14-15, Extruded March 2015 at MTU	197
Table 8.57: Tensile Results for VD4K: 4 wt% TC307 GNP in KetaSpire KT-880 NT: Injection Molded at Solvay 5-14-15, Extruded March 2015 at MTU	198
Table 8.58: Tensile Results for VD6K: 6 wt% TC307 GNP in KetaSpire KT-880 NT: Injection Molded at Solvay 5-14-15, Extruded March 2015 at MTU	199
Table 8.59: Tensile Results for VD8K: 8 wt% TC307 GNP in KetaSpire KT-880 NT: Injection Molded at Solvay 5-14-15, Extruded March 2015 at MTU	200
Table 8.60: Tensile Results for VD10K: 10 wt% TC307 GNP in KetaSpire KT-880 NT: Injection Molded at Solvay 5-14-15, Extruded March 2015 at MTU	201
Table 8.61: Tensile Results for VD12.5K: 12.5 wt% TC307 GNP in KetaSpire KT-880 NT: Extruded April 13 2016 at MTU, IM at MTU April 20 2016	202
Table 8.62: Tensile Results for VD15K: 15 wt% TC307 GNP in KetaSpire KT-880 NT: Extruded 8-25-15 at MTU screw 7-8-15, IM at MTU Sept 10 2015.....	203

Table 8.63: Tensile Results for VF10K: 10 wt% Carbon Fiber from KT-880 CF30 in KetaSpire KT-880 NT: Extruded 3-2-16 at MTU screw 8-27-15, IM at MTU 3-23-16	204
Table 8.64: Tensile Results for VF20K: 20 wt% Carbon Fiber from KT-880 CF30 in KetaSpire KT-880 NT: Extruded 4-13-16 at MTU screw 8-27-15, IM at MTU April 20 2016	205
Table 8.65: Tensile Results for VF30K: 30 wt% Carbon Fiber from KT-880 CF30, IM at Solvay May 2015	206
Table 8.66: Tensile Results for VA2.5K: 2.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black in Solvay KetaSpire KT-880 NT: Extruded 2-16-16 at MTU screw 8-27-15, IM at MTU 2-24-16.....	207
Table 8.67: Tensile Results for VA5K: 5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black in Solvay KetaSpire KT-880 NT: Extruded 2-16-16 at MTU screw 8-27-15, IM at MTU 2-24-16.....	208
Table 8.68: Tensile Results for VA7.5K: 7.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black in Solvay KetaSpire KT-880 NT: Extruded 3-2-16 at MTU screw 8-27-15, IM at MTU 5-10-16.....	209
Table 8.69: Tensile Results for VA10K: 10 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black in Solvay KetaSpire KT-880 NT: Extruded 3-2-16 at MTU screw 8-27-15, IM at MTU 5-10-16.....	210
Table 8.70: Tensile Results for VA2.5F30K: 2.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black and 30 wt% Carbon Fiber from Solvay KT-880 CF30 in Solvay KetaSpire KT-880 NT: Extruded 6-19-16 at MTU screw 8-27-15, IM at MTU 6-23-16	211
Table 8.71: Tensile Results for VA5F30K: 5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black and 30 wt% Carbon Fiber from Solvay KT-880 CF30 in Solvay KetaSpire KT-880 NT: Extruded 5-30-16 at MTU screw 8-27-15, IM at MTU 6-7-16	212
Table 8.72: Tensile Results for VA7.5F30K: 7.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black and 30 wt% Carbon Fiber from Solvay KT-880 CF30 in Solvay KetaSpire KT-880 NT: Extruded 5-30-16 at MTU screw 8-27-15, IM at MTU 6-7-16	213
Table 8.73: Tensile Results for VA2.5F10K: 2.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black and 10 wt% Carbon Fiber from Solvay KT-880 CF30 in Solvay KetaSpire KT-880 NT: Extruded 3-2-16 at MTU screw 8-27-15, IM at MTU 3-23-16	214
Table 8.74: Tensile Results for VA2.5F20K: 2.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black and 20 wt% Carbon Fiber from Solvay KT-880 CF30 in Solvay KetaSpire KT-880 NT: Extruded 4-13-16 at MTU screw 8-27-15, IM at MTU 4-20-16	215
Table 8.75: Electrical Resistivity Results for Neat Cycloaliphatic Epoxy	216
Table 8.76: Electrical Resistivity Results for 1 wt% Cabot Carbon Black 2 in Cycloaliphatic Epoxy.....	216

Table 8.77: Electrical Resistivity Results for 1 wt% Cabot Carbon Black 3 in Cycloaliphatic Epoxy.....	217
Table 8.78: Electrical Resistivity Results for 0.5 wt% Cabot Carbon Black 1 in Cycloaliphatic Epoxy.....	217
Table 8.79: Electrical Resistivity Results for 1 wt% Cabot Carbon Black 1 in Cycloaliphatic Epoxy.....	218
Table 8.80: Electrical Resistivity Results for 2 wt% Carbon Black 1 in Cycloaliphatic Epoxy	218
Table 8.81: Electrical Resistivity Results for 1 wt% Cabot Silica1 in Cycloaliphatic Epoxy	219
Table 8.82: Electrical Resistivity Results for 2 wt% Cabot Silica1 in Cycloaliphatic Epoxy	219
Table 8.83: Electrical Resistivity Results for 3 wt% Cabot Silica1 in Cycloaliphatic Epoxy	219
Table 8.84: Electrical Resistivity Results for 4 wt% Cabot Silica1 in Cycloaliphatic Epoxy	220
Table 8.85: Thermal Conductivity Results for Neat Cycloaliphatic Epoxy	221
Table 8.86: Thermal Conductivity Results for 1 wt% Cabot Carbon Black 2 in Cycloaliphatic Epoxy.....	221
Table 8.87: Thermal Conductivity Results for 1 wt% Cabot Carbon Black 3 in Cycloaliphatic Epoxy.....	222
Table 8.88: Thermal Conductivity Results for 0.5 wt% Cabot Carbon Black 1 in Cycloaliphatic Epoxy.....	222
Table 8.89: Thermal Conductivity Results for 1 wt% Cabot Carbon Black 1 in Cycloaliphatic Epoxy.....	223
Table 8.90: Thermal Conductivity Results for 2 wt% Cabot Carbon Black 1 in Cycloaliphatic Epoxy.....	223
Table 8.91: Thermal Conductivity Results for 1 wt% Silica1 in Cycloaliphatic Epoxy.....	223
Table 8.92: Thermal Conductivity Results for 2 wt% Silica1 in Cycloaliphatic Epoxy.....	224
Table 8.93: Thermal Conductivity Results for 3 wt% Silica1 in Cycloaliphatic Epoxy.....	224
Table 8.94: Thermal Conductivity Results for 4 wt% Silica1 in Cycloaliphatic Epoxy.....	224
Table 8.95 Tensile Results Neat Cycloaliphatic Epoxy.....	225
Table 8.96 Tensile Results 1 wt% Cabot Carbon Black 2 in Cycloaliphatic Epoxy	226
Table 8.97 Tensile Results 1 wt% Cabot Carbon Black 3 in Cycloaliphatic Epoxy	227
Table 8.98 Tensile Results 0.5 wt% Cabot Carbon Black 1 in Cycloaliphatic Epoxy ..	228
Table 8.99 Tensile Results 1 wt% Cabot Carbon Black 1 in Cycloaliphatic Epoxy	229
Table 8.100 Tensile Results 2 wt% Cabot Carbon Black 1 in Cycloaliphatic Epoxy ...	230
Table 8.101 Tensile Results 1 wt% Silica1 in Cycloaliphatic Epoxy.....	231
Table 8.102 Tensile Results 2 wt% Silica1 in Cycloaliphatic Epoxy.....	232
Table 8.103 Tensile Results 3 wt% Silica1 in Cycloaliphatic Epoxy.....	233
Table 8.104 Tensile Results 4 wt% Silica1 in Cycloaliphatic Epoxy.....	234
Table 8.105: DMA results for Neat Cycloaliphatic Epoxy made 12-4-15, tested 5-2-16 and 5-3-16	235

Table 8.106: DMA results for 0.5 wt% Carbon Black 1 in Cycloaliphatic Epoxy made 10-1-15, tested 12-15-15	235
Table 8.107: DMA results for 1 wt% Carbon Black 1 in Cycloaliphatic Epoxy made 10-22-15, tested 12-11-15	236
Table 8.108: DMA results for 2 wt% Carbon Black 1 in Cycloaliphatic Epoxy made 10-29-15, tested 12-14-15	236
Table 8.109: DMA results for 1 wt% Silical in Cycloaliphatic Epoxy made 3-15-16, tested 4-5-16.....	238
Table 8.110: DMA results for 2 wt% Silical in Cycloaliphatic Epoxy made 2-16-16, tested 4-19-16.....	239
Table 8.111: DMA results for 3 wt% Silical in Cycloaliphatic Epoxy made 2-23-16, tested 3-31-16.....	239
Table 8.112: DMA results for 4 wt% Silical in Cycloaliphatic Epoxy made 02-29-16, tested 4-12-16.....	239
Table 8.113: Electrical Resistivity Results for Neat Cycloaliphatic Epoxy	242
Table 8.114: Electrical Resistivity Results for 5 wt% Talc in Cycloaliphatic Epoxy ...	242
Table 8.115: Electrical Resistivity Results for 10 wt% Talc in Cycloaliphatic Epoxy .	243
Table 8.116: Electrical Resistivity Results for 15 wt% Talc in Cycloaliphatic Epoxy .	243
Table 8.117: Electrical Resistivity Results for 20 wt% Talc in Cycloaliphatic Epoxy .	244
Table 8.118: Electrical Resistivity Results for 5 wt% Glass Microspheres in Cycloaliphatic Epoxy.....	244
Table 8.119: Electrical Resistivity Results for 10 wt% Glass Microspheres in Cycloaliphatic Epoxy.....	245
Table 8.120: Electrical Resistivity Results for 20 wt% Glass Microspheres in Cycloaliphatic Epoxy.....	245
Table 8.121: Electrical Resistivity Results for 30 wt% Glass Microspheres in Cycloaliphatic Epoxy.....	246
Table 8.122: Electrical Resistivity Results for 40 wt% Glass Microspheres in Cycloaliphatic Epoxy.....	246
Table 8.123: Thermal Conductivity Results for Neat Cycloaliphatic Epoxy	247
Table 8.124: Thermal Conductivity Results for 5 wt% Talc in Cycloaliphatic Epoxy .	247
Table 8.125: Thermal Conductivity Results for 10 wt% Talc in Cycloaliphatic Epoxy	247
Table 8.126: Thermal Conductivity Results for 15 wt% Talc in Cycloaliphatic Epoxy	248
Table 8.127: Thermal Conductivity Results for 20 wt% Talc in Cycloaliphatic Epoxy	248
Table 8.128: Thermal Conductivity Results for 5 wt% Glass Microspheres in Cycloaliphatic Epoxy.....	248
Table 8.129: Thermal Conductivity Results for 10 wt% Glass Microspheres in Cycloaliphatic Epoxy.....	249
Table 8.130: Thermal Conductivity Results for 20 wt% Glass Microspheres in Cycloaliphatic Epoxy.....	249
Table 8.131: Thermal Conductivity Results for 30 wt% Glass Microspheres in Cycloaliphatic Epoxy.....	249
Table 8.132: Thermal Conductivity Results for 40 wt% Glass Microspheres in Cycloaliphatic Epoxy.....	250

Table 8.133: Tensile Results for Neat Cycloaliphatic Epoxy.....	251
Table 8.134: Tensile Results for 5 wt% Talc in Cycloaliphatic Epoxy.....	252
Table 8.135: Tensile Results for 10 wt% Talc in Cycloaliphatic Epoxy.....	253
Table 8.136: Tensile Results for 15 wt% Talc in Cycloaliphatic Epoxy.....	254
Table 8.137: Tensile Results for 20 wt% Talc in Cycloaliphatic Epoxy.....	255
Table 8.138: Tensile Results for 5 wt% Glass Microspheres in Cycloaliphatic Epoxy	256
Table 8.139: Tensile Results for 10 wt% Glass Microspheres in Cycloaliphatic Epoxy	257
Table 8.140: Tensile Results for 20 wt% Glass Microspheres in Cycloaliphatic Epoxy	258
Table 8.141: Tensile Results for 30 wt% Glass Microspheres in Cycloaliphatic Epoxy	259
Table 8.142: Tensile Results for 40 wt% Glass Microspheres in Cycloaliphatic Epoxy	260
Table 8.143: Water Mass Gain Data for GNP/Epoxy Composites, ± 1 Standard Deviation	261
Table 8.144: Flexural Results for Neat Cycloaliphatic Epoxy Unaged.....	262
Table 8.145: Flexural Results for Neat Cycloaliphatic Epoxy aged 50 hours.....	263
Table 8.146: Flexural Results for Neat Cycloaliphatic Epoxy aged 100 hours.....	264
Table 8.147: Flexural Results for Neat Cycloaliphatic Epoxy aged 200 hours.....	265
Table 8.148: Flexural Results for Neat Cycloaliphatic Epoxy aged 400 hours.....	266
Table 8.149: Flexural Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Unaged	267
Table 8.150: Flexural Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 50 hours.....	268
Table 8.151: Flexural Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 100 hours.....	269
Table 8.152: Flexural Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 200 hours.....	270
Table 8.153: Flexural Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 400 hours.....	271
Table 8.154: Flexural Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Unaged	272
Table 8.155: Flexural Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Age 50 hour	273
Table 8.156: Flexural Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Age 100 hour	274
Table 8.157: Flexural Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Age 200 hour	275
Table 8.158: Flexural Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Age 400 hour	276
Table 8.159: DMA Results for Neat Cycloaliphatic Epoxy Unaged.....	277
Table 8.160: DMA Results for Neat Cycloaliphatic Epoxy Aged 50 Hours.....	277
Table 8.161: DMA Results for Neat Cycloaliphatic Epoxy Aged 100 Hours.....	277

Table 8.162: DMA Results for Neat Cycloaliphatic Epoxy Aged 200 Hours.....	278
Table 8.163: DMA Results for Neat Cycloaliphatic Epoxy Aged 400 Hours.....	278
Table 8.164 DMA Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Unaged..	280
Table 8.165 DMA Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 50 Hours.....	281
Table 8.166 DMA Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 100 Hours.....	281
Table 8.167 DMA Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 200 Hours.....	281
Table 8.168 DMA Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 400 Hours.....	282
Table 8.169 DMA Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Unaged..	284
Table 8.170 DMA Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 50 Hours.....	284
Table 8.171 DMA Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 100 Hours.....	284
Table 8.172 DMA Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 200 Hours.....	285
Table 8.173 DMA Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 400 Hours.....	285
Table 8.174: Water Mass Gain Data for Talc/Epoxy Composites, ± 1 Standard Deviation	287
Table 8.175: Flexural Results for Neat Cycloaliphatic Epoxy Unaged.....	288
Table 8.176: Flexural Results for Neat Cycloaliphatic Epoxy aged 50 hours.....	289
Table 8.177: Flexural Results for Neat Cycloaliphatic Epoxy aged 100 hours.....	290
Table 8.178: Flexural Results for Neat Cycloaliphatic Epoxy aged 200 hours.....	291
Table 8.179: Flexural Results for Neat Cycloaliphatic Epoxy aged 400 hours.....	292
Table 8.180: Flexural Results for 10% Talc in Cycloaliphatic Epoxy Unaged.....	293
Table 8.181: Flexural Results for 10% Talc in Cycloaliphatic Epoxy Aged 50 hours..	294
Table 8.182: Flexural Results for 10% Talc in Cycloaliphatic Epoxy Aged 100 hours	295
Table 8.183: Flexural Results for 10% Talc in Cycloaliphatic Epoxy Aged 200 hours	296
Table 8.184: Flexural Results for 10% Talc in Cycloaliphatic Epoxy Aged 400 hours	297
Table 8.185: Flexural Results for 20% Talc in Cycloaliphatic Epoxy Unaged.....	298
Table 8.186: Flexural Results for 20% Talc in Cycloaliphatic Epoxy Age 50 hour	299
Table 8.187: Flexural Results for 20% Talc in Cycloaliphatic Epoxy Age 100 hour ...	300
Table 8.188: Flexural Results for 20% Talc in Cycloaliphatic Epoxy Age 200 hour ...	301
Table 8.189: Flexural Results for 20% Talc in Cycloaliphatic Epoxy Age 400 hour ...	302
Table 8.190: DMA Results for Neat Cycloaliphatic Epoxy Unaged.....	303
Table 8.191: DMA Results for Neat Cycloaliphatic Epoxy Aged 50 Hours.....	303
Table 8.192: DMA Results for Neat Cycloaliphatic Epoxy Aged 100 Hours.....	303
Table 8.193: DMA Results for Neat Cycloaliphatic Epoxy Aged 200 Hours.....	304
Table 8.194: DMA Results for Neat Cycloaliphatic Epoxy Aged 398 Hours.....	304
Table 8.195: DMA Results for 10 wt% Talc in Cycloaliphatic Epoxy Unaged.....	304
Table 8.196 DMA Results for 10 wt% Talc in Cycloaliphatic Epoxy Aged 50 Hours.	305

Table 8.197: DMA Results for 10 wt% Talc in Cycloaliphatic Epoxy Aged 100 Hours	305
Table 8.198: DMA Results for 10 wt% Talc in Cycloaliphatic Epoxy Aged 200 Hours	305
Table 8.199: DMA Results for 10 wt% Talc in Cycloaliphatic Epoxy Aged 398 Hours	306
Table 8.200: DMA Results for 20 wt% Talc in Cycloaliphatic Epoxy Unaged.....	306
Table 8.201: DMA Results for 20 wt% Talc in Cycloaliphatic Epoxy Aged 50 Hours	306
Table 8.202: DMA Results for 20 wt% Talc in Cycloaliphatic Epoxy Aged 100 Hours	307
Table 8.203: DMA Results for 20 wt% Talc in Cycloaliphatic Epoxy Aged 200 Hours	307
Table 8.204: DMA Results for 20 wt% Talc in Cycloaliphatic Epoxy Aged 398 Hours	307

Preface

The work contained in this dissertation was conducted in the department of Chemical Engineering at Michigan Technological University from May 2015 to August 2018. This dissertation is a compilation of published journal articles, and a journal article submitted for publication.

“Effects of Carbon Fillers on the Conductivity and Tensile Properties of Polyetheretherketone (PEEK) Composites” was published in *Polymer Composites* in 2016. This article was slightly modified and used as Chapter 2. PEEK composite extrusion and injection molding was performed by Dr. Julia King (1st author) and Tim Gasperich. Dr. Todd King (6th author) and Dr. John Sutherland (7th author) assisted in data analysis.

“Electrical, Thermal, and Tensile Properties of Cycloaliphatic Epoxy/Carbon Black and Cycloaliphatic Epoxy/Fumed Silica Nanocomposites” was published in the conference proceedings for 2018 AIAA (American Institute of Aeronautics and Astronautics) SciTech Conference. This article was slightly modified and used as Chapter 3. The data for the neat epoxy, CB2, and CB3 was obtained by Danielle Klimek-McDonald (3rd author).

“Thermal, Electrical, and Mechanical Properties of Talc- and Glass Microsphere-Reinforced Cycloaliphatic Epoxy Composites” was published in *Polymer Composites* in 2017. This article was slightly modified and used as Chapter 4.

“Accelerated Hydrothermal Aging of Cycloaliphatic Epoxy/Graphene Nanoparticle Composites” was published in *Polymer Degradation and Stability* in 2016. This article was slightly modified and used as Chapter 5.

“Accelerated Hydrothermal Aging of Talc/Cycloaliphatic Epoxy Composites” was submitted to *Polymer Composites* in January 2018. This article was slightly modified and used as Chapter 6.

All experiments and measurements discussed within this dissertation were performed by Julie Tomasi with the assistance of the undergraduate researchers listed in the acknowledgements section of each chapter, and several co-authors of these papers: Danielle Klimek-McDonald, Aaron Krieg, Will Pisani, Sorayot Chinkanjanarot, Nate Herline, Ian Helman, and Nick Jensen.

Additional contributions included writing formatting, journal article submissions and technical advice from Dr. Julia King and Dr. Gregory Odegard. Dr. Ibrahim Miskioglu also provided guidance and technical advice throughout this project.

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Thank you to my fiancé, Wil Jakeway, for his patience while I finish my degree.

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So many more people have kept me going and learning that I do not think I can name them all. Thank you.

Abstract

Composites have many applications due to their high strength to weight ratio, chemical resistance, and the ability to tune properties like electrical resistivity (ER). In this work, polyetheretherketone (PEEK) and cycloaliphatic epoxy (CE) composites with different fillers were studied. PEEK composites contained up to 10 wt% Akzo Nobel Ketjenblack carbon black (CB), up to 15 wt% Asbury Carbons TC307 graphene nanoplatelets (GNP), up to 30 wt% Solvay carbon fiber (CF), and combinations of the three carbons.

Formulations were developed for electrostatic dissipative and electrically conductive applications. The most conductive formulation was 7.5 wt% CB/30 wt% CF/PEEK, which had an electrical resistivity of 0.56 ohm-cm, a thermal conductivity twice that of the neat PEEK, a tensile strength of 145 MPa, and a tensile modulus of 18.4 GPa.

CE composites have high voltage and temperature applications such as aluminum conductor composite core (ACCC) power transmission lines because of their high ER, high glass transition temperature (T_g), stiffness, and aging resistance. Cabot CB and fumed silica were tested in CE. The results showed that up to 2 wt% carbon black or 4 wt% fumed silica had little effect on the electrical, thermal, or tensile properties of CE. Asbury TC307 GNP was added to CE and aged at elevated temperature and moisture levels. The results from flexure and dynamic mechanical testing indicate that the addition of up to 4 wt% GNP provides modest improvements in the stiffness and T_g for all aging levels, while the strength is improved for aging times below 400 hours. Imerys Nicron talc and Potters Spherglass glass microspheres (glass MS) were tested in CE.

Thermal conductivity increased from 0.15 W/m-K for neat CE to 0.25 W/m-K for both 20 wt% talc/CE and 40 wt% glass MS/CE. Tensile modulus increased for both fillers, and compared well with predicted values from micromechanical models. The talc/CE composites were aged at elevated temperature and moisture levels. Talc reduced water absorption and did not negatively affect aging effects on flexural strength and stiffness. Prior to these experiments, properties of these composites had not been previously reported in the open literature.

1. Introduction

1.1 Introduction and Motivation

Polymer composites have many applications due to their high specific strength, chemical resistance, and the ability to tune properties by changing the type and amount of fillers. Many composite materials need to withstand high temperatures, humidity, and stresses over long lifetimes. It is important to understand how different fillers change the composite properties in order to select the right composite for an application.

This work was funded through the National Science Foundation (NSF) Industry/University Cooperative Research Center (I/UCRC) for Novel High Voltage/Temperature Materials and Structures. The work in this dissertation focuses on two separate applications and composite materials within the I/UCRC: A polyetheretherketone (PEEK) based composite is studied for aerospace applications, and a cycloaliphatic epoxy (CE) based composite is studied for power transmission applications. The PEEK composites are discussed in Chapter 2, and the CE composites are discussed in Chapters 3 to 6. Each chapter is a journal article with minor changes and additions.

1.1.1 Aerospace Introduction

Polymer composites are often used in aerospace applications. One well-known example is the Boeing 787 Dreamliner, which is 50% composite by weight [1]. Composites are useful for aerospace applications because they are lightweight, which cuts down on fuel needs, and they are corrosion resistant, which cuts down on maintenance cost. High stiffness and strength are desirable for aerospace applications. A high thermal conductivity allows a composite to serve as a heat sink. Electrically conductive composites can be used for electrostatic dissipative (between 10^3 and 10^{10} ohm-cm), semiconductive (between 10^1 and 10^2 ohm-cm), and electromagnetic and radio frequency interference (below 10^0 ohm-cm) applications.

PEEK was chosen as the polymer for the aerospace work due to its high chemical resistance, wear resistance, and operating temperatures up to 250 °C [2]. The fillers chosen are carbon black (CB), graphene nanoplatelets (GNP), and chopped carbon fiber (CF). All three are conductive carbon fillers that increase the electrical and thermal conductivities of a composite. The carbon black used here has a highly branched structure (surface area = 1250 m²/g), which increases electrical conductivity at lower concentrations [3]. Graphene nanoplatelets are short stacks of graphene layers that have been shown to increase composite stiffness and electrical conductivity [4]. Carbon fiber greatly improves composite strength and stiffness in the fiber direction. The PEEK composites designed for aerospace components in this work maximize electrical conductivity, thermal conductivity, and tensile modulus. Electrically resistive

composites were also found, which could be useful in other aerospace applications, depending on the component target values. A more extensive introduction can be found in Chapter 2. The following PEEK composites are discussed in this document:

- GNP: 2 (1.3), 4 (2.6), 6 (4.0), 8 (5.3), 10 (6.7), 12.5 (8.5), 15 (10.3) wt% (vol%)
- CF: 10 (7.4), 20 (15.3), 30 (23.6) wt% (vol%)
- CB: 2.5 (1.8), 5 (3.7), 7.5 (5.5), 10 (7.4) wt% (vol%)
- CB/CF combinations:
 - 2.5 wt% CB/10 wt% CF (1.9 vol% CB/7.5 vol% CF)
 - 2.5 wt% CB/20 wt% CF (1.9 vol% CB/15.4 vol% CF)
 - 2.5 wt% CB/30 wt% CF (2.0 vol% CB/23.8 vol% CF)
 - 5.0 wt% CB/30 wt% CF (4.0 vol% CB/24.0 vol% CF)
 - 7.5 wt% CB/30 wt% CF (6.1 vol% CB/24.2 vol% CF)

1.1.2 Power Transmission Line Introduction

Globally, power demands have risen, and utility companies aim to transmit more electricity over existing right-of-ways. To reach this goal, new power transmission lines have been developed. Traditionally, a line consists of an aluminum conductor and steel reinforcement (ACSR), where the aluminum transmits electricity and the steel core supports the line. The new aluminum conductor composite core (ACCC) lines are

supported by a composite fiber core. The current design consists of an inner core of continuous carbon fiber and epoxy that primarily supports the line, and a thin outer core of continuous glass fiber and epoxy that electrically insulates the inner core [5-9]. The stronger, lighter composite core can fully support the transmission line, which means that an aluminum conductor with a lower mechanical strength and corresponding lower electrical resistance than the aluminum in traditional ACSR lines can be used [8]. The lower resistance aluminum decreases power losses in the transmission line. The composite core also allows for a higher operating temperature (around 180 °C) than the steel core operating temperature (around 100 °C), which means that more electricity can be transmitted over the same diameter line [9]. At high temperatures, steel expands and ACSR lines sag and potentially break, but the ACCC lines have a much lower coefficient of thermal expansion (less sag at high temperatures), allowing a higher operating temperature [9].

Currently, ACCC lines are made without any particulate filler in either the inner carbon fiber/epoxy core, or the outer glass fiber/epoxy layer. In this work, we will investigate adding carbon black, fumed silica, talc, glass microspheres, and graphene nanoplatelet particulates to the epoxy matrix that can be used in the inner carbon fiber core or the outer glass fiber layer on the composite core. The addition of particulates is expected to provide composite properties that are more favorable than the neat epoxy. The inner carbon fiber/epoxy core and the outer glass fiber/epoxy layer in the ACCC core both need to have a low electrical conductivity (high resistivity) so that it does not degrade

during normal operation of the transmission line. A higher glass transition temperature (T_g) would allow a higher operating temperature, so the same diameter line could transmit more electricity. High thermal conductivity would reduce the presence of local hot spots that could degrade the epoxy. High modulus and resistance to aging means fewer downed lines and replacement costs.

The epoxy chosen for this application has a high T_g and is resistant to ozone and ultraviolet aging mechanisms [6]. The fillers used with the epoxy are carbon black, fumed silica, graphene nanoplatelets, talc, and glass microspheres (MS). Carbon black and graphene nanoplatelets are electrically conductive carbon fillers, so they will be used in low amounts to keep the composites electrically resistive. Fumed silica, talc, and glass MS are all electrically resistive. Fumed silica has a similar branched structure to carbon black, but it is not electrically conductive. The talc is a flaky, microcrystalline mineral that is 98% pure, and has an average particle size of 6 μm [10]. The glass microspheres used here are solid, coated to improve bonding, and have an average diameter of 11 μm [11]. All five fillers were chosen because they increase composite stiffness [12-22]. Some have also shown an increase in aging resistance [19, 23, 24]. The epoxy composites designed for transmission line composite cores in this work maximize electrical resistivity, tensile modulus, glass transition temperature, and resistance to hygrothermal aging. More detailed background information and introductions can be found in the introduction sections of Chapters 3 to 6. The following epoxy composites are discussed in this document:

- Carbon Black (CB):
 - CB1 (low surface area): 0.5 (0.33), 1 (0.67), 2 (1.34) wt% (vol%)
 - CB2 (mid surface area): 1 (0.67) wt% (vol%)
 - CB3 (high surface area): 1 (0.67) wt% (vol%)
- Fumed Silica: 1 (0.55), 2 (1.1), 3 (1.65), 4 (2.2) wt% (vol%)
- GNP: 2 (1.2), 4 (2.4) wt% (vol%)
- Talc: 5 (2.2), 10 (4.6), 15 (7.1), 20 (9.8) wt% (vol%)
- Glass MS: 5 (2.5), 10 (5.1), 20 (10.9), 30 (17.3), 40 (24.5) wt% (vol%)

1.2 Objectives

There were two main goals for this work, which are listed and explained below:

1. Characterize electrically conductive thermoplastic composites with various fillers for structural aerospace applications.
 - Extrude and injection mold polyetheretherketone (PEEK) composites with graphene nanoplatelet (GNP), carbon black (CB), and chopped carbon fiber (CF) fillers.
 - Test tensile, thermal, and electrical properties of the PEEK composites.
2. Characterize electrically resistive epoxy composites with various fillers for power transmission line support applications.
 - Develop fabrication methods for epoxy composites with carbon black (CB), fumed silica, talc, and glass microsphere (glass MS) fillers.

- Test tensile, thermal, and electrical properties of the epoxy composites.
- Investigate filler and aging effects on GNP/epoxy and talc/epoxy composites that have been artificially aged at 50 °C and 100 % relative humidity.
- Test flexural properties and glass transition temperature of aged composites.

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2. Effects of Carbon Fillers on the Conductivity and Tensile Properties of Polyetheretherketone (PEEK) Composites¹

2.1 Abstract

Polyetheretherketone (PEEK) based composites are used in aerospace applications due to their high mechanical strengths at temperatures $>250^{\circ}\text{C}$, excellent chemical resistance properties, and outstanding dimensional stability. In this work, varying amounts of three different carbon fillers [carbon black (CB), graphene nanoplatelets (GNP), and carbon fiber (CF)] were added to PEEK to produce conductive composites for aerospace applications. Researchers extruded, injection molded, and performed electrical and thermal conductivity and tensile tests on GNP/PEEK, CB/PEEK, CF/PEEK, and CB/CF/PEEK composites. Five formulations could be used for electrostatic dissipative applications: 15 wt% GNP/PEEK, 20 wt% CF/PEEK, 2.5 wt% CB/10 wt% CF/PEEK, and 2.5 wt% CB/20 wt% CF/PEEK. Seven formulations could be used for electrically conductive applications: 30 wt% CF/PEEK, 5 wt% CB/PEEK, 7.5 wt% CB/PEEK, 10 wt% CB/PEEK, 2.5 wt% CB/30 wt% CF/PEEK, 5 wt% CB/30 wt% CF/PEEK, 7.5 wt% CB/30 wt% CF/PEEK. The most conductive composite produced was 7.5 wt% CB/30 wt% CF/PEEK, which had an electrical resistivity (inverse electrical conductivity) of

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0.56 ohm-cm, a thermal conductivity twice that of the neat PEEK, a tensile strength of 145 MPa, and a tensile modulus of 18.4 GPa.

2.2 Introduction

Composites capable of withstanding higher temperatures ($>250\text{ }^{\circ}\text{C}$) are often needed for aerospace applications. Polyetheretherketone (PEEK) is a semi-crystalline thermoplastic polymer that is often used in these higher temperature applications since it has a melt temperature of about $340\text{ }^{\circ}\text{C}$. PEEK based composites also have excellent chemical resistance properties, high mechanical strengths at temperatures greater than $250\text{ }^{\circ}\text{C}$, and low moisture absorption, which helps provide outstanding dimensional stability. For these reasons, PEEK was chosen as the polymer for this work to produce conductive composites. Three different conductive carbon fillers were used. Graphene nanoplatelets (GNP) are short stacks of individual layers of graphite (graphene) that are available at a lower cost ($\sim\$15\text{-}\$50/\text{lb}$) as compared to carbon nanotubes [1-5]. Highly structured carbon black (CB) is a relatively inexpensive filler ($\sim\$10/\text{lb}$) that is often used to increase the composite conductivity [6]. Carbon fibers (CF, $\sim \$15/\text{lb}$) are often used to increase the tensile and conductivity properties of a composite [7, 8]. Thermally conductive composites can be used for heat sink applications. Electrically conductive composites can be used for electrostatic dissipative [ESD, Electrical Resistivity (ER) $\sim 10^{10}$ to 10^3 ohm-cm], semiconductive (ER $\sim 10^2$ to 10^1 ohm-cm), and electromagnetic and radio frequency interference applications (ER $<10^0\text{ ohm-cm}$). This study focuses on

fabricating, testing, and analyzing the tensile and conductivity properties of carbon filled/PEEK composites that could be used in conductive aerospace applications.

In this work, researchers performed extrusion followed by injection molding of GNP/PEEK, CF/PEEK, CB/PEEK and CB/CF/PEEK composites. Composites containing varying amounts of GNP, CB, and CF were fabricated and tested for tensile properties in addition to thermal and electrical conductivity (inverse electrical resistivity). The first objective of this work is to determine the effects of these fillers on composite thermal and electrical conductivity and tensile properties. The second objective is to determine the electrical percolation threshold of each filler type. Per the authors' knowledge, properties on these GNP/PEEK, CB/PEEK, and CB/CF/PEEK composites have not been reported previously in the open literature.

2.3 Materials and Experimental Methods

2.3.1 Materials

In this study, Solvay's KetaSpire KT-880 NT polyetheretherketone (PEEK) pellets (with 0.01 wt% calcium stearate lubricant to aid in conveying the pellets in processing) were used. KetaSpire KT-880 NT is a semi-crystalline thermoplastic with a glass transition temperature (T_g) of 147 °C, melting point (T_m) of 343°C, melt flow index of 36 g/10 min, and density of 1.3 g/cc [9]. In addition, KetaSpire KT-880 CF30 pellets, which contain 30 wt% carbon fiber (CF) in this same PEEK, were used.

One graphene nanoplatelet (GNP), Asbury Carbons' TC307 GNP, was used. TC307 has an average particle diameter less than 1 μm , a surface area of 350 m^2/g , a density of 2 g/cc , and consists of about 8 layers of graphene [1].

One electrically conductive carbon black (CB), specifically Akzo Nobel's Ketjenblack EC-600 JD, was used. The highly branched, high surface area (1250 m^2/g) carbon black structure allows it to produce electrically conductive composites at low carbon black concentrations. The density of this carbon black is 1.8 g/cc . The carbon black is sold in the form of pellets that are 100 μm to 2 mm in size and, upon mixing into a polymer, easily separate into primary aggregates 30 to 100 nm long [10].

The concentrations (shown in wt% and the corresponding vol%) for composites tested in this research are shown in Table 2.1. Table 2.1 also shows all the thermal conductivity and electrical resistivity results that will be discussed later in this chapter.

Table 2.1: Filler loading levels in PEEK along with electrical resistivity and thermal conductivity results

Formulation	Filler wt%/(vol%)	Electrical Resistivity ohm-cm	Through-Plane Thermal Conductivity W/m ² K
PEEK	0.0/(0.0)	$2.81 \times 10^{17} \pm 2.45 \times 10^{16}$ n = 8	0.254 ± 0.004 n = 8
2GNP	2.0/(1.31)	$2.34 \times 10^{17} \pm 7.59 \times 10^{15}$ n = 6	0.273 ± 0.001 n = 4
4GNP	4.0/(2.64)	$7.73 \times 10^{16} \pm 2.21 \times 10^{15}$ n = 6	0.286 ± 0.001 n = 4
6GNP	6.0/(3.98)	$4.81 \times 10^{16} \pm 4.56 \times 10^{15}$ n = 6	0.305 ± 0.001 n = 4
8GNP	8.0/(5.35)	$2.86 \times 10^{16} \pm 1.70 \times 10^{15}$ n = 6	0.323 ± 0.004 n = 4
10GNP	10.0/(6.74)	$1.45 \times 10^{16} \pm 9.30 \times 10^{14}$ n = 6	0.348 ± 0.004 n = 4
12.5GNP	12.5/(8.50)	$9.78 \times 10^{13} \pm 5.09 \times 10^{13}$ n = 6	0.376 ± 0.008 n = 5
15GNP	15.0/(10.29)	$2.96 \times 10^7 \pm 2.51 \times 10^6$ n = 5	0.427 ± 0.001 n = 4
10CF	10.0/(7.43)	$1.25 \times 10^{16} \pm 8.75 \times 10^{14}$ n = 6	0.284 ± 0.006 n = 6
20CF	20.0/(15.30)	$1.98 \times 10^7 \pm 9.29 \times 10^6$ n = 5	0.338 ± 0.007 n = 5
30CF	30.0/(23.64)	32.02 ± 6.33 n = 8	0.415 ± 0.015 n = 8
2.5 CB	2.5/(1.82)	$1.92 \times 10^{15} \pm 2.40 \times 10^{14}$ n = 8	0.279 ± 0.003 n = 8
5 CB	5.0/(3.66)	30.09 ± 4.82 n = 8	0.307 ± 0.004 n = 8
7.5CB	7.5/(5.53)	6.85 ± 0.22 n = 9	0.328 ± 0.005 n = 8
10CB	10/(7.43)	2.81 ± 0.07 n = 10	0.353 ± 0.007 n = 4
2.5 CB/30CF	2.5/(1.98) CB 30.0/(23.82) CF	27.85 ± 0.62 n = 9	0.427 ± 0.005 n = 8
5 CB/30CF	5.0/(4.00) CB 30.0/(24.00) CF	2.01 ± 0.15 n = 10	0.473 ± 0.008 n = 8
7.5 CB/30CF	7.5/(6.05) CB 30.0/(24.19) CF	0.56 ± 0.04 n = 10	0.515 ± 0.008 n = 8
2.5CB/10 CF	2.5/(1.87) CB 10.0/(7.48) CF	$1.07 \times 10^6 \pm 8.17 \times 10^5$ n = 5	0.314 ± 0.008 n = 6
2.5CB/20 CF	2.5/(1.93) CB 20.0/(15.41) CF	$7.28 \times 10^3 \pm 5.34 \times 10^2$ n = 5	0.369 ± 0.004 n = 5

2.3.2 Test Specimen Fabrication

Prior to extrusion and injection molding, the KetaSpire KT-880 NT and KetaSpire KT-880 CF30 were dried in an indirect heated dehumidifying drying oven at 150 °C for 4 hours. Ketjenblack EC-600 JD and Asbury TC307 were used as received. The extruder

used was a 27 mm co-rotating intermeshing twin screw extruder from American Leistritz Extruder Corporation (Model ZSE 27). This extruder has 10 heating zones and a length/diameter ratio of 40. Two different extruder screw designs were used. Figure 2.1 shows the screw design used for the GNP/PEEK composites. The PEEK pellets and GNP were added at the main feed (zone 1) using 2 different gravimetric feeders. The screw design used for the composites containing CF and CB has been shown elsewhere [11]. In this screw design, the KetaSpire KT-880 NT and KT-880 CF30 pellets were added at the main feed port (zone 1) and CB was added at the zone 5 side stuffer. A K-Tron KCL-SFS-KT-20 twin screw gravimetric feeder with a 2 blade agitator was used to feed the GNP and CB. In order to extrude at the high temperatures needed for PEEK (~375 °C), compressed air was used for cooling on the extrusion zones 2 to 9.

Figure 2.1: Extruder screw design used for TC307 GNP/PEEK composites

After passing through the extruder, the polymer strands (3 mm diameter) entered a water bath and then a pelletizer that produced nominally 3 mm long pellets. After extrusion,

the composites were dried in an indirect heated dehumidifying drying oven at 150 °C for 4 hours and then stored in moisture barrier bags prior to injection molding.

A Niigata injection molding machine, model NE85UA₄, was used to produce test specimens. This machine has a 40 mm diameter single screw with a length/diameter ratio of 18. The lengths of the feed, compression, and metering sections of the single screw are 396 mm, 180 mm, and 144 mm, respectively. An additional heater (near the nozzle end, farthest from feed end) and insulation blankets were added to this Niigata injection molding machine to maintain the high temperatures needed to injection mold PEEK. A four cavity mold was used to produce 3.3 mm thick ASTM Type I tensile bars (end gated) and 3.3 mm thick, 6.4 cm diameter disks (end gated). Appendix 8.1 contains detailed extrusion and injection molding conditions and notes for each formulation.

2.3.3 Field Emission Electron Microscope (FESEM) Test Method

A Hitachi S-4700 Field Emission Scanning Electron Microscope (FESEM) was used to view the GNP/PEEK and CF/PEEK composites. Micrographs were collected when using a 2 kV accelerating voltage with a working distance of 10.8 mm for the GNP/PEEK and 12.9 mm for the CF/PEEK composites. The upper secondary electron detector was used to collect the images of the fracture tensile surface. The samples were cut to 3 mm x 3 mm x 3 mm using a scroll saw, glued to sample studs, and coated with carbon paint. The fracture surface was sputter coated with 1.5 nm thick platinum using an Anatech

Hummer 6.2 Sputtering System for GNP/PEEK composites. For the CF/PEEK composites, the fracture surface was sputter coated with 1.5 nm thick platinum/palladium using a Cressington 208HR Sputtering System.

2.3.4 Carbon Fiber Length Test Method

To determine the length of the carbon fiber in the PEEK based composite, a 0.2 g sample cut from a tensile bar was dissolved in boiling concentrated (70%) nitric acid. The carbon fiber was then filtered, dried, and dispersed on a glass slide, and viewed using an Olympus BX60 optical microscope with reflected light at 50x magnification. An Olympus DP-10 digital camera was used to collect the images, which were analyzed using ImageJ software. Because of the extremely small size of the CB and GNP, the length of these fillers were not measured.

2.3.5 Electrical Resistivity (ER) Test Method

Prior to any electrical resistivity testing, the samples were conditioned at 23 °C and 50% relative humidity for 2 days. ASTM D257 was used to measure the volumetric electrical resistivity for samples with an electrical resistivity $>10^8$ ohm-cm [12]. A constant voltage (100 V) was applied to the as-molded test specimen, and the resistivity was measured using a Keithley 6517A Electrometer/High Resistance Meter and an 8009 Resistivity Test Fixture. The Keithley 6524 High Resistance Measurement Software was used to automate the conductivity measurement. Each test specimen was an injection

molded disk that was 6.4 cm in diameter and 3.3 mm thick. At least six samples were tested for each formulation.

For samples with an electrical resistivity $<10^8$ ohm-cm, the volumetric electrical resistivity of the center rectangular 60 mm of the 3.3 mm thick, 12.7 mm wide injection molded tensile bars was determined according to ASTM D 4496 at 23 °C [13] using this two probe method along with a Keithley 2400 Source Meter. In the two probe method, the tensile bar was scratched with a razor blade, placed in liquid nitrogen, and then broken manually at the desired location. At least five samples were tested for each formulation. Additional detailed test method information is shown elsewhere [14].

2.3.6 Thermal Conductivity (TC) Test Method

The through-plane thermal conductivity of a 3.3 mm thick, 5 cm diameter disk shaped test specimen was measured at 55 °C using a Holometrix Model TCA-300 Thermal Conductivity Analyzer, which uses the ASTM F433 guarded heat flow meter method [15]. The thermal conductivity was measured at 55 °C because this was as close to ambient temperature as could be measured, while still maintaining a temperature gradient in the analyzer. Prior to testing, the samples were conditioned at 23 °C and 50% relative humidity for 2 days.

2.3.7 Tensile Test Method

An Instru-Met Sintech screw driven mechanical testing machine was used to determine the tensile properties (at ambient conditions, 16.5 cm long, 3.3 mm thick, 12.7 mm wide ASTM Type I sample geometry) according to ASTM D638 at a crosshead rate of 5 mm/min [16]. Tensile modulus was calculated from the initial linear portion of the stress-strain curve. For each formulation, at least five samples were tested. Prior to testing, the samples were conditioned at 22 °C and 50% relative humidity for 2 days.

2.4 Results

2.4.1 Field Emission Scanning Electron Microscopy (FESEM) Results

Figure 2.2 shows a FESEM image of a tensile fracture surface containing 4 wt% TC307 GNP in PEEK. This figure clearly shows the GNP layered structure. Figure 2.3 shows a FESEM image of the tensile fracture surface of 10 wt% TC307 in PEEK at a higher magnification. This image shows PEEK microfibrils adhering well to the GNP flake. Figure 2.4 displays the tensile fracture surface of the 30 wt% CF/PEEK composite. This micrograph shows PEEK adhering well to the carbon fiber, clinging to the sides of the fibers instead of pulling away. It also shows the alignment of the CF in the longitudinal tensile direction caused during injection molding (end gated).

Figure 2.2: FESEM micrograph of composite containing 4 wt% TC307 in PEEK

Figure 2.3: FESEM micrograph of composite containing 10 wt% TC307 in PEEK

Figure 2.4: FESEM micrograph of composite containing 30 wt% carbon fiber in PEEK

2.4.2 Carbon Fiber Length Results

For 10CF, 20CF, and 30CF, the mean length of the carbon fiber in the composites varied from 0.10 to 0.18 mm with >400 carbon fibers measured in each formulation and standard deviation was approximately half of the mean. The diameter of the carbon fiber is 7 μm.

2.4.3 Electrical Resistivity Results

Table 2.1 and Appendix 8.2 show the ER results for all the formulations. Initially, formulations containing varying amounts of each of the following single fillers were fabricated and tested.

TC307 GNP: 2.0, 4.0, 6.0, 8.0, 10.0, 12.5 and 15.0 wt%

CF: 10.0, 20.0, and 30.0 wt%

CB: 2.5, 5.0, 7.5, and 10.0 wt%

Filler concentrations above the values listed here became too viscous to extrude and injection mold into test specimens. Figure 2.5 shows all the ER data for the composites containing varying amounts of one filler type. At low filler loadings, the ER is similar to that of the neat PEEK polymer (2.81×10^{17} ohm-cm). At the electrical percolation threshold, the ER dramatically decreases over a narrow range of filler concentrations. At filler concentrations significantly above the percolation threshold, the ER decreases at a much slower rate and forms a plateau, such that further increases in conductive filler content do not appreciably reduce ER. This figure clearly shows that the electrical percolation threshold varies based on filler type. For TC307 GNP, the electrical percolation threshold is around 13 wt% (9 vol%) TC307. At the highest TC307 filler loading (15 wt% = 10.3 vol%), the ER was 2.96×10^7 ohm-cm, which could be used for electrostatic dissipative (ESD) applications. Similar ER values have been reported in the open literature for GNP composites in other thermoplastics. For example, XG Sciences' xGnP graphene nanoplatelets (5 μm average particle diameter with a thickness of 6 to 8

nm) in polycarbonate that were also extruded and injection molded had a percolation threshold of 7.4 wt% (4.6 vol%) [14].

Figure 2.5: Log(electrical resistivity in ohm-cm) vs. filler content for composites containing varying amounts of a single filler type for GNP/PEEK, CB/PEEK, and CF/PEEK

Figure 2.5 also indicates that for the CB/PEEK composites, the percolation threshold occurs around 3 wt% (~2 vol%), which is much lower than that of the GNP/PEEK or CF/PEEK composites. The CB/PEEK composites clearly show that increasing amounts of CB above the percolation threshold slowly decreases ER. At the highest CB concentration of 10 wt% CB (7.4 vol%), the ER was 2.8 ohm-cm, which is approximately 17 orders of magnitude lower than the neat PEEK. This same CB has

previously been studied in polycarbonate and an electrical percolation threshold of 3.6 wt% (2.4 vol%) and an ER of 20 ohm-cm for 10 wt% CB (6.9 vol%) was obtained [14]. Therefore, these CB/PEEK results are similar to the CB/polycarbonate composites. This low percolation threshold is likely due to the highly branched carbon black structure. For the CF/PEEK composites, the percolation threshold occurs between 10 wt% CF (7.4 vol%) and 20 wt% CF (15.3 vol%), which is similar to the GNP/PEEK composites. At the highest CF concentration of 30 wt% CF (23.6 vol%), the ER was 32 ohm-cm. Saleem et al. reported an electrical percolation threshold between 30 and 35 wt% carbon fiber in PEEK [17]. Clearly, the CF percolation threshold in our study occurs at a much lower amount of CF.

Since the composites containing CB had the lowest percolation threshold, the authors decided to investigate composites containing CB and CF in PEEK. (CF gave the highest tensile strength and modulus, which will be discussed later in this chapter.) Initially, composites containing 2.5 wt% CB/10 wt% CF/PEEK and 2.5 wt% CB/20 wt% CF/PEEK were produced and tested. Table 2.1 shows that as the CF content was increased from 0 to 10 and 20 wt% with 2.5 wt% CB, the ER decreased from 2.81×10^{17} to 1.07×10^6 to 7.28×10^3 ohm-cm. Since one objective was to develop electrically conductive composites, we fabricated the following 2 filler, 2 concentration level (2^2) factorial design formulations: 2.5 wt% CB/30 wt% CF/PEEK, 5 wt% CB/30 wt% CF/PEEK, and 7.5 wt% CB/30 wt% CF/PEEK that are shown in Table 2.2 (original and replicate with mean, standard deviation, and number of samples tested). Composites

containing 10 wt% CB along with any amount of CF were too viscous to extrude and injection mold into specimens. Table 2.1 and Table 2.2 show that the composite containing the lowest ER was recorded at 0.56 ohm-cm for the composite containing 7.5 wt% CB/30 wt% CF/PEEK.

Table 2.2: Electrical resistivity and thermal conductivity results for factorial design formulations

Formulations	Electrical Resistivity (ohm-cm)	Thermal Conductivity (W/m·K)
No filler (PEEK)		
Original	$2.67 \times 10^{17} \pm 2.73 \times 10^{16}$ n = 4	0.2525 ± 0.0046 n = 4
Replicate	$2.95 \times 10^{17} \pm 1.18 \times 10^{16}$ n = 4	0.2568 ± 0.0032 n = 4
2.5CB		
Original	$2.02 \times 10^{15} \pm 2.78 \times 10^{14}$ n = 4	0.2796 ± 0.0012 n = 4
Replicate	$1.82 \times 10^{15} \pm 1.70 \times 10^{14}$ n = 4	0.2784 ± 0.0037 n = 4
5CB		
Original	29.26 ± 4.64 n = 4	0.3066 ± 0.0059 n = 4
Replicate	30.91 ± 5.56 n = 4	0.3064 ± 0.0036 n = 4
7.5CB		
Original	7.00 ± 0.12 n = 5	0.3270 ± 0.0058 n = 4
Replicate	6.66 ± 0.14 n = 4	0.3294 ± 0.0049 n = 4
30 CF		
Original	29.35 ± 7.17 n = 4	0.4163 ± 0.0158 n = 4
Replicate	34.69 ± 4.81 n = 4	0.4127 ± 0.0170 n = 4
2.5CB*30CF		
Original	27.93 ± 0.78 n = 5	0.4265 ± 0.0056 n = 4
Replicate	27.75 ± 0.43 n = 4	0.4266 ± 0.0063 n = 4
5 CB*30CF		
Original	2.07 ± 0.10 n = 5	0.4768 ± 0.0075 n = 4
Replicate	1.96 ± 0.19 n = 5	0.4697 ± 0.0071 n = 4
7.5 CB*30 CF		
Original	0.559 ± 0.043 n = 5	0.5206 ± 0.0047 n = 4
Replicate	0.560 ± 0.038 n = 5	0.5092 ± 0.0081 n = 4

Minitab version 17 statistical software was used to evaluate six separate 2^2 factorial designs using the Log(ER in ohm-cm) as the response. For the six 2^2 factorial designs, the low and high level of each filler are listed here:

- 25. CB: 0 and 2.5 wt%; CF: 0 and 30 wt%
- 26. CB: 0 and 5.0 wt%; CF: 0 and 30 wt%
- 27. CB: 0 and 7.5 wt%; CF: 0 and 30 wt%
- 28. CB: 2.5 and 5.0 wt%; CF: 0 and 30 wt%
- 29. CB: 5.0 and 7.5 wt%; CF: 0 and 30 wt%
- 30. CB: 2.5 and 7.5 wt%; CF: 0 and 30 wt%

Table 2.3 displays the results showing the effects and p values. Large effects and $p < 0.05$ indicate that a factor (filler) has a statistically significant effect on composite ER at the 95% confidence level. Factorial designs are used to determine the effect of each filler and any possible interactions between two fillers on the Log(ER) by calculating the effect that each factor (filler) has as the wt% of a filler is increased. These effects are then used to rank which fillers and combinations of fillers produced a larger change in Log(ER) [18].

Table 2.3: 2² factorial results for Log(electrical resistivity, ohm-cm)

Term	Effect	t	p
CB: 0 & 2.5 wt% / CF: 0 & 30 wt%			
CB	-1.112	-46.02	0.000
CF	-14.891	-616.13	0.000
CB*CF	1.053	43.57	0.000
CB: 0 & 5 wt% / CF: 0 & 30 wt%			
CB	-8.585	-377.67	0.000
CF	-8.559	-376.54	0.000
CB*CF	7.385	324.89	0.000
CB: 0 & 7.5 wt% / CF: 0 & 30 wt%			
CB	-9.185	-421.29	0.000
CF	-8.515	-390.57	0.000
CB*CF	7.429	340.74	0.000
CB: 2.5 & 5 wt% / CF: 0 & 30 wt%			
CB	-7.473	-517.28	0.000
CF	-7.506	-519.60	0.000
CB*CF	6.332	438.32	0.000
CB: 5 & 7.5 wt% / CF: 0 & 30 wt%			
CB	-0.600	-60.04	0.000
CF	-1.130	-113.09	0.000
CB*CF	0.044	4.38	0.012
CB: 2.5 & 7.5 wt% / CF: 0 & 30 wt%			
CB	-8.073	-624.08	0.000
CF	-7.462	-576.89	0.000
CB*CF	6.376	492.90	0.000

Table 2.3 gives some important information regarding the effects that fillers have on the Log(ER). In all six factorial designs, Table 2.3 shows that CB and CF cause a statistically significant decrease in Log(ER) (because the effect is negative and $p < 0.05$). Also, in all cases, the CB*CF interaction effect term is statistically significant ($p < 0.05$) and positive. Therefore, when some CF is present in a composite, then the effect of the CB on the Log(ER) is diminished and vice versa.

The effect terms in Table 2.3 describe the slope of the Log(ER). For example, for the 2 filler, 2 level factorial design with 0 and 2.5 wt% CB and 0 and 30 wt% CF, the CB effect term is much smaller (at -1.1) than the CF effect term (at -14.9) since going from 0 to 2.5 wt% CB only caused the Log(ER) to decrease from 17.5 (2.81×10^{17} ohm-cm) to 15.3 (1.92×10^{15} ohm-cm), whereas going from 0 to 30 wt% CF caused the Log(ER) to decrease from 17.5 (2.81×10^{17} ohm-cm) to 1.4 (32 ohm-cm). For the factorial design with 0 and 5 wt% CB and 0 and 30 wt% CF, the CB and CF effect terms are approximately the same (at -8.6), since going from 0 to 5 wt% CB and from 0 to 30 wt% CF caused the Log(ER) to decrease from 17.5 (2.81×10^{17} ohm-cm) to 1.5 (~30 ohm-cm) in both cases. For the factorial design with 5 and 7.5 wt% CB and 0 and 30 wt% CF, the CB and CF effect terms are all lower (-0.6 for CB and -1.1 for CF) since all the Log(ER) values for these 4 composites only vary between -0.3 (0.56 ohm-cm for 7.5CB/30CF/PEEK) and 1.5 (30 ohm-cm for 5CB/PEEK).

The Log(ER) data from Table 2.1 for all 13 CB/PEEK, CF/PEEK, and CB/CF/PEEK formulations produced (shown as dots) are also shown as a contour plot in Figure 2.6. Several different regions are evident in Figure 2.6. The polymer's electrically insulating properties dominate near the origin of the graph where the Log(ER) varies from 17.5 to 12. At the electrically conductive region, when 30 wt% CF or ≥ 5 wt% CB is in a

composite, the $\text{Log}(\text{ER})$ is <2 in all cases. The transition region occurs between $\text{Log}(\text{ER})$ values of 3.9 to 7.

Figure 2.6: Log(electrical resistivity in ohm-cm) contour plot for all PEEK-based formulations

2.4.4 Thermal Conductivity (TC) Results

Table 2.1 and Appendix 8.3 show the TC results for all the formulations. Figure 2.7 shows all the TC data (error bars showing $\pm$ one standard deviation) for the composites containing varying amounts of one filler type. This graph shows that adding similar

amounts of TC307 GNP or CB to PEEK resulted in similar composite TC. For the composite containing 15 wt% (10.3 vol%) GNP in PEEK, the TC increased from 0.25 W/m-K for the neat PEEK to 0.43 W/m-K. Equation (2.1) shows the regression that fit the TC data for the GNP/PEEK composites where W_{GNP} is the wt% of GNP in the composite.

$$TC \left(\frac{W}{m \cdot K} \right) = 0.25703 + 0.00535 \cdot W_{GNP} + 0.000378 \cdot W_{GNP}^2 \quad R^2 = 0.995 \quad (2.1)$$

Similar TC values have been reported in the open literature for XG Sciences' xGnP graphene nanoplatelets (5 micron average particle diameter with a thickness of 6 to 8 nm) in polycarbonate which had a TC of 0.5 W/m-K for 15 wt% GNP (9.6 vol%) compared to 0.21 W/m-K for neat polycarbonate [19].

For the composite containing 10 wt% (7.4 vol%) CB in PEEK, the TC increased from 0.25 W/m-K for the neat PEEK to 0.35 W/m-K. Equation (2.2) shows the regression that fit the TC data for the CB/PEEK composites where W_{CB} is the wt% of CB in the composite.

$$TC \left(\frac{W}{m \cdot K} \right) = 0.2536 + 0.00992 \cdot W_{CB} \quad R^2 = 0.998 \quad (2.2)$$

The TC of this same CB has been reported in polycarbonate, and for the composite containing 10 wt% (6.9 vol%) CB, the TC increased from 0.21 for the neat polycarbonate to 0.29 W/m-K [19].

Figure 2.7: Thermal conductivity in W/m-K vs. filler content for composites containing varying amounts of a single filler type for GNP/PEEK, CB/PEEK, and CF/PEEK

For the CF/PEEK composites, the TC increases from 0.25 W/m-K for the neat PEEK to 0.42 W/m-K) for composites containing 30 wt% (23.6 vol%) CF in PEEK. Equation (2.3) shows the regression that fit the TC data for the CF/PEEK composites where W_{CF} is the wt% of CF in the composite.

$$TC \left(\frac{W}{m \cdot K} \right) = 0.2529 + 0.00204 \cdot W_{CF} + 0.00011 \cdot W_{CF}^2 \quad R^2 = 0.998 \quad (2.3)$$

Saleem et al. reported an increase in thermal conductivity from that of neat PEEK at 0.13 W/m-K to 0.18 W/m-K for composites containing 30 wt% CF in PEEK [17]. The TC increase reported in our study is higher.

Table 2.2 shows the TC results for all six of the 2 factor, 2 level factorial design formulations (original and replicate). Table 2.4 displays the results showing the effects and p values. In every case, CB and CF caused a statistically significant increase (positive effect term) in composite TC at the 95% confidence level ($p < 0.05$). In addition, in every case the CF effect term is larger than the CB effect term since the composite containing 30 wt% CF in PEEK had a TC of 0.42 W/m-K as compared to the highest TC value for the composite containing 7.5 wt% CB in PEEK of 0.33 W/m-K. For four of the six cases (CB: 0 and 7.5 wt% CF: 0 and 30 wt%, CB: 2.5 and 7.5 wt% CF: 0 and 30 wt%, CB: 5 and 7.5 wt% CF: 0 and 30 wt%, CB: 2.5 and 7.5 wt% CF: 0 and 30 wt%), the CB*CF interaction term was statistically significant and caused the composite TC to increase. This means that the composite TC is higher than what would be expected from the additive effect of each single filler. For the 2 filler, 2 level factorial design with 0 and 5 wt% CB and 0 and 30 wt% CF, the CB*CF interaction effect term is not statistically significant ($p > 0.05$). For the 2 filler, 2 level factorial design with 0 and 2.5 wt% CB and 0 and 30 wt% CF, the CB*CF interaction effect term is negative (causes TC to decrease) and is statistically significant ($p < 0.05$); however this CB*CF interaction effect term is 3 times smaller than the CB effect term and 25 times smaller than the CF effect term.

Table 2.4: 2² factorial results for thermal conductivity in W/m-K

Term	Effect	t	p
CB: 0 & 2.5 wt% / CF: 0 & 30 wt%			
CB	0.0182	12.69	0.000
CF	0.1537	107.19	0.000
CB*CF	-0.0061	-4.29	0.013
CB: 0 & 5 wt% / CF: 0 & 30 wt%			
CB	0.0553	24.44	0.000
CF	0.1633	72.18	0.000
CB*CF	0.0034	1.52	0.202
CB: 0 & 7.5 wt% / CF: 0 & 30 wt%			
CB	0.0870	26.91	0.000
CF	0.1733	53.61	0.000
CB*CF	0.0134	4.15	0.014
CB: 2.5 & 5 wt% / CF: 0 & 30 wt%			
CB	0.0371	20.60	0.000
CF	0.1571	87.26	0.000
CB*CF	0.0096	5.33	0.006
CB: 5 & 7.5 wt% / CF: 0 & 30 wt%			
CB	0.0317	9.29	0.001
CF	0.1767	51.81	0.000
CB*CF	0.0100	2.92	0.043
CB: 2.5 & 7.5 wt% / CF: 0 & 30 wt%			
CB	0.0688	23.49	0.000
CF	0.1671	57.08	0.000
CB*CF	0.0196	6.69	0.003

2.4.5 Tensile Results

Table 2.5 and Appendix 8.4 show the ultimate tensile strength, strain at ultimate tensile strength, and tensile modulus for all the composites fabricated in this study. Figure 2.8 shows the mean tensile modulus (error bars shown are $\pm$ one standard deviation) for all the composites containing varying amounts of a single filler type. For each filler, increasing the amount of a single type of filler causes the tensile modulus to increase. Due to the larger size of the carbon fiber in the composite (length of 0.1 mm to 0.18 mm as opposed to <0.001 mm for GNP and CB), the tensile modulus is much higher for the CF/PEEK composites. For example, the tensile modulus increases from 3.7 GPa for neat PEEK to 23.5 GPa for the composite containing 30 wt% (23.6 vol%) CF in PEEK. For carbon fiber/PEEK, Li and Zhang reported a tensile strength and modulus of 75 MPa and 3.5 GPa for 10 wt% CF/PEEK, and 95 MPa and 4.4 GPa for 20 wt% CF/PEEK, respectively [20]. Saleem et al. reported a tensile modulus of 0.68 GPa, 0.75 GPa, and 0.8 GPa for 10 wt% CF/PEEK, 20 wt% CF/PEEK, and 30 wt% CF/PEEK composites, respectively [17]. The values reported in our work are much higher (10 wt% CF/PEEK: tensile strength of 116 MPa and modulus of 6.2 GPa; 20 wt% CF/PEEK: tensile strength of 144 MPa and modulus of 10.7 GPa; 30 wt% CF/PEEK: tensile strength of 230 MPa and modulus of 23.5 GPa). The tensile modulus for the composite containing 15 wt% (10.3 vol%) GNP in PEEK was 4.9 GPa. The tensile modulus for the composite containing 10 wt% (7.4 vol%) CB in PEEK was 4.6 GPa.

Table 2.5: Tensile properties of all PEEK based formulations

Formulation	Ultimate Tensile Strength MPa	Strain at Ultimate Tensile Strength %	Tensile Modulus GPa
PEEK	95.4 ± 0.1 n = 6	4.85 ± 0.12 n = 6	3.70 ± 0.11 n = 6
2GNP	95.0 ± 0.5 n = 7	5.57 ± 0.22 n = 7	3.81 ± 0.07 n = 7
4GNP	95.4 ± 0.4 n = 5	5.40 ± 0.15 n = 5	3.97 ± 0.08 n = 5
6GNP	94.9 ± 0.6 n = 5	5.25 ± 0.12 n = 5	4.23 ± 0.04 n = 5
8GNP	95.4 ± 0.2 n = 5	5.22 ± 0.22 n = 5	4.38 ± 0.11 n = 5
10GNP	95.2 ± 0.4 n = 5	4.93 ± 0.11 n = 5	4.55 ± 0.05 n = 5
12.5GNP	90.0 ± 2.5 n = 6	2.70 ± 0.23 n = 6	4.81 ± 0.17 n = 6
15GNP	81.1 ± 2.7 n = 8	2.14 ± 0.10 n = 8	4.90 ± 0.20 n = 8
10CF	116.4 ± 1.8 n = 8	3.62 ± 0.55 n = 8	6.17 ± 0.31 n = 8
20CF	144.0 ± 0.4 n = 5	2.96 ± 0.15 n = 5	10.70 ± 0.49 n = 5
30CF	230.4 ± 2.3 n = 9	1.95 ± 0.07 n = 9	23.47 ± 0.73 n = 9
2.5 CB	89.2 ± 3.0 n = 8	3.08 ± 0.21 n = 8	4.06 ± 0.18 n = 8
5 CB	89.4 ± 2.9 n = 6	3.12 ± 0.41 n = 6	4.06 ± 0.19 n = 6
7.5CB	89.4 ± 3.1 n = 7	2.68 ± 0.14 n = 7	4.26 ± 0.12 n = 7
10CB	80.0 ± 4.3 n = 9	2.04 ± 0.19 n = 9	4.59 ± 0.13 n = 9
2.5 CB/30CF	165.0 ± 1.4 n = 11	1.92 ± 0.10 n = 11	16.63 ± 0.79 n = 11
5 CB/30CF	144.2 ± 0.9 n = 5	1.52 ± 0.03 n = 5	16.18 ± 0.50 n = 5
7.5 CB/30CF	144.9 ± 4.1 n = 6	1.29 ± 0.09 n = 6	18.42 ± 1.23 n = 6
2.5 CB/10 CF	117.6 ± 2.0 n = 10	3.57 ± 0.57 n = 10	6.47 ± 0.26 n = 10
2.5 CB/20 CF	145.2 ± 0.5 n = 8	2.72 ± 0.13 n = 8	11.00 ± 0.54 n = 8

Figure 2.8: Tensile modulus vs. filler content for composites containing varying amounts of a single filler type for GNP/PEEK, CB/PEEK, and CF/PEEK

Table 2.5 also shows that for the tensile modulus, the presence of CF in a composite dominates. For example, for 10CF/PEEK and 2.5CB/10CF/PEEK composites, the tensile modulus is about 6 GPa. For 20CF/PEEK and 2.5CB/20CF/PEEK composites, the tensile modulus is about 11 GPa. For the composites containing 30 wt% CF, (30CF/PEEK, 2.5CB/30CF/PEEK, 5CB/30CF/PEEK, and 7.5CB/30 CF/PEEK), the tensile modulus varies from 16 to 23 GPa.

Figure 2.9 shows the mean ultimate tensile strength (UTS, error bars shown are $\pm$ one standard deviation) for all the composites containing varying amounts of a single filler

type. Again, due to the longer length of the carbon fiber in the composite, the ultimate tensile strength increases as the amount of CF increases. In addition, the tensile strength is much higher for the CF/PEEK composites (230 MPa for 30CF/PEEK) as compared to the GNP/PEEK (ranges from 81 to 95 MPa) and CB/PEEK (ranges from 80 to 89 MPa) composites. For the GNP/PEEK composites, the UTS and strain at UTS (see Table 2.5) remain approximately constant at 95 MPa and 5% strain for composites containing ≤ 10 wt% (6.4 vol%) GNP. It is interesting to note that this drop in UTS and strain at UTS occurs at a point similar to the electrical percolation threshold evident in Figure 5. For the CB/PEEK composites, the UTS and strain at UTS (see Table 2.5) remain approximately constant at 89 MPa and 3% strain for composites containing ≤ 7.5 wt% (5.5 vol%) CB.

Figure 2.9: Ultimate tensile strength vs filler content for composites containing varying amounts of a single filler type for GNP/PEEK, CB/PEEK, and CF/PEEK

Table 2.5 also shows that for the UTS, the presence of CF in a composite dominates. For example, for 10CF/PEEK and 2.5CB/10CF/PEEK composites, the UTS is about 116 MPa. For 20CF/PEEK and 2.5CB/20CF/PEEK composites, the UTS is about 145 MPa. In addition, when ≥ 5 wt% CB is added with 30 wt% CF, the UTS and strain at UTS decreases as compared to the 30CF/PEEK and 2.5CB/30CF/PEEK composites.

2.5 Conclusions

In this work, GNP/PEEK, CF/PEEK, CB/PEEK, and CB/CF/PEEK composites were extruded, injection molded, and tested for tensile and conductivity properties with the aim of developing conductive composites that could be used in aerospace applications. Concerning electrical conductivity, the percolation threshold for each single filler is around 13 wt% (9 vol%) GNP, around 3 wt% (2 vol%) CB, and between 10 wt% (7.4 vol%) and 20 wt% (15.3 vol%) CF. The highly branched structure of the carbon black likely causes the low percolation threshold. The following composites could be used for ESD applications: 15 wt% GNP/PEEK, 20 wt% CF/PEEK, 2.5 wt% CB/10 wt% CF/PEEK, and 2.5 wt% CB/20 wt% CF/PEEK. The following composites could be used in electrically conductive applications ($ER \leq 30$ ohm-cm): 30 wt% CF/PEEK, 5 wt% CB/PEEK, 7.5 wt% CB/PEEK, 10 wt% CB/PEEK, 2.5 wt% CB/30 wt% CF/PEEK, 5 wt% CB/30 wt% CF/PEEK, 7.5 wt% CB/30 wt% CF/PEEK (lowest ER of 0.56 ohm-cm measured in this study). The highest thermal conductivity reported in this chapter is 0.52 W/m-K for 7.5 wt% CB/30 wt% CF/PEEK, which is about twice the thermal conductivity of the neat PEEK (0.25 W/m-K). Doubling the thermal conductivity would be advantageous for aerospace applications that require heat dissipation.

Several important observations were made concerning tensile properties. Due to the longer filler length, as expected the carbon fiber composites had the highest strength and modulus, which were 230 MPa and 23.5 GPa respectively for 30 wt% CF/PEEK.

Composites containing ≤ 10 wt% GNP maintained their tensile strength (~ 95 MPa) and strain ($\sim 5\%$). Composites containing ≤ 7.5 wt% CB maintained their tensile strength (~ 89 MPa) and strain ($\sim 3\%$).

The most conductive composite produced in this study was 7.5 wt% CB/30 wt% CF/PEEK which had a tensile strength of 145 MPa, tensile modulus of 18.4 GPa, ER of 0.56 ohm-cm, and TC of 0.52 W/m-K. This composite could be used in conductive aerospace applications. Per the authors' knowledge, properties on these GNP/PEEK, CB/PEEK, and CB/CF/PEEK composites have not been reported previously in the open literature.

2.6 Future Work

The conductive composites with ER under 50 ohm-cm in this work showed promise for use in shielding effectiveness applications. The shielding effectiveness should be tested for these conductive samples: 30 wt% CF/PEEK, 5 wt% CB/PEEK, 7.5 wt% CB/PEEK, 10 wt% CB/PEEK, 2.5 wt% CB/30 wt% CF/PEEK, 5 wt% CB/30 wt% CF/PEEK, 7.5 wt% CB/30 wt% CF/PEEK. Shielding effectiveness is measured as the difference between an electromagnetic signal's intensity before shielding and after shielding, as described in ASTM D4935. Conductive materials can shield electronics from electromagnetic and radio frequencies, which is beneficial in structural components that house electronic components.

In this study, the percolation threshold for CF in PEEK was determined to be between 10 and 20 wt%. By making composites between 10 and 20 wt% CF, a narrower range for the percolation threshold could be found.

It is possible that greater carbon fiber content would continue to increase the tensile modulus and strength, thermal conductivity, and electrical conductivity. The materials used here limited the composites to 30% CF or lower, but if more carbon fiber from a different source was added, improved properties might be achieved.

2.7 Acknowledgements

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3. Electrical, Thermal, and Tensile Properties of Cycloaliphatic Epoxy/Carbon Black and Cycloaliphatic Epoxy/Fumed Silica Nanocomposites²

3.1 Abstract

Carbon black and fumed silica have previously been shown to reinforce polymer nanocomposites. Their effects on electrical and thermal properties are also of interest for potential use in high voltage and temperature applications. Cycloaliphatic epoxy is examined in this study because of its resistance to aging. The objective of this study was to determine the effects of adding 0.5 to 2 wt% carbon black or 1 to 4 wt% fumed silica to cycloaliphatic epoxy. Electrical conductivity, thermal conductivity, tensile properties, and glass transition temperature as determined by dynamic mechanical analysis were tested. The results of these tests showed that up to 2 wt% carbon black or 4 wt% fumed silica had little effect on the electrical, thermal, or tensile properties of cycloaliphatic epoxy.

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3.2 Introduction

Polymer nanocomposites are used for a wide range of applications wherever lightweight, chemically resistant, or specialized materials are needed. The properties of traditional polymer composites can be enhanced with the addition of nano- or micro-fillers into the matrix phase. Specifically, these fillers can alter the electrical, thermal, and mechanical properties of epoxy resins, allowing tailoring of these materials to specific application requirements. The ability to tailor electro-thermo-mechanical properties is particularly important in the aerospace and power transmission industries, which are both seeing continually increasing levels of polymer composites implementation. Unfortunately, there does not yet exist a comprehensive database of structure-property relationships for various combinations of fillers and polymer resins. The purpose of this study is to understand the influence of two particular filler systems on the electro-thermo-mechanical properties of an epoxy resin.

The epoxy examined in this study is a cycloaliphatic epoxy resin with an anhydride curing agent. Cycloaliphatic epoxy systems have been shown to prevent ozone, UV, and hygrothermal aging [1, 2]. This is important for the transmission lines to have a long lifetime and require less maintenance. The anhydride curing agent gives good mechanical properties at higher temperatures than amine curing agents [3].

Two fillers are examined here: carbon black and fumed silica. Carbon black was chosen because it increases the stiffness and thermal conductivity of composites [4, 5]. Since

carbon black is a conductive filler, small amounts will be used so that we do not pass the percolation threshold. The percolation threshold is the filler content at which the electrical resistivity drops dramatically [6]. The percolation threshold of carbon black has been reported as low as 0.06 vol% (about 0.09 wt%) and as high as 5 wt% (about 3.4 vol%) [7-9]. The glass transition temperature (T_g) of carbon black/epoxy composites is generally described as increasing at lower carbon black content (up to 0.7 wt%) and decreasing at higher carbon black content (starting at 1 wt% or 2.5 wt%) [7-9].

Fumed silica was chosen because it is not electrically conductive, and it has been shown to increase the stiffness of composites [10-15]. It is often used to increase the crystallinity and therefore the stiffness of thermoplastics [14-16], but is also used in thermosets [11-13, 17, 18]. There are contradictory reported effects of the filler on T_g . It has been described as increasing, decreasing, or having no effect on T_g of the composite [11, 12, 15, 18, 19].

3.3 Materials

The epoxy matrix used is a cycloaliphatic epoxy (CE) resin (3,4-epoxycyclohexylmethyl-3',4'-epoxycyclohexane carboxylate) and aromatic epoxy resin (DGEBA = diglycidyl ether of bisphenol A) mixture cured by an anhydride curing agent (ACA) hardener (4,7-Methanoisobenzofuran-1,3-dione-3a,7,7a-tetrahydromethyl). These compounds are shown in Figure 3.1. A Brookfield viscometer was used to

measure the viscosity at 23 °C of the resin and hardener, which were 20.5 Poise and 30.6 Poise, respectively.

Figure 3.1: a) Resin Components, b) ACA Hardener Components

Three different carbon blacks from Cabot were used: CB1, CB2, and CB3. The three carbon blacks from Cabot differ in surface area as measured by BET (Brunauer–Emmett–Teller analysis) using N₂. CB1 is a low surface area carbon black. CB2 is a medium surface area carbon black. CB3 is a high surface area carbon black. Exact properties are proprietary.

A commercial grade fumed silica from Cabot called Silica1 is used in the epoxy composites. Its exact properties are proprietary.

3.4 Experimental

In this section, the fabrication and testing procedures are described for carbon black/epoxy composites and fumed silica/epoxy composites. Batches of 450 g were fabricated to produce bars and disks. These samples were used for testing electrical resistivity (which is the inverse of electrical conductivity), thermal conductivity, tensile properties, and glass transition temperature from dynamic mechanical analysis (DMA).

3.4.1 Fabrication

3.4.1.1 Neat Epoxy

For the neat epoxy material, a ratio of 100 g resin was added to 143 g hardener and poured into a beaker. The beaker was placed under a Ross High Shear Mixer HSM-100 LSKI with 2" dispersion blade and mixed at 1000 rpm for 10 min. The mixture was degassed at 100 °C and 29 inHg vacuum until no bubbles appeared.

The mixture was then poured into a pre-heated mold and degassed again at a full vacuum of 29 inHg. Preheating and degassing were done at 100 °C. The mold had previously been coated with Mann Ease Release 300, then assembled and pre-heated in a vacuum oven. For the neat epoxy, the preheating and final degassing were done at 100 °C. The curing cycle for all the composites was 100 °C for 1 hour, then heated to 200 °C at 2°C/min, then held at 200 °C for two hours. The oven was then turned off, and the cured

epoxy was allowed to cool in the oven to room temperature at a ~ 1 °C/min cooling rate. Typically, the 450 g batches produced 20 rectangular bars (165 mm x 19 mm x 3.3 mm) and 5 disks (64 mm diameter and 3 mm thick). This degassing and curing cycle was used for all epoxy composite fabrication, unless otherwise stated.

3.4.1.2 Carbon Black in Ross Mixer for CB2 and CB3

Initially, all work was done using the Ross mixer. Carbon black (CB) was added to ACA hardener at the lowest speed (~ 500 rpm) under a Ross High Shear Mixer HSM-100 LSKI with 2" dispersion blade. The mixture was mixed at 3000 rpm for 10 min. The mixture was allowed to cool back to room temperature (~ 20 min), and mixed at 3000 rpm for another 10 min. The CB/hardener mixture was then poured slowly into the necessary amount of CE resin (to result in a ratio of 100 g resin added to 143 g hardener) and mixed at 1000 rpm for 10 min. The mixture was then degassed at 100 °C and 29 inHg, alternating vacuum and atmospheric pressure to avoid overflow, until no more bubbles appeared. The mixture was poured into the mold, degassed, and cured as described in the neat epoxy fabrication section. Composites containing 1 wt% CB2 composites and 1 wt% CB3 were made using this procedure (0.67 vol% CB2 and 0.67 vol% CB3).

3.4.1.3 Carbon Black in SpeedMixer for CB1

A FlackTek SpeedMixer DAC 150.1 FVZ was purchased to allow more reproducible mixing of fillers in epoxy. All work in this chapter from this point on uses this SpeedMixer, including all CB1 composites and Silical composites. The SpeedMixer works without a blade by using centrifugal forces to mix the fillers into the epoxy resin. The SpeedMixer has a maximum mixing weight of 100 g. A master batch was used in order to keep the batch sizes under the SpeedMixer maximum batch size of 100 g and increase the viscosity of the master batch for improved mixing.

A master batch containing all the carbon black was made and then split into 5 separate batches of 90 g each for a total of 450 g. CE was measured into the master batch cup, and then CB1 was weighed on top of the resin. The master batch was mixed in the SpeedMixer at 2500 rpm for 2 minutes to blend the CE and CB1. It was then degassed at 70 °C and 29 inHg until no bubbles appeared (~30 minutes). Approximately 1/5th of the master batch was poured into each of 5 mixing cups to make the batches. Additional CE and ACA were poured on top. The final ratio of CE to ACA was 100 g to 143 g in each batch, with 0.5, 1, or 2 wt% CB1. Each batch was mixed at 2500 rpm for 1 min, degassed in the cups at 70 °C and 29 inHg vacuum, and then poured into the mold. The final degassing and curing cycle was the same as for the neat epoxy, with the mold preheating and degassing at 70 °C. Composites containing 0.5, 1, and 2 wt% CB1 were made with this procedure (0.33, 0.67, and 1.34 vol% CB1).

3.4.1.4 Fumed Silica in SpeedMixer

In order to make this material, master batches of 15 wt% Silica1 were fabricated by adding fumed silica (Silica1) on top of CE resin in a mixing cup. The CE and Silica1 was mixed for 1 minute at 3500 rpm in the SpeedMixer. Master batches were then stored at 23 °C overnight (~16 hr). The next morning this master batch was mixed at 3500 rpm for 1 minute. To achieve the desired amount of filler in epoxy, resin and the required amount of master batch were poured into mixing cups and mixed at 1540 rpm for 3 min in the SpeedMixer. The cups were degassed at 70 °C by cycling between vacuum and atmospheric pressure until bubbles stopped forming (~25 minutes). Next, ACA hardener was added to the cup to achieve the desired ratio of 100 g of CE added to 143 g of ACA, and the cup was mixed at 1540 rpm for 1.5 minutes in the SpeedMixer. The cup was degassed again at 70 °C cycling between vacuum and atmospheric pressure until bubbles stopped forming (~10 minutes). The cups were poured into the mold, and the final degassing and curing cycle was the same as for the neat epoxy, but with the mold preheating and degassing at 70 °C. 1, 2, 3, and 4 wt% Silica1 composites were made using this procedure (0.55, 1.10, 1.65, and 2.21 vol% Silica1).

3.4.2 Electrical Resistivity Testing

ASTM D257 was used to measure the volumetric electrical resistivity at 23 °C. A constant voltage (100 V) was applied to the test specimen. Resistivity was measured using a Keithley 6517A Electrometer/High Resistance Meter with an 8009 Resistivity Test Fixture and Keithley 6524 High Resistance Measurement Software. The specimens

tested were disks (6.4 cm diameter and 3 mm thick). Prior to testing, the samples were conditioned at 23 °C and 50% relative humidity for 2 days.

3.4.3 Thermal Conductivity Testing

Through-plane thermal conductivity was measured according to ASTM F433 guarded heat flow meter method using a Holometrix Model TCA-300 Thermal Conductivity Analyzer. The through-plane thermal conductivity of a 3 mm thick, 5 cm diameter disk-shaped test specimen was measured at 55 °C. Prior to testing, the samples were conditioned at 23 °C and 50% relative humidity for 2 days.

3.4.4 Tensile Testing

The tensile properties (at 23 °C, ASTM type I sample geometry) were measured according to ASTM D638 at a crosshead rate of 1 mm/min. An InstruMet Sintech screw-driven mechanical testing machine was used with an axial extensometer to collect strain values. The tensile strength, strain, and modulus were measured at 23 °C.

3.4.5 Glass Transition Temperature Testing

Dynamic Mechanical Analysis (DMA) was used to find the glass transition temperature of the CB/epoxy or fumed silica/epoxy samples. The sample tests were completed on the TA Instruments Q800 DMA the dual/single cantilever clamp. The single cantilever was

used for all testing with a set length of 17.5 mm between clamps. The sample dimensions were 3 mm x 12 mm x 35 mm long cut from 3 mm x 25 mm x 150 mm bars. Samples were rinsed with isopropyl alcohol before testing. Each sample was loaded into the clamps and tightened down using 8 in-lb of torque. Tweezers were used to keep the samples free of oil from hands and fingers.

Once the sample was in place in the clamps, an amplitude test was performed. The test mode was set to “DMA Multi Strain” with the test set to “Strain Sweep”. The frequency was kept at 1 Hz with the isothermal temperature at 35 °C and a soak time of 5 minutes, the amplitude was swept from 5 μm to 50 μm . Following the test, the graph of amplitude versus loss modulus was analyzed to determine the amplitude needed for the temperature sweep test. An amplitude of 30 μm was used for all formulations.

To determine the modulus and tan delta, the program mode was set to “DMA multi frequency strain” and the test was “temp ramp/frequency sweep”. The procedure parameters were an amplitude of 30 μm , an initial temperature of 50 °C with a soak time of 5 minutes, a temperature ramp rate of 3 °C/min, and a final temperature of 290 °C with a hold time of 5 minutes. The frequency was held at 1 Hz for the entire duration of the test. Once the test was completed, the data were analyzed to determine the storage modulus, loss modulus, and tan delta graphs, which were then used to find the glass transition temperature.

3.5 Results and Discussion

This section describes electrical resistivity, thermal conductivity, tensile properties, and glass transition temperature found for 0.5, 1, and 2 wt% carbon black/epoxy and 1, 2, 3, and 4 wt% fumed silica/epoxy composites.

3.5.1 Electrical Resistivity

Figure 3.2 and Table 3.1 show the electrical resistivity for neat CE/ACA epoxy, 0.5 wt%, 1 wt%, and 2 wt% CB1/epoxy; 1 wt% CB2/epoxy; 1 wt% CB3/epoxy; 1 wt%, 2 wt%, 3 wt%, and 4 wt% Silica1/epoxy. The electrical resistivity decreases slightly with increasing filler content for all formulations, but the composites are always electrically insulating. For the conductive carbon black fillers, this means that the percolation threshold has not been reached for up to 2 wt% CB1, 1 wt% CB2, or 1 wt% CB3. The higher surface area carbon blacks (CB2 and CB3) have slightly lower electrical resistivities than CB1. That is, the lower surface area carbon black produces a less electrically conductive sample. Since CB1 is less electrically conductive than CB2 or CB3, its effects at different mass contents were investigated further for use in the epoxy composites. No further work was done with CB2 and CB3. Fumed silica is electrically insulating, so a high electrical resistivity is expected at all filler content levels. Electrical resistivity data can be found in Appendix 8.5.

Figure 3.2: Electrical Resistivity of Epoxy with Different Fillers

Table 3.1: Electrical Resistivity and Thermal Conductivity at 55 °C for Carbon Black/Epoxy and Fumed Silica/Epoxy Composites

Formulation	Electrical Resistivity (Ω-cm)	Thermal Conductivity at 55 °C (W/m-K)
Neat Epoxy	$9.65 \times 10^{16} \pm 2.35 \times 10^{16}$ n=4	0.1496 ± 0.0022 n=4
1 wt% CB2	$5.81 \times 10^{10} \pm 2.13 \times 10^{10}$ n=5	0.1600 ± 0.0022 n=5
1 wt% CB3	$7.74 \times 10^{11} \pm 3.87 \times 10^{11}$ n=5	0.1551 ± 0.0009 n=5
0.5 wt% CB1	$7.55 \times 10^{14} \pm 3.53 \times 10^{14}$ n=4	0.1538 ± 0.0022 n=4
1 wt% CB1	$2.12 \times 10^{14} \pm 2.54 \times 10^{13}$ n=4	0.1529 ± 0.0018 n=4
2 wt% CB1	$5.28 \times 10^{13} \pm 3.94 \times 10^{13}$ n=4	0.1517 ± 0.0029 n=4
1 wt% Silica1	$1.64 \times 10^{15} \pm 9.76 \times 10^{14}$ n=4	0.1534 ± 0.0020 n=4
2 wt% Silica1	$6.13 \times 10^{14} \pm 1.34 \times 10^{14}$ n=3	0.1584 ± 0.0001 n=3
3 wt% Silica1	$9.00 \times 10^{14} \pm 4.94 \times 10^{14}$ n=5	0.1594 ± 0.0013 n=5
4 wt% Silica1	$7.76 \times 10^{14} \pm 3.00 \times 10^{14}$ n=5	0.1614 ± 0.0022 n=5

3.5.2 Thermal Conductivity Results

Figure 3.3 and Table 3.1 show the through-plane thermal conductivity at 55 °C for neat CE/ACA epoxy, 0.5 wt%, 1 wt%, and 2 wt% CB1/epoxy; 1 wt% CB2/epoxy; 1 wt% CB3/epoxy; 1 wt%, 2 wt%, 3 wt%, and 4 wt% Silica1/epoxy. The thermal conductivity does not change significantly for any filler, ranging from 0.15 to 0.16 W/m•K. Thermal conductivity data can be found in Appendix 8.6.

Figure 3.3: Thermal Conductivity of Epoxy with Different Fillers

3.5.3 Tensile Results

Figure 3.4, Figure 3.5, and Table 3.2 show the tensile strength and tensile modulus for neat CE/ACA epoxy, 0.5 wt%, 1 wt%, and 2 wt% CB1/epoxy; 1 wt% CB2/epoxy; 1

wt% CB3/epoxy; 1 wt%, 2 wt%, 3 wt%, and 4 wt% Silica1/epoxy. Both figures indicate that adding these amounts of fillers to CE/ACA epoxy did not have a major effect on the tensile properties. Tensile strength, strain at ultimate tensile stress, and tensile modulus data can be found in Appendix 8.7.

Figure 3.4: Ultimate Tensile Stress of Epoxy with Different Fillers

Figure 3.5: Tensile Modulus of Epoxy with Different Fillers

Table 3.2: Ultimate Tensile Stress, Strain at Ultimate Tensile Stress, and Tensile Modulus for Carbon Black/Epoxy and Fumed Silica/Epoxy Composites

Formulation	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Modulus (GPa)
Neat Epoxy	52.82 ± 3.84 n=6	3.41 ± 0.48 n=6	2.06 ± 0.05 n=6
1 wt% CB2	50.43 ± 1.50 n=7	4.01 ± 0.41 n=7	2.07 ± 0.12 n=7
1 wt% CB3	46.84 ± 1.91 n=8	3.27 ± 0.32 n=8	2.00 ± 0.08 n=8
0.5 wt% CB1	49.94 ± 3.14 n=7	3.83 ± 0.68 n=7	1.92 ± 0.06 n=7
1 wt% CB1	52.15 ± 2.98 n=7	3.82 ± 0.46 n=7	2.05 ± 0.09 n=7
2 wt% CB1	50.42 ± 2.29 n=8	3.34 ± 0.36 n=8	2.07 ± 0.06 n=8
1 wt% Silica1	46.05 ± 1.06 n=9	3.02 ± 0.21 n=9	1.96 ± 0.08 n=9
2 wt% Silica1	50.16 ± 3.15 n=8	3.63 ± 0.52 n=8	1.92 ± 0.08 n=8
3 wt% Silica1	55.33 ± 1.05 n=8	4.60 ± 0.46 n=8	1.97 ± 0.11 n=8
4 wt% Silica1	53.22 ± 1.98 n=11	3.84 ± 0.46 n=11	2.09 ± 0.11 n=11

3.5.4 Glass Transition Temperature

The glass transition temperature (T_g) for the neat CE/ACA epoxy was found to be about 233 °C as determined by the peak of the tan delta curve from dynamic mechanical analysis (DMA). Figure 3.6 and shows the peak of the tan delta curve for neat CE/ACA epoxy, 0.5 wt%, 1 wt%, and 2 wt% CB1/epoxy; 1 wt%, 2 wt%, 3 wt%, and 4 wt% Silica1/epoxy. Data for tan delta peak, storage modulus onset, and loss modulus peak can be seen in Appendix 8.8. Overall, the T_g measured by all three methods is approximately constant for 0 to 2 wt% CB1 and 0 to 4 wt% Silica1 in CE/ACA epoxy. The peak of the tan delta curve varies from 233 °C for neat CE/ACA epoxy to 236 °C for the 0.5 and 1 wt% CB1 in CE/ACA epoxy, and the 4 wt% Silica1 in CE/ACA epoxy.

Figure 3.6: Glass Transition Temperature for Epoxy with Different Fillers as Determined by the Peak of the Tan Delta Curve from DMA

Table 3.3: Storage Modulus Onset, Loss Modulus Peak, and Tan Delta Peak for Carbon Black/Epoxy and Fumed Silica/Epoxy Composites

Formulation	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
Neat Epoxy	199.05 ± 0.94 n=4	219.07 ± 0.20 n=4	233.38 ± 0.28 n=4
0.5 wt% CB1	204.88 ± 0.84 n=2	221.65 ± 0.72 n=2	236.05 ± 0.76 n=2
1 wt% CB1	198.89 ± 0.29 n=3	222.36 ± 0.66 n=3	236.26 ± 0.35 n=3
2 wt% CB1	200.79 ± 1.34 n=3	217.57±0.29 n=3	232.37 ± 0.21 n=3
1 wt% Silica1	196.43 ± 6.11 n=3	219.11 ± 1.24 n=3	234.16 ± 0.10 n=3
2 wt% Silica1	194.45 ± 7.44 n=3	220.34 ± 0.81 n=3	234.98 ± 0.59 n=3
3 wt% Silica1	198.40 ± 1.71 n=3	219.59 ± 0.26 n=3	234.68 ± 0.43 n=3
4 wt% Silica1	195.96 ± 0.50 n=3	220.57 ± 0.55 n=3	236.71 ± 0.18 n=3

3.6 Conclusions

This work has shown that adding up to 2 wt% of the carbon black or up to 4 wt% of the fumed silica to this cycloaliphatic epoxy does not appreciably affect the electrical resistivity, thermal conductivity, tensile properties, or glass transition temperature. There are slight decreases in electrical resistivity with the higher surface area carbon blacks CB2 and CB3. The composite is still very electrically resistive up to 2 wt% CB1.

3.7 Future Work

The tests described in this chapter did not indicate a change in properties due to the addition of these fillers, but different tests could show benefits from adding carbon black or fumed silica to this epoxy system. Aging and additional testing could show if the fillers improve this epoxy's resistance to degradation due to moisture absorption,

temperature changes, or UV radiation. For these electrically resistive materials, various other tests would help to describe the electrical properties. Depending on the potential application of these composites, dielectric strength, dielectric constant, dissipation factor, capacitance, or arc resistance could give interesting results.

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4. Thermal, Electrical, and Mechanical Properties of Talc- and Glass Microsphere- Reinforced Cycloaliphatic Epoxy Composites³

4.1 Abstract

Cycloaliphatic epoxy (CE) is used in high voltage and temperature applications because of its high glass transition temperature and resistance to ultraviolet, ozone, and hygrothermal aging mechanisms. Fillers can be used to increase the tensile modulus and thermal conductivity without a corresponding increase in electrical conductivity (inverse electrical resistivity), which would be detrimental in a high voltage environment. In this study, two fillers were examined in a CE system: talc and glass microspheres (MS). Up to 20 wt% talc/CE and up to 40 wt% glass MS/CE composites were fabricated and tested for electrical resistivity, thermal conductivity, and tensile properties. As desired, all composites remained electrically resistive. Composite thermal conductivity increased with increasing filler content from 0.15 W/m-K for the neat epoxy to 0.25 W/m-K for 20 wt% talc and for 40 wt% glass MS. This thermal conductivity increase could help to dissipate heat in high voltage and temperature applications. Tensile modulus increased

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from 2.7 GPa for the neat epoxy to 3.6 GPa for 20 wt% talc/CE and to 5.2 GPa for 40 wt% glass MS/CE composites. Increasing the tensile modulus is useful in the newly developed Polymer Core Composite Conductors (PCCCs) that are used to transmit power.

4.2 Introduction

The world's power demand continues to rise and the utility companies are being asked to transmit more power, primarily by using existing right-of-ways. Current regional power grids typically use high voltage transmission lines that consist of aluminum cables that transmit power surrounding a steel core that provides the strength needed to support the power line. Recently, Polymer Core Composite Conductors (PCCCs) have been developed that can transmit more electricity than the typical power line. This PCCC technology also decreases sagging of transmission lines, which can cause power grid 'black-outs', by adding a composite core containing carbon fiber (CF)/epoxy and glass fiber (GF)/epoxy for strength and stiffness, which is surrounded by aluminum strands for conduction [1-2]. This study examines adding a nanofiller to epoxy that could be used with either carbon fiber or glass fiber in the polymer composite core. These nanofillers could increase the composite tensile modulus and thermal conductivity, while still producing the desired electrically resistive composite. An increased thermal conductivity could reduce localized hot spots.

Cycloaliphatic resins combined with anhydride curing agents are ideal for PCCC applications. Cycloaliphatic epoxy (CE) resins have been shown to be resistant to ultraviolet, ozone, and hygrothermal aging [2, 3]. Reduced aging effects are important for transmission lines, which are often in service for decades. Anhydride curing agents give the epoxy a higher glass transition temperature (T_g) than an amine curing agent [4]. Higher T_g materials allow the line to operate at a higher temperature and transmit more electricity.

Talc (hydrous magnesium silicate) is an inexpensive, hydrophobic filler that disperses and bonds well in polymers. Thermoplastics such as polypropylene and nylon are often used for talc composites. The thermal conductivity of talc is about ten times that of polypropylene, nylon, and epoxy [5]. In fact, adding 15 vol% talc to polypropylene allowed the composite thermal conductivity to increase from 0.27 W/m-K for the neat polypropylene to around 1.5 W/m-K [6]. Talc has also been shown to increase the composite tensile modulus in polypropylene composites [7-8]. For example, adding 10 vol% talc to polypropylene increased the tensile modulus from 1 GPa for the neat polypropylene to about 2.6 GPa [8]. For nylon composites, Unal et al. reported an increase in tensile modulus from around 2.1 GPa (neat nylon) to around 4.3 GPa (10 wt% talc) [9]. Alves et al. noted an increase in viscosity resulting from adding talc to epoxy [10]. There is very little information in the literature concerning the tensile properties and thermal and electrical conductivity of talc/epoxy composites.

Hollow and solid glass microspheres (MS) are another inexpensive filler that has been used in the past to increase the tensile modulus of epoxy-based composites. For example, Quesenberry et al. reported that the addition of 10 vol% solid glass microsphere produced a composite with a tensile modulus of about 3.5 GPa compared to about 2.8 GPa for the neat epoxy [11]. Patankar et al. found that the addition of 10 wt% hollow glass microsphere caused the tensile modulus to increase approximately 20% in high density polyethylene composites [12]. Gupta et al. has shown that increasing the amount of hollow glass spheres in epoxy also increases the tensile modulus [13].

In this work, talc/cycloaliphatic epoxy (CE) and glass microsphere (MS)/cycloaliphatic epoxy (CE) composites were fabricated and tested for tensile properties, thermal conductivity, and electrical conductivity (inverse electrical resistivity). The objective of this work is to determine the effects of these fillers on the composite thermal and electrical conductivity and tensile properties. Per the authors' knowledge, properties on these talc/CE and glass MS/CE composites have not been reported previously in the open literature.

4.3 Materials and Experimental Methods

4.3.1 Materials

The epoxy matrix used is a cycloaliphatic epoxy (CE) resin (3,4-epoxycyclohexylmethyl-3',4'-epoxycyclohexane carboxylate) cured with an anhydride curing agent (ACA) hardener (4,7-methanoisobenzofuran-1,3-dione-3a,7,7a-tetrahydromethyl). These compounds are shown in Figure 4.1. A Brookfield viscometer at 25 °C was used to measure the viscosity of 5.2 Poise for the mixture of 100 g of resin and 138 g of hardener.

Figure 4.1: a) CE Resin Component, b) ACA Hardener Component

The fillers used in this study were talc and glass MS. The talc used is Imerys Nicron 503, which is a high purity microcrystalline talc (>98 % talc, <2 % dolomite and chlorite) that consists of flakes that have a mean particle diameter of 6 μm , an aspect ratio of 10, and a specific gravity of 2.8. The talc is electrically insulating and has a thermal conductivity of 2.1 W/m-K [5].

The glass used was Potters Spherglass® 5000 CP-03 solid glass microspheres (MS) with a proprietary amino functionalized silane coupling agent (<1 %) to improve interfacial bonding with an epoxy matrix. This is a soda lime A-glass material with a mean diameter of 11 μm and a specific gravity of 2.5. This glass has a thermal conductivity of 1 W/m-K and an electrical resistivity of about 10^{13} ohm-cm [14].

The concentrations (shown in wt% and the corresponding vol%) for composites tested in this research are shown in Table 4.1. Table 4.1 also shows all the thermal conductivity and electrical resistivity results that will be discussed later in this paper. Above 20 wt% talc or 40 wt% glass MS, the uncured batches became too viscous to degas and pour into the molds to produce samples.

4.3.2 Test Specimen Fabrication

The fabrication procedure used for the neat epoxy is described here. The mold was coated with mold release (Mann Ease Release 300) and then assembled and pre-heated in an oven at 100 °C. Cycloaliphatic epoxy (CE) resin and hardener (a ratio of 100 g of CE was added to 138 g of ACA hardener) were poured into 5 mixing cups (containing 90 g in each cup), for a total batch size of 450 g. Each cup was mixed in a FlackTek SpeedMixer DAC 150.1 FVZ, which uses centrifugal forces, for 2 minutes at 2500 rpm. The mixture was degassed at 100 °C by alternating between atmospheric pressure and 29 inHg vacuum. The mixture was poured into the preheated mold. Then the mold was

immediately placed in the oven at 100 °C, and the mixture was degassed again. The material was cured using the following cycle: 100 °C for 1 hour, then heated to 200 °C at 2 °C/min, and then held at 200 °C for two hours. The oven was then turned off and the cured epoxy was allowed to cool in the oven to 25 °C at a cooling rate of 1 °C/min.

For the talc/epoxy composites, the mold was coated with Mann Ease Release 300, then assembled and pre-heated in an oven at 100 °C. For this entire project, a total of 450g was made in each batch and 5 mixing cups containing 90 g each were used. A ratio of 100 g of CE was added to 138 g of hardener throughout this project. Master batches of 50 wt% talc were fabricated by adding talc on top of CE resin in a mixing cup. The CE and talc were mixed for a total of 1.5 minutes at 2500 rpm in the SpeedMixer. Mixing was initially done for 30 seconds at 2500 rpm, which caused temperature of the talc/CE resin in the cup to increase. Hence, the master batch mixture was allowed to cool to <30 °C and then it was mixed again at 2500 rpm for 1 additional minute. To achieve the desired amount of filler in the composite, neat CE resin, master batch, and hardener were poured into mixing cups and mixed at 2500 rpm for 2 min in the SpeedMixer. The cups were degassed at 100 °C by cycling between 29 inHg vacuum and atmospheric pressure for about 35 minutes until bubbles stopped forming. The degassed mixture was then poured into the mold, degassed for the final time, and cured as described in the paragraph above.

For the glass MS/epoxy composites, the mold was coated with Mann Ease Release 300, then assembled and pre-heated in an oven at 100 °C. Master batches of 75 wt% glass MS were fabricated by adding glass MS on top of CE resin in a mixing cup. The CE and glass MS were mixed for a total of 3.5 minutes at 2500 rpm in the SpeedMixer. As described in the paragraph above, mixing was done for 30 seconds and then 1 minute at a time, allowing cooling between each minute of mixing so that the temperature of the master batch stayed under 50 °C. To achieve the desired amount of filler in composite, CE resin, master batch, and hardener were poured into cups and mixed at 2500 rpm for 1 minute in the SpeedMixer. The cups were degassed at 100 °C by cycling between 29 inHg vacuum and atmospheric pressure until bubbles stopped forming. The degassed mixture was then poured into the mold, degassed for the final time, and cured as described previously.

4.3.3 Scanning Electron Microscope (SEM) Test Methods

Talc/CE samples were prepared for the environmental scanning electron microscope (ESEM) by cutting a 3 mm thick x 12 mm long x 12 mm wide composite sample from a tensile bar and mounting it in a cast epoxy puck. The composite surface was ground using 320 grit, then 600 grit, and lastly 1200 grit sandpaper. The sample was then polished using 9 µm diamond suspension and then with 3 µm diamond suspension on a Buehler Ecomet 4 variable speed grinder-polisher. The surface was polished further in the Ecomet 4 using a 1 µm alumina/water slurry, and then with a 0.05 µm alumina/water slurry. The polished samples were then coated with 20 nm of carbon using a Denton

DV-502A High Vacuum Evaporator. The composites were viewed using a Philips XL 40 Environmental Scanning Microscope at 15 kV accelerating voltage and 10 mm working distance using the backscattering detector.

Glass MS/CE composites were prepared for field emission scanning electron microscope (FESEM) by cutting a 3 mm x 3 mm x 3 mm section from the tensile fracture surface, mounting it to a sample stud, and coating it with carbon paint. The samples were sputter coated with 1.5 nm of platinum using an Anatech Hummer 6.2 Sputtering System. Images were taken using the lower secondary electron detector using a 2 kV accelerating voltage and 13.5 mm working distance on a Hitachi S-4700 FESEM.

4.3.4 Electrical Resistivity (ER) Test Method

Prior to testing, the samples were conditioned at 23 °C and 50% relative humidity for 2 days. ASTM D257 was used to measure the electrical resistivity at 23 °C [15]. A constant voltage of 100 V was applied to the test specimen. Resistivity was measured using a Keithley 6517A Electrometer/High Resistance Meter with an 8009 Resistivity Test Fixture and Keithley 6524 High Resistance Measurement Software. The specimens tested were disks (6.4 cm diameter and 3 mm thick).

4.3.5 Thermal Conductivity (TC) Test Method

Prior to testing, the samples were conditioned at 23 °C and 50% relative humidity for 2 days. Through-plane thermal conductivity was measured according to ASTM F433 guarded heat flow meter method using a Holometrix Model TCA-300 Thermal Conductivity Analyzer [16]. The through-plane thermal conductivity of a 3 mm thick, 5 cm diameter disk-shaped test specimen was measured at 55 °C.

4.3.6 Tensile Test Method

Prior to testing, the samples were conditioned at 23 °C and 50 % relative humidity for 2 days. The tensile properties (at 23 °C, ASTM type I dog bone shaped sample geometry) were measured according to ASTM D638 at a crosshead rate of 1 mm/min [17]. An InstruMet Sintech screw-driven mechanical testing machine was used with an axial extensometer to collect strain values. The tensile strength, strain, and modulus were measured at 23 °C.

4.4 Results

4.4.1 Scanning Electron Microscopy (SEM) Results

An ESEM image of 20 wt% talc in epoxy can be seen in Figure 4.2. The z-direction (coming out of the page) is along the length of the molded bar. The image confirms the flake shape, and also shows that the flake is randomly oriented in the epoxy matrix. A

FESEM image of 40 wt% glass MS/epoxy composites can be seen in Figure 4.3. The figure shows that, as expected, the glass MS is spherical. In addition, bits of epoxy can be seen adhering onto the glass MS surface in the lower image.

Figure 4.2: Scanning Electron Microscopy Image of 20 wt% Talc/Epoxy Composite

Figure 4.3: Field Emission Scanning Electron Microscopy Image of 40 wt% Glass MS/Epoxy Composite

4.4.2 Electrical Resistivity (ER) Results

Table 4.1 shows the ER results (mean, standard deviation, and number of samples tested) for all the talc/CE and glass MS/CE composites. Appendix 8.9 contains the detailed electrical data. For the talc/epoxy composites, the ER varied from 3.7×10^{16} ohm-cm for the neat epoxy to 1.9×10^{16} ohm-cm for the 20 wt% talc/epoxy composite. For the glass MS/epoxy composites, the ER varied from 3.7×10^{16} ohm-cm for the neat

epoxy to 5.8×10^{14} ohm-cm for the 40 wt% glass MS/epoxy composite. Obviously, adding the electrically resistive talc or glass MS to epoxy still produced an electrically resistive composite, which is desirable for possible PCCC applications.

Table 4.1: Filler Loading Levels and Electrical Resistivity and Thermal Conductivity

Results for Talc/Epoxy Composites and Glass MS/Epoxy Composites

Formulation	Filler Content wt% (vol%)	Electrical Resistivity ohm-cm	Thermal Conductivity W/m-K
Neat	0.0 (0.0)	$3.65 \times 10^{16} \pm 1.05 \times 10^{15}$ n=5	0.149 ± 0.001 n=6
5 Talc	5.0 (2.2)	$3.53 \times 10^{16} \pm 1.43 \times 10^{15}$ n=5	0.168 ± 0.001 n=5
10 Talc	10.0 (4.6)	$2.12 \times 10^{16} \pm 1.26 \times 10^{15}$ n=4	0.188 ± 0.001 n=5
15 Talc	15.0 (7.1)	$1.87 \times 10^{16} \pm 1.74 \times 10^{15}$ n=5	0.222 ± 0.004 n=5
20 Talc	20.0 (9.8)	$1.94 \times 10^{16} \pm 1.11 \times 10^{15}$ n=5	0.255 ± 0.001 n=6
5 Glass MS	5.0 (2.5)	$1.05 \times 10^{16} \pm 4.88 \times 10^{14}$ n=5	0.148 ± 0.001 n=5
10 Glass MS	10.0 (5.1)	$4.89 \times 10^{15} \pm 2.84 \times 10^{14}$ n=5	0.160 ± 0.001 n=5
20 Glass MS	20.0 (10.9)	$1.74 \times 10^{15} \pm 8.86 \times 10^{13}$ n=5	0.184 ± 0.002 n=5
30 Glass MS	30.0 (17.3)	$1.23 \times 10^{15} \pm 9.42 \times 10^{13}$ n=5	0.204 ± 0.004 n=5
40 Glass MS	40.0 (24.5)	$5.79 \times 10^{14} \pm 2.79 \times 10^{13}$ n=5	0.241 ± 0.005 n=6

4.4.3 Thermal Conductivity (TC) Results

Table 4.1 also shows the TC results (mean, standard deviation, and number of samples tested) for all the talc/CE and glass MS/CE composites. Appendix 8.10 contains the detailed TC results. Adding 20 wt% talc to epoxy increased the thermal conductivity from 0.15 W/m-K for the neat epoxy to 0.26 W/m-K. The addition of 40 wt% glass MS to epoxy increased the thermal conductivity from 0.15 W/m-K for the neat epoxy to 0.24 W/m-K. The thermal conductivity of talc is 2 W/m-K as compared to 1 W/m-K for glass MS so it is logical that approximately twice as much glass MS had to be added to the

epoxy to approach the same thermal conductivity level that was achieved in the talc/epoxy composites. Adding 20 wt% talc or 40 wt% glass MS to epoxy increased the composite thermal conductivity by about 67%, which could be useful to dissipate heat in PCCC applications. Weidenfeller et al. reported an increase in TC from 0.25 W/m-K (pure polypropylene) to 1.4 W/m-K for 15 vol% talc in polypropylene (TC of this talc was 10.7 W/m-K as compared to 2 W/m-K in our work) [6]. Due to the lower TC of the talc we used for this project, the TC of our samples would be expected to be lower.

4.4.4 Tensile Results

Table 4.2 and Appendix 8.11 show the tensile results (mean, standard deviation, and number of samples tested) for the talc/CE and glass MS/CE composites. For the talc/CE composites, the ultimate tensile strength remains relatively constant near 50 MPa and the strain at ultimate tensile strength is relatively unchanged around 2%. Adding 5 to 40 wt% glass MS to epoxy caused a decrease in ultimate tensile strength from 47 MPa for the neat epoxy to about 40 MPa. Adding glass MS to epoxy caused a decrease in strain at ultimate tensile strength from about 1.8% to 0.8% for 40 wt% glass MS. Figure 4.4 shows the ultimate tensile strength (mean with error bars showing ± 1 standard deviation) for the talc/CE and glass MS/CE composites. Karrad et al. recorded a decrease in tensile strength from around 14 MPa to around 11 MPa from the addition of 12 vol % talc to high density polyethylene [18]. Unal et al. reported that the tensile strength increased from around 65 MPa (neat nylon) to around 68 MPa as a result of adding 20 wt% talc to nylon [9]. Leong et al. noticed that the tensile strength increased

from about 17 MPa to about 25 MPa when 12 vol% talc was added to polypropylene, and the strain at ultimate tensile strength decreased from 50 to around 18 % [8]. Lee et al. reported that the tensile strength stayed relatively constant near 87 MPa as up to 30 vol% glass beads were added to epoxy [19]. Our lower strain results are consistent with the reduction in tensile strain noted by other researchers when adding talc and glass MS. Increases in tensile strength noted by others as a result of adding talc are due to the filler orientation induced during injection molding of the thermoplastic polymers (nylon and polypropylene) and to the increase in thermoplastic polymer crystallinity caused by adding nucleation sites (talc). The fillers in our talc/epoxy and glass MS/epoxy composites are randomly oriented. The random orientation can be visually confirmed in Figure 4.2, and by the closest matching Halpin-Tsai model for 3D random orientation in the next section, instead of 2D random or aligned, which were checked but not included.

Table 4.2: Tensile Properties of Talc/Epoxy Composites and Glass MS/Epoxy

Composites

Formulation	Tensile Modulus GPa	Ultimate Tensile Strength MPa	Strain at Ultimate Tensile Strength %
Neat	2.73 ± 0.13 n=8	47.2 ± 4.1 n=8	1.82 ± 0.22 n=8
5 Talc	2.91 ± 0.09 n=7	54.9 ± 2.3 n=7	2.11 ± 0.13 n=7
10 Talc	3.15 ± 0.09 n=7	50.3 ± 3.5 n=7	1.79 ± 0.16 n=7
15 Talc	3.25 ± 0.08 n=9	50.8 ± 2.5 n=9	1.81 ± 0.16 n=9
20 Talc	3.59 ± 0.14 n=8	50.8 ± 2.2 n=8	1.64 ± 0.11 n=8
5 Glass MS	2.85 ± 0.08 n=9	39.5 ± 3.3 n=9	1.42 ± 0.14 n=9
10 Glass MS	3.21 ± 0.11 n=8	41.1 ± 3.1 n=8	1.34 ± 0.14 n=8
20 Glass MS	3.84 ± 0.16 n=6	40.2 ± 2.3 n=6	1.09 ± 0.07 n=6
30 Glass MS	4.49 ± 0.17 n=7	37.6 ± 1.8 n=7	0.86 ± 0.05 n=7
40 Glass MS	5.22 ± 0.09 n=5	39.2 ± 3.0 n=5	0.78 ± 0.08 n=5

Figure 4.4: Ultimate Tensile Strength in MPa vs. Volume Fraction Talc or Glass MS

Figure 4.5 and Figure 4.6 show the tensile modulus (mean with error bars showing ± 1 standard deviation) for the talc/CE and glass MS/CE composites, respectively. Also shown here are models that will be discussed later. For the talc/CE composites, the tensile modulus increased from 2.7 GPa for the neat CE to 3.6 GPa for the 20 wt% (9.8 vol%) talc/CE composite. Injection molded talc/thermoplastic composite results were found in the literature. For example, Unal et al. reported an increase in tensile modulus from about 2.2 GPa (neat nylon) to about 3.6 GPa for 20 wt% talc/nylon composite [9]. Leong et al. reported an increase in tensile modulus from 1 GPa (neat polypropylene) to around 2.7 GPa for 12 vol% talc/polypropylene [8]. As expected, our tensile modulus is lower due the random orientation of talc in our composites.

Figure 4.5: Tensile Modulus in GPa vs. Volume Fraction Talc

Figure 4.6: Tensile Modulus in GPa vs. Volume Fraction Glass MS

For the glass MS/CE composites, the tensile modulus increased from 2.7 GPa for the neat CE to 5.2 GPa for the 40 wt% (24.5 vol%) glass MS/CE composite. Quesenberry et al. reported an increase in tensile modulus from about 2.8 GPa (neat epoxy) to about 4.5 GPa for 25 vol% solid glass spheres/epoxy composite [11]. Similarly, Lee et al. reported an increased modulus from about 2.8 GPa for the neat epoxy, to about 4.9 GPa for 20 vol% glass MS [19]. Our tensile modulus results reported here are similar.

Various models were used to study the experimental tensile modulus results. Einstein's model, shown in Equation (4.1), takes the volume fraction of filler (V_f) and the matrix modulus (E_m) into account to calculate the composite tensile modulus (E_c) [18, 20].

$$E_c = E_m(1 + 2.5V_f) \quad (4.1)$$

Guth and Smallwood expanded upon the Einstein equation by considering interparticle interactions as shown in Equation (4.2) [18, 21].

$$E_c = E_m(1 + 2.5V_f + 14.1V_f^2) \quad (4.2)$$

The Halpin-Tsai model describes composites with aligned, short fibers, and can be seen in Equations (4.3)-(4.6). It takes into account the aspect ratio (L or length/ D or diameter)

of the filler, and the filler tensile modulus (E_f), as well as the filler volume fraction and the matrix modulus.

$$E_L = E_m \frac{1 + 2\left(\frac{L}{D}\right)\eta_L V_f}{1 - \eta_L V_f} \quad (4.3)$$

$$E_T = E_m \frac{1 + 2\eta_T V_f}{1 - \eta_T V_f} \quad (4.4)$$

$$\eta_L = \frac{\left(\frac{E_f}{E_m}\right) - 1}{\frac{E_f}{E_m} + 2\left(\frac{L}{D}\right)} \quad (4.5)$$

$$\eta_T = \frac{\left(\frac{E_f}{E_m}\right) - 1}{\left(\frac{E_f}{E_m}\right) + 2} \quad (4.6)$$

In the equations above, E_L is the longitudinal composite tensile modulus, and E_T is the transverse composite tensile modulus. Equation (4.7) shows the composite tensile modulus for three dimensionally (3D) randomly oriented fillers, which is what we produced in this study [18, 22-24].

$$E_c = 0.2E_L + 0.8E_T \quad (4.7)$$

The Nielsen tensile modulus model is more versatile for short fibers and particulates. It takes into account the filler packing as well as the aspect ratio, and can be seen in Equations (4.8)-(4.11) [23-26]:

$$E_C = E_m \frac{1 + ABV_f}{1 - B\psi V_f} \quad (4.8)$$

$$A = k_E - 1 \quad (4.9)$$

$$B = \frac{\left(\frac{E_f}{E_m}\right) - 1}{\frac{E_f}{E_m} + A} \quad (4.10)$$

$$\psi = 1 + V_f \frac{1 - \phi_m}{\phi_m^2} \quad (4.11)$$

In the equations above, k_E is the Einstein coefficient and ϕ_m is the maximum packing fraction of the filler. The Einstein coefficient, and therefore the constant A , is related to the filler aspect ratio and filler orientation.

McGee and McGullough proposed another equation for ψ , which was originally developed for glass sphere/epoxy composites. This relation is shown in Equation (4.12), which includes the matrix volume fraction (V_m) [27].

$$\psi = 1 + \frac{V_m}{\phi_m} [\phi_m V_f + V_m (1 - \phi_m)] \quad (4.12)$$

This modified ψ term can be used in the Nielsen Equations (4.8)-(4.10) to calculate a composite modulus.

Figure 4.5 shows the tensile modulus for the talc/CE composites along with the models discussed above. In the models described, the following values were used [5, 23, 25, 26, 28].

$$E_m = 2.73 \text{ GPa (see Table 4.2)}$$

$$E_{talc} = 20.0 \text{ GPa}$$

$$L/D = 10$$

$$A = 4.93 \text{ (which corresponds to } L/D = 10)$$

$$\phi_m = 0.206$$

Figure 4.5 shows that the 3D Halpin-Tsai model fits the experimental data the best. The Einstein model underpredicts the composite tensile modulus. The Guth-Smallwood and Nielsen models overpredict the composite tensile modulus.

Figure 4.6 shows the tensile modulus for the glass MS/CE composites along with the models discussed above. In the models above, the following values were used [14, 23, 25, 26].

$$E_m = 2.73 \text{ GPa (see Table 4.2)}$$

$$E_{\text{glass MS}} = 69.0 \text{ GPa}$$

$$L/D = 1 \text{ (sphere)}$$

$$A = 1.5 \text{ (which corresponds to } L/D = 1)$$

$$\phi_m = 0.545$$

Figure 4.6 shows that the Nielsen model with the modified ψ fits the experimental glass MS/CE results the best up to 17 vol% (30 wt%) glass MS. The Einstein model and Halpin-Tsai models underpredict the composite tensile modulus. The Guth-Smallwood model overpredicts the composite tensile modulus.

4.5 Conclusions

In this work, talc/CE and glass MS/CE composites were fabricated and tested for tensile properties as well as thermal and electrical conductivity with the aim of developing composites for use in polymer core composite conductors (PCCCs). Per the authors' knowledge, conductivity and tensile properties have never been previously reported in the open literature on these talc/CE and glass MS/CE composites. Currently, no particulate filler is used in PCCC lines, so any improvement in properties over the neat epoxy corresponds to an improvement in the properties of the power transmission lines. As desired for PCCC applications, the composites remained electrically resistive for all formulations, ranging from 3.7×10^{16} ohm-cm for the neat CE to 1.9×10^{16} ohm-cm for 20 wt% talc in CE and 5.8×10^{14} ohm-cm for 40 wt% glass MS in CE. The thermal

conductivity increased from 0.15 W/m-K (neat CE) to about 0.25 W/m-K for 20 wt% talc or 40 wt% glass MS in CE. This 67% increase in thermal conductivity could be useful to dissipate heat in PCCCs.

This work produced some interesting tensile results. The tensile strength and strain remained relatively constant at 50 MPa and 2 %, respectively, as a result of adding 5 to 20 wt% talc. These results are similar to that of the neat epoxy. Being able to add up to 20 wt% talc without decreasing strength and strain is an important finding for PCCC applications. Adding 5 to 40 wt% glass MS to epoxy caused the tensile strength and strain to decrease to around 40 MPa and 1 %, respectively. The tensile modulus increased from 2.7 GPa for the neat epoxy to 3.6 for 20 wt% talc and 5.2 GPa for 40 wt% glass MS. This increase in tensile modulus is useful for PCCC applications to support the conductive aluminum cables in the transmission line. The 3D Halpin-Tsai model fits the talc/CE tensile modulus data the best. The Nielsen model with the modified ψ fits the glass MS/CE results the best up to 17 vol% (30 wt%) glass MS.

4.6 Future Work

Both talc and glass MS showed promise as fillers for PCCC power transmission lines. It would be useful to do further testing to determine other benefits or drawbacks of adding talc or glass MS. The impact of these fillers on composite aging effects due to UV radiation, humidity, elevated temperatures, and temperature cycling could be tested to help evaluate the lifetime of these PCCC cores. In Chapter 5, the aging effects of

elevated temperature and humidity on talc/CE composites are examined. It could also be useful to test the mechanical, electrical, and thermal properties of these fillers/epoxy systems combined with the glass or carbon fibers that will be used in PCCCs, and with the aluminum conductors that would be used in the final transmission lines.

Further electrical testing would provide more information on these high resistivity composites. Arc resistance, dissipation factor, capacitance, dielectric constant, and dielectric strength could be useful tests for PCCC applications or other high voltage applications.

4.7 Acknowledgements

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5. Accelerated Hygrothermal Aging of Cycloaliphatic Epoxy/Graphene Nanoparticle Composites⁴

5.1 Abstract

Cycloaliphatic epoxy (CE) resin systems are of primary interest in applications that require improved resistance to harsh conditions relative to other epoxy systems. Because other epoxy systems have demonstrated improved resistance to hygrothermal aging with the addition of carbon-based nanoparticle reinforcement, it is expected that the hygrothermal resistance of CE resins will likewise be improved with incorporation of nanoparticles. Therefore, the objective of this study is to determine the influence of graphene nanoparticles (GNP) on the hygrothermal aging resistance of CE resins. CE specimens are fabricated with varying levels of GNP and exposed to elevated temperatures and moisture levels for varying amounts of time up to 400 h. The results from flexure and dynamic mechanical testing indicate that the addition of GNP provides modest improvements in the stiffness and glass transition temperature for all aging levels, while the strength is improved for aging times below 400 h.

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5.2 Introduction

Cycloaliphatic epoxies (CE) are commonly used in structural applications requiring improved resistance to elevated temperatures and UV radiation relative to diglycidyl ether of bisphenol A (DGEBA) or bisphenol F (DGEBF) epoxies. In one particular application, CE-based epoxy composites are used for structural reinforcement in aluminum conductor composite core (ACCC) power lines [1, 2]. ACCC lines must withstand harsh environmental conditions, such as long-term mechanical fatigue, temperature, moisture, UV, and high electric field exposure [3-6]. Thus, CE epoxy composites must be designed with optimal resistance to aging to prevent the catastrophic failure of ACCC power lines.

Graphene nanoparticles (GNPs) are known to increase the Young's modulus of epoxies [7-9] and carbon fiber/epoxy composites [10] when sufficiently dispersed, with or without chemical functionalization. These increases are known to originate from the increasing level of load transfer from the epoxy to the stiffer GNPs with increasing levels of GNP volume fraction. A limited number of studies have focused on examining the influence of graphene-based fillers on the aging characteristics of epoxies. Starkova et al. [11] demonstrated the influence of hygrothermal aging on the properties of DGEBA reinforced with thermally reduced graphene oxide (TRGO) particles. Prolongo et al. [12] examined the moisture uptake of GNP/DGEBA composites for various levels of GNP loading. To the authors' knowledge, no study has yet been performed on the effects of hygrothermal aging on the mechanical properties of GNP/CE nanocomposites.

The goal of this study is to determine the influence of GNPs on the mechanical properties of hygrothermally aged CEs. The following sections describe the materials, fabrication techniques, and test methods used to establish the dependency of CE mechanical properties on GNP loading and hygrothermal aging time. The results are compared to the results of similar studies found in the literature.

5.3 Materials and Experimental Methods

5.3.1 Materials

The CE-based epoxy used in this study consisted of a CE resin (3,4-epoxycyclohexylmethyl-3,4-epoxycyclohexylcarboxylate) blended with relatively small amounts of DEGBA (specific blend ratio is proprietary with the supplier). This blend was cured by an anhydride (4,7-methanoisobenzofuran-1,3-dione-3a,7,7a-tetrahydromethyl). The chemical structures of these three molecules are shown in Figure 5.1. The curing mechanism is a chain reaction that starts with the opening of an anhydride ring that exposes a carboxyl group. The carboxyl group reacts with an epoxide group to form a chain with a new exposed hydroxyl group, which is used in a subsequent reaction with either an epoxide or an anhydride group. These reactions proceed until

there are no more epoxide groups available for reaction and the crosslinked system is a solid matrix.

Figure 5.1: Monomer Molecules used for Solid Epoxy Matrix

The GNPs used in this study were Asbury Carbon TC307. TC307 has an average particle diameter of $<1\ \mu\text{m}$, a surface area of $350\ \text{m}^2/\text{g}$, a density of about $2\ \text{g/mL}$, and consist of 8 layers of graphene on average as received. It was previously shown by the current research group in the study of Hadden et al. [10] that the material preparation procedures described below are effective in reducing the average number of layers from 8 to about 4 in the final composite material. It was further shown by Hadden et al. [10] that the number of layers, and thus the level of dispersion, plays a critical role in the overall performance of composite materials. Although an exact laboratory-based determination of the average number of graphene layers is very difficult, Hadden et al. [10] demonstrated that a combination of modeling and experiment could be used efficiently to establish the effect of graphene layers on overall composite properties.

5.3.2 Test Specimen Fabrication

For the neat epoxy material, a multi-part mold was coated with Mann Ease Release 300, then assembled and pre-heated in a vacuum oven at 100 °C. The appropriate amounts of resin and hardener (proprietary mixing ratio) were weighed into a beaker. The beaker was placed under a Ross High Shear Mixer (HSM-100 LSKI) with 2” dispersion blade. The mixture was mixed at 1000 rpm for 10 min, until the two parts were thoroughly mixed. The mixture was then degassed at 100 °C and 29 inHg, alternating vacuum with atmospheric pressure to avoid overflow, until no more bubbles appeared. The mixture was then poured into the pre-heated mold and degassed once more. The curing cycle used was 100 °C for 1 h, then heated to 200 °C at 2 °C/min then held at 200 °C for 2 h. The oven was then turned off and the cured epoxy was allowed to cool in the oven to room temperature at a ~1 °C/min cooling rate.

For the 2 and 4 wt% GNP material systems, a multi-part mold was coated with Mann Ease Release 300, then assembled and pre-heated in a vacuum oven at 100 °C. The amount of GNP needed was weighed into a beaker, along with the corresponding amount of hardener. The beaker was then placed under a Ross High Shear Mixer (HSM-100 LSKI) with 2” dispersion blade. The mixture was mixed at 3000 rpm for 1 h, and then the mixture was placed in a Branson Sonicator CPX2800H operating at 40 kHz and sonicated for 1 h. The corresponding amount of resin was added to the mixture, then mixed at 1000 rpm for 10 min. The mixture was then degassed at 100 °C and 29 inHg, alternating vacuum with atmospheric pressure to avoid overflow, until no more bubbles

appeared. The mixture was poured into the pre-heated mold and degassed once more. The curing cycle used was 100 °C for 1 h, then heated to 200 °C at 2 °C/min, then held at 200 °C for 2 h. The oven was turned off and the cured epoxy was allowed to cool in the oven to room temperature, ~1 °C/min cooling rate.

From the fabricated plaques, a series of 3-point bend and dynamic mechanical analysis (DMA) specimens were machined. The 3-point bend specimens had dimensions of 127 mm x 12.7 mm x 3.2 mm per ASTM D790, and the DMA single-cantilever beam specimens had dimensions of 35 mm x 12.7 mm x 3.2 mm. A total of 20 3-point bend and 20 DMA specimens were machined for each of the three materials systems: neat, 2 wt%, and 4 wt% GNP. Thus, a total of 60 3-point bend and 60 DMA specimens were prepared. Figure 5.2 shows a SEM image of a fracture surface of the composite material.

Figure 5.2: Scanning Electron Microscopy Image of the GNP/Epoxy Composite

5.3.3 Testing Procedure

A QUV Accelerated Weathering Testing Machine was used to condition the specimens under controlled temperature (50 °C) and humidity conditions (100 % relative humidity). Both 3-point bend and DMA specimens were exposed to these conditions for 0, 50, 100, 200, and 400 h. Before aging, all specimens were kept at 50 % relative humidity and 23 °C for two days.

During the conditioning, mass measurements were made on the DMA specimens at regular intervals to monitor the moisture uptake. For each mass measurement, the

specimens were quickly removed from the weathering chamber and sealed in a moisture barrier bag to cool to room temperature (10 min). After cooling, the specimens were removed from the bags, excess surface moisture removed, and mass measurements performed. The specimens were returned to the weathering chamber immediately after measuring.

For both the 3-point bend and DMA specimens for each material system (neat, 2 wt%, 4 wt% GNP), 4 specimens were left unaged, 4 specimens aged for 50 h, 4 aged for 100 h, 4 aged for 200 h, and 4 were aged for 400 h. After aging was complete, each specimen was immediately removed from the chamber and the excess surface moisture removed. The specimens were promptly sealed in moisture barrier bags and allowed to cool to room temperature before testing.

The 3-point bend tests were performed per the specifications of ASTM D790 with a crosshead rate of 5.3 mm/min on an Instru-Met Sintech screw-driven mechanical testing machine with an LVDT (linear variable differential transformer) to record specimen deflection. The flexural modulus and flexural strength values were calculated based on the load-deflection data using the equations outlined in ASTM D790.

The DMA tests were performed on a TA Instruments Q800 DMA using the single cantilever clamp fixture. An oscillating amplitude of 30 μm was applied to the specimen, with an initial temperature of 50 °C with a soak time of 5 min, a temperature ramp rate

of 3 °C/min, and a final temperature of 290 °C with a hold time of 5 min. The frequency was held at 1 Hz for the entire duration of the test. Once the test was completed, the data was analyzed to determine the tan delta values for each specimen. The glass transition temperature was assumed to correspond to the peak of the tan delta values for each material system and aging level.

5.4 Results and Discussion

5.4.1 Moisture Content Results

Figure 5.3 and Appendix 8.12 show the mass gain of the nanocomposite over the 400 h of hygrothermal exposure. The error bars represent a standard deviation over the multiple specimens measured for each exposure time interval. From the figure, it is clear that during the first 100 h the neat resin specimen absorbed water more quickly than the specimens with GNP content. The likely cause of this behavior is the increase in moisture diffusion path length in the presence of hydrophobic GNP. After 100 h the specimens started to approach saturation, and the moisture content of all three material systems stabilized around 2%. For the composite systems, these results are similar to those of Starkova et al. [11] and Prolongo et al. [12]. The equilibrium water absorption

level in the neat epoxy is consistent with that observed by Prolongo et al. [12] and less than half of the amount reported by Starkova et al. [11].

Figure 5.3: Water Mass Gain for GNP/Epoxy Nanocomposites

5.4.2 Flexural Modulus Results

Figure 5.4 shows the flexural modulus for all three material systems over the entire exposure time. The error bars represent a standard error over the multiple specimens tested for each material at each aging time. The flexural modulus data can also be seen in Table 5.1 (± 1 standard deviation, and number of samples) and Appendix 8.13. As is observed in many unaged epoxy nanocomposite systems [13–20], the increasing levels of nanoreinforcement resulted in increasing flexural modulus (stiffness) values for all exposure times, which indicates that the load transfer capability of the system remains effective throughout the aging process for sub-failure stress levels. For 50 and 100 h of

exposure, no significant change in the flexural modulus was observed for the materials relative to the unaged state. After 200 h of exposure, a very modest decrease of a few percent in the flexural modulus was observed for the neat resin and 2 wt% GNP materials. This result is consistent with Starkova et al. [11], who demonstrated a slight decrease in storage modulus with hygrothermally aged neat epoxy and graphene/epoxy composites (0.3 wt% TRGO) tested near room temperature. It is also apparent from Figure 5.4 that within the error bars, the 4 wt% GNP material appears to have a constant modulus over the full aging time. The observed change in modulus at 200 h of exposure in the neat resin and 2 wt% GNP material could be due to plasticization with increasing moisture content, which is partially mitigated with higher levels of GNP content.

Figure 5.4: Flexural Modulus for GNP/Epoxy Nanocomposites

Table 5.1: Flexural Modulus for GNP/Epoxy Nanocomposites

Aging Time (hr)	Neat Epoxy Modulus (GPa)	2 wt% GNP Modulus (GPa)	4 wt% GNP Modulus (GPa)
0	1.94 ± 0.03 n=4	2.02 ± 0.04 n=5	2.08 ± 0.24 n=5
50	1.95 ± 0.04 n=4	2.03 ± 0.06 n=4	2.08 ± 0.04 n=4
100	1.93 ± 0.04 n=4	1.99 ± 0.05 n=4	2.14 ± 0.11 n=3
200	1.87 ± 0.02 n=4	1.94 ± 0.02 n=3	2.02 ± 0.01 n=4
400	1.87 ± 0.02 n=4	1.94 ± 0.03 n=4	2.07 ± 0.04 n=4

5.4.3 Flexural Strength Results

The flexural strength of each of the material systems for the entire exposure time is shown in Figure 5.5 and Table 5.2. The error bars represent a standard error, and the table includes standard deviation and number of samples. There is a small increase in the material strength with increasing GNP loading for the complete set of aging times, which is typically observed in other epoxy nanocomposite systems [17, 18, 21]. There is no clear change in the flexural strength for all three materials up to 200 h of exposure. At 400 h, there is a few percent drop in strength for the 2 wt% and 4 wt% GNP materials, and no change for the neat resin. The subtle decrease in the strength at 400 h could be due to degradation of the epoxy/GNP interface region in the nanocomposites that affects the ultimate failure of the interface but not the load transfer at lower loads (per the discussion above).

Figure 5.5: Flexural Strength for GNP/Epoxy Nanocomposites

Table 5.2: Flexural Strength for GNP/Epoxy Nanocomposites

Aging Time (hr)	Neat Epoxy Strength (MPa)	2 wt% GNP Strength (MPa)	4 wt% GNP Strength (MPa)
0	78.0 ± 2.2 n=4	81.2 ± 7.2 n=5	84.6 ± 4.5 n=5
50	76.9 ± 1.3 n=4	77.9 ± 7.1 n=4	78.3 ± 1.3 n=4
100	76.4 ± 0.8 n=4	78.9 ± 3.7 n=4	80.7 ± 3.1 n=3
200	76.1 ± 2.3 n=4	79.7 ± 1.5 n=3	84.1 ± 7.0 n=4
400	77.4 ± 0.5 n=4	74.6 ± 5.1 n=4	78.6 ± 1.9 n=4

5.4.4 Glass Transition Temperature (T_g) Results

The glass transition temperatures measured in the DMA tests are shown in Figure 5.6 and Table 5.3, ± 1 standard deviation over the multiple specimens measured for each exposure time interval (some error bars are hidden by the data points). Additional DMA data can be found in Appendix 8.14. The data demonstrates about a 1-2% drop in the

glass transition temperature for the three material systems with the first increment of aging. Thereafter, the glass transition temperature remains relatively constant for higher levels of aging. Starkova et al. [11] observed a much more drastic drop in glass transition of neat DEGBA resin and DEGBA/TRGO composites with aging. The data in Figure 5.6 suggests that GNP loading does not strongly affect the glass transition in the unaged material (similar to trends observed in other epoxy nanocomposites [11, 13, 19, 22]). However, for nearly all of the aged specimens there appears to be an increase in glass transition with addition of GNP into the epoxy. Thus, it appears that the epoxy/GNP interface can retain load transfer capability during the aging process despite the effects of moisture plasticity on the epoxy resin.

Figure 5.6: Glass Transition Temperature for GNP/Epoxy Nanocomposites

Table 5.3: Glass Transition Temperature for GNP/Epoxy Nanocomposites

Aging Time (hr)	Neat Epoxy T_g (°C)	2 wt% GNP T_g (°C)	4 wt% GNP T_g (°C)
0	231.4 ± 0.1 n=3	231.6 ± 0.3 n=3	230.8 ± 0.2 n=3
50	226.3 ± 0.1 n=3	228.2 ± 0.4 n=3	228.1 ± 0.5 n=3
100	227.1 ± 0.1 n=3	227.1 ± 0.2 n=3	227.7 ± 0.3 n=3
200	225.9 ± 0.2 n=3	227.3 ± 0.4 n=3	227.4 ± 0.5 n=3
400	226.7 ± 0.4 n=3	227.6 ± 0.3 n=3	228.0 ± 0.6 n=4

5.5 Conclusions

The results of this study indicate that GNP reinforcement provides increases in the magnitude of stiffness regardless of the aging level and increases in material strength for aging times less than 400 h. The results also suggest that GNP reinforcement provides increases in the glass transition temperature relative to the neat epoxy for hygrothermally aged specimens. However, despite these improvements, the composite material strengths were compromised at 400 h and the overall magnitude of glass transition of the composite and neat resin materials was reduced relative to the unaged state. Therefore, it follows that the detrimental effects of epoxy moisture plasticity are partially mitigated by the load transfer capability of GNP, except in the case of the 400 h aging time, where the composite strength drops and the neat resin strength remains unaffected.

It is also important to note that the neat CE resin shows a remarkable resistance to hygrothermal aging relative to the DEGBA resin reported by Starkova et al. [11]. While the CE resin flexural modulus dropped about 4% between the unaged and 400 h aging

conditions, the DEGBA resin storage modulus dropped about 19% at 30 °C. In addition, while the glass transition temperature of the CE resin dropped 2% after aging, that of the DEGBA resin dropped 61%. Therefore, regardless of the GNP reinforcement loading level, the CE resin performs better under hygrothermal aging conditions than the DEGBA resin.

5.6 Future Work

This study assessed the effects of hygrothermal aging at 100 %RH and 50 °C. For ACCC and other outdoor applications, other aging mechanisms also affect the properties and lifetime of the composite. It would be useful to test the effects of adding GNP on the aging process for long-term mechanical fatigue, temperature cycling, ozone, and UV effects. The results could be compared to previous work done without GNP fillers on the same or similar epoxy systems [1-5].

For high voltage applications, several tests could be used to characterize the composite's response to high electrical field exposure. Arc resistance, dissipation factor, dielectric strength, and dielectric constant could be tested to determine the effects of adding small amounts of the conductive GNP.

5.7 Acknowledgements

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6. Accelerated Hygrothermal Aging of Talc/Cycloaliphatic Epoxy Composites⁵

6.1 Abstract

Cycloaliphatic epoxy (CE) systems are resistant to many aging mechanisms, which make them useful in applications with harsh environments. Reinforcement has been used in epoxy systems to decrease water absorption and improve aging resistance, thus it is expected that talc would benefit CE systems in a similar manner. Neat, 10, and 20 wt% talc/CE composites were aged at 50 °C and 100 % relative humidity for up to 398 hours. Three-point bend testing and dynamic mechanical analysis were performed on the aged and unaged samples. Talc reduced the amount of water absorbed in the composites significantly, and increased the composite flexural stiffness at all aging levels. Talc does not appear to mitigate the negative effects of aging on flexural strength or glass transition temperature (T_g).

⁵ The material contained within this chapter has been submitted to the journal “*Polymer Composites*.” The Future Work section was added for this document. See Appendix 8.18 for copyright agreement and permissions.

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6.2 Introduction

Cycloaliphatic epoxies (CE) have shown greater resistance to many aging mechanisms relative to other epoxies, including hydrothermal, UV, and ozone aging mechanisms [1-3]. This aging resistance is desirable for CE-based composite materials used in outdoor applications or other harsh environments. One potential application of CE composites is as the core reinforcement in aluminum conductor composite core (ACCC) power transmission lines, which consist of aluminum strands for conduction wound around an electrically insulating, stiff carbon fiber and glass fiber/epoxy composite [4, 5]. ACCC lines (and thus the composite core) must withstand harsh environmental conditions and elevated temperatures, as well as mechanical fatigue and high electrical field exposure over extended periods.

The effects of reinforcement on epoxy aging have been studied with various clay and graphene reinforcements [3, 5-7]. In general, these particles decrease the amount of water absorbed during hygrothermal aging, which could decrease the effects of water on the mechanical properties of the epoxy matrix, and more specifically, the CE composite core of ACCC lines.

Talc is an inexpensive filler that may have beneficial effects in epoxy composites. Talc is hydrated magnesium silicate with the formula $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$. As a filler, it increases the thermal conductivity of polymers and is electrically insulating [8]. Talc has

often been used to increase the stiffness of thermoplastic composites, but there is little information on the effects of talc in thermosets [9-13]. Adding talc to CE in an ACCC power transmission line could increase composite stiffness and thermal conductivity while still maintaining the desired electrically insulating properties needed for the composite core. Talc exhibits hydrophilic behavior in some situations, and hydrophobic in others [14]. The hydrophobic and hydrophilic properties indicate that talc could have interesting impacts on the aging response of talc/epoxy composites. Talc has also been shown to decrease aging effects of UV radiation on compressive strength in a concrete repair composite containing epoxy [11].

The goal of this study is to determine the effects of talc reinforcement on the mechanical properties of hygrothermally aged CE composites. Samples containing 0, 10, and 20 wt% talc were fabricated, aged, and tested for flexural properties and glass transition temperature at 0, 50, 100, 200, and 398 hours of aging in 50 °C and 100 % relative humidity. To the authors' knowledge, no studies of aging effects have been done on talc in CE.

6.3 Materials and Experimental Methods

6.3.1 Materials

The CE-based epoxy used in this study consisted of a CE resin (3,4-epoxycyclohexylmethyl-3,4-epoxycyclohexylcarboxylate) cured by an anhydride (4,7-

methanoisobenzofuran-1,3-dione-3a,7,7a-tetrahydromethyl). The chemical structures of these monomers are shown in Figure 6.1. A Brookfield viscometer at 25 °C was used to measure a viscosity of 5.2 Poise for the mixture of 100 g of resin and 138 g of curing agent. Curing is initiated at elevated temperatures when a hydroxyl group catalyst opens the anhydride ring, creating a carboxyl group. The carboxyl group opens the epoxide ring on the CE monomer, creates a chemical crosslink between the CE and anhydride monomers, and creates a new hydroxyl group, which initiates further reactions with other epoxide rings or anhydride rings. The resulting CE/anhydride crosslinks propagate to form a solid product.

Figure 6.1: Monomer Molecules used for Solid Epoxy Matrix: a) Cycloaliphatic Epoxy Resin, and b) Anhydride Curing Agent Hardener

The talc used in this study was Nicron® 503 supplied by Imerys. The median particle diameter of the >98% pure talc (with <2% dolomite and chlorite) is 6 microns, and the particle specific gravity is 2.8 [8]. Talc has a thermal conductivity of 2.1 W/m-K, and is electrically insulating [15].

6.3.2 Test Specimen Fabrication

For the fabrication of neat epoxy plaques, a multi-part mold was coated with Mann Ease Release 300, then assembled and pre-heated in a vacuum oven at 100 °C. Resin and hardener were poured in a 100 g to 138 g ratio into 5 mixing cups of 90 g each, for a total batch size of 450 g. Each cup was mixed in a FlackTek SpeedMixer™ DAC 150.1 FVZ for 2 minutes at 2500 rpm. The SpeedMixer™ uses centrifugal forces to mix by spinning a cup containing the material one way while rotating a base holding the cup the opposite way. After mixing, the mixture was degassed at 100 °C and 29 inHg, alternating vacuum with atmospheric pressure to avoid overflow, until no more bubbles appeared. The mixture was then poured into the pre-heated mold and degassed once more. The curing cycle used was 100 °C for an hour, then heated to 200 °C at 2 °C/min, then held at 200 °C for two hours. The oven was then turned off and the cured epoxy was allowed to cool in the oven to room temperature at an approximately 1 °C/min cooling rate.

For the fabrication of 10 and 20 wt% talc/epoxy plaques, a multi-part mold was coated with Mann Ease Release 300, then assembled and pre-heated in a vacuum oven at 100 °C. Master batches of 50 wt% talc were fabricated by adding talc on top of CE resin in a mixing cup. The CE and talc were mixed for a total of 1.5 minutes at 2500 rpm in the SpeedMixer™. Mixing was performed for 30 seconds and then 1 minute, allowing

cooling between mixing so that the temperature of the master batch stayed under 50 °C. To achieve the desired amount of filler in the epoxy, the resin, master batch, and hardener portions were poured into mixing cups and mixed at 2500 rpm for 2 min in the SpeedMixer™. The cups were degassed at 100 °C by cycling between vacuum and atmospheric pressure for about 35 minutes until bubbles stopped forming. The cups were poured into the mold, and degassed once more. The epoxy was cured at 100 °C for an hour, then heated to 200 °C at 2 °C/min, then held at 200 °C for two hours. The oven was turned off and the cured epoxy was allowed to cool in the oven to room temperature at about 1 °C/min cooling rate.

A set of three-point bend and dynamical mechanical analysis (DMA) specimens were machined from the fabricated plaques. The three-point bend specimens had dimensions of 127 mm × 12.7 mm × 3.2 mm per ASTM D790, and the DMA single-cantilever beam specimens had dimensions of 35 mm × 12.7 mm × 3.2 mm.

6.3.3 Aging and Moisture Gain Measurement Method

A QUV Accelerated Weathering Testing Machine was used to condition the specimens under a controlled temperature of 50 °C and a relative humidity of 100 %. For all three material systems, both three-point bend and DMA specimens were exposed to these conditions for 0, 50, 100, 200, and 398 hours. Before aging, all specimens were conditioned at 50 % relative humidity and 23°C for two days.

During the aging process, mass measurements were made on the three-point bend specimens at regular intervals to monitor the moisture uptake. For each mass measurement, the specimens were quickly removed from the weathering chamber and sealed in a moisture barrier bag to cool to room temperature (10 minutes). After cooling, the specimens were removed from the bags, excess surface moisture removed, and mass measurements performed. The specimens were returned to the weathering chamber immediately after measuring.

For the three-point bend specimens of each material system (neat, 10 wt%, 20 wt% talc), 5 specimens were left unaged, 5 specimens aged for 50 hours, 5 aged for 100 hours, 5 aged for 200 hours, and 5 were aged for 398 hours. For the DMA specimens for each material system, 4 specimens were aged for each amount of time (unaged, 50 hours, 100 hours, 200 hours, and 398 hours). After aging was complete, each specimen was immediately removed from the chamber and patted dry. The specimens were promptly sealed in moisture barrier bags and allowed to cool to room temperature before testing. Due to limited space in the QUV chamber, 24 additional three-point bend specimens of each formulation were aged using the same procedure in a separate run, and tested for moisture gain only. The additional neat specimens were also tested for flexural properties.

6.3.4 Environmental Scanning Electron Microscope (ESEM) Test Method

A Philips XL 40 Environmental Scanning Microscope was used to image the talc/CE. Samples were prepared by mounting a 3 mm thick x 12 mm long x 12 mm wide talc/CE sample in a cast epoxy puck. The composite surface was ground using 320 grit, then 600 grit, and lastly 1,200 grit sandpaper. The sample was then polished on a Buehler Ecomet 4 variable speed grinder-polisher using 9 micron diamond suspension, then 3 micron diamond suspension, then 1 micron alumina/water slurry, and finally 0.05 micron alumina/water slurry. Between each step, samples were rinsed and sonicated to remove all polishing media. Polished samples were coated with 20 nm of carbon by a Denton DV-502A High Vacuum Evaporator. The composites were imaged on the ESEM at 15 kV accelerating voltage and 10 mm working distance. The backscattering detector was used so that the talc would stand out against the polymer.

6.3.5 Three-Point Bend Test Method

The three-point bend tests were performed per the specifications of ASTM D790 with a crosshead rate of 5.3 mm/min on an Instru-Met Sintech screw-driven mechanical testing machine with an Epsilon LVDT to record specimen deflection. The flexural modulus and flexural strength values were calculated based on the load-deflection data using ASTM D790.

6.3.6 Dynamic Mechanical Analysis (DMA) Test Method

The DMA tests were performed on a TA Instruments Q800 DMA using the single cantilever clamp fixture. An oscillating amplitude of 30 μm was applied to the specimens, with an initial temperature of 50 °C with a soak time of 5 minutes, a temperature ramp rate of 3 °C/min, and a final temperature of 290 °C with a hold time of 5 minutes. The frequency was held at 1 Hz for the entire duration of each test. Once the tests were complete, the data was analyzed to determine the storage modulus, loss modulus, and tan delta values for each specimen. The glass transition temperatures were measured at the peak of the tan delta values and the peak of loss modulus for each material system and aging level.

6.4 Results

6.4.1 Environmental Scanning Electron Microscopy (ESEM) Results

Figure 6.2 shows a representative ESEM image of a polished surface of the talc/epoxy composite material. This image indicates good dispersion. The talc flakes are oriented parallel, perpendicular, and at angles with respect to the polished surface, indicating that the talc is randomly oriented in three dimensions in the epoxy.

Figure 6.2: Scanning Electron Microscopy Image of a Polished Surface of the Talc/Epoxy Composite

6.4.2 Moisture Content Results

Figure 6.3 shows the moisture content in the talc/epoxy composites at varying aging times. In all graphs, error bars represent ± 1 standard deviation. Moisture gain data can also be found in Appendix 8.15. The moisture content increased up to about 200 hours then slowed or leveled out for all talc contents. The composites containing talc had lower moisture content at all levels of aging. The final moisture content was 3.2 wt% for the neat epoxy compared to 2.1 wt% for the 20 wt% talc composite. This is a dramatic improvement over previous aging research using graphene nanoplatelets as a filler, where there were decreased moisture contents in the filled samples at shorter aging times, but small/no differences at saturation [3]. Starkova et al. observed more

significant impacts with thermally reduced graphene oxide (TRGO); water absorption decreased from about 5 wt% for unfilled epoxy to about 2 wt% for up to 0.5 wt% TRGO [6]. The talc in this study limits the absorption of water more than is reported for clay fillers in epoxy, which either had no effect or an increase in water content with increasing filler content [7, 16].

Figure 6.3: Water Mass Gain for Talc/Epoxy Composites

6.4.3 Flexural Modulus Results

Figure 6.4 and Table 5.1 show the flexural modulus in the talc/epoxy composites at varying aging times, ± 1 standard deviation. Appendix 8.16 shows detailed flexural data. The addition of talc increased flexural modulus for all aging times, from 3.2 GPa for the neat epoxy to 4.1 GPa for the 20 wt% talc/epoxy composite. This is expected for reinforcement in epoxy [3, 6, 16] and for talc in polymers in general [13, 17, 18]. The

trend from this data indicates that higher talc content leads to higher flexural modulus, but other studies with similar filler and epoxy systems indicate there may be an optimal amount of talc above 20 wt% after which the modulus decreases [10, 16, 19, 20]. For all levels of talc, hygrothermal aging does not affect the flexural modulus. In a previous study with the same epoxy system and aging procedure, flexural modulus decreased slightly after 200 hours of aging [3]. There is not a similar drop in modulus with talc as the filler, which indicates that the talc/epoxy composite is more resistant to aging effects on modulus than graphene/epoxy.

Figure 6.4: Flexural Modulus for Talc/Epoxy Composites

Table 6.1: Flexural Modulus for Talc/Epoxy Composites

Aging Time (hr)	Neat Epoxy Modulus (GPa)	10 wt% Talc Modulus (GPa)	20 wt% Talc Modulus (GPa)
0	3.22 ± 0.06 n=7	3.49 ± 0.21 n=5	4.01 ± 0.20 n=5
50	3.15 ± 0.11 n=10	3.47 ± 0.19 n=4	4.09 ± 0.06 n=5
100	3.19 ± 0.08 n=11	3.46 ± 0.09 n=4	4.05 ± 0.04 n=4
200	3.19 ± 0.07 n=10	3.46 ± 0.07 n=5	4.10 ± 0.13 n=5
400	3.18 ± 0.04 n=4	3.55 ± 0.05 n=5	4.10 ± 0.10 n=5

6.4.4 Flexural Strength Results

Figure 6.5 and Table 6.2 show the flexural strength of the talc/epoxy composites with respect to aging time, ± 1 standard deviation. At all aging levels, the addition of talc decreased the flexural strength by about 30 MPa for 20 wt% talc. In polypropylene, talc has been shown to both increase and decrease the strength, possibly depending on interface quality [8, 9, 17]. The talc used in this study was not treated to improve the interface, so it makes sense that adding talc would decrease the strength. Aging decreased the strength at all talc levels, then stopped changing at around 200 hours. This could be related to water absorption, which also leveled out around 200 hours. Since a similar trend is followed for all levels of talc, it appears that the addition of talc does not change the aging effects on the flexural strength. In a previous study with graphene in the same CE, aging only caused a slight drop in strength in graphene/epoxy composites at 400 hours of aging [3]. The decrease did not appear in the neat epoxy, so the drop in strength was attributed to degradation of the interface [3]. The talc/epoxy interface in this study did not increase strength, so any degradation did not affect the composite's strength.

Figure 6.5: Flexural Strength for Talc/Epoxy Composites

Table 6.2: Flexural Strength for Talc/Epoxy Composites

Aging Time (hr)	Neat Epoxy Strength (MPa)	10 wt% Talc Strength (MPa)	20 wt% Talc Strength (MPa)
0	114.7 ± 5.7 n=7	96.6 ± 5.2 n=5	83.3 ± 5.6 n=5
50	107.0 ± 13.1 n=10	92.4 ± 6.8 n=4	79.8 ± 2.8 n=5
100	98.1 ± 13.3 n=11	82.3 ± 5.9 n=4	70.7 ± 3.0 n=4
200	105.3 ± 10.7 n=10	79.6 ± 4.4 n=5	66.1 ± 7.1 n=5
400	103.4 ± 19.1 n=4	71.5 ± 9.9 n=5	68.9 ± 5.1 n=5

6.4.5 Glass Transition Temperature (T_g) Results

Figure 6.6, Figure 6.7, and Table 6.3 show the glass transition temperatures (T_g) based on the DMA results for the talc/epoxy composites, ± 1 standard deviation. Appendix 8.17 shows detailed DMA data. The T_g decreased with aging for all samples; about 5 °C based on the tan delta peak, or 30 °C based on the storage modulus onset at 398 hours of

aging. In Figure 6.7, talc decreases the T_g in unaged samples. In the aged samples, aging time is the dominant effect, and the presence of talc has little effect on the aging response. For other filler/epoxy systems, the filler has shown little effect on the T_g with respect to aging time [5, 21-23]. By both measurements (tan delta peak and storage modulus onset), the T_g appears to still be decreasing at 398 hours of aging, well after the water absorption has slowed or stopped, around 200 hours. This indicates that the T_g change is not based on water content alone, and is likely due to other aging mechanisms, such as physical aging [24]. The decrease in T_g with increased aging time is expected, based on other epoxy aging studies [6, 21, 22]. Starkova et al. reported a drop from 148 °C for unaged DGEBA based resin and anhydride curing agent to 57 °C after 2 weeks immersed in 80 °C water [6]. Fernandez-Garcia et al. observed a T_g drop of 12 °C for every 1 wt% water absorbed in their particle-filled epoxy-based adhesive film, regardless of aging temperature and cyclical desorption/reabsorption, with T_g measured by DSC [21]. Ivanova et al. show a similar T_g drop of 13 °C for every 1 wt% water regardless of desorption/reabsorption in their rubber-toughened DGEBA-based epoxy, filled with fumed silica and calcium silicate [22]. Since the drop in T_g observed in the talc/CE composite in this paper is constant regardless of moisture content, which varies from 2.1 wt% to 3.2 wt%, it is clear that there are factors other than moisture content affecting T_g in this system. In some literature cases where the aging temperature was near the curing temperature, the T_g increased due to post-curing increasing the crosslink density, but the relatively low aging temperature in this study did not have this effect [5, 23]. In a previous study with the same CE, the same 5 °C drop in T_g was observed based

on the tan delta peak, and the addition of graphene did not have a major effect on the T_g [3]. Neither talc nor GNP mitigated the effects of aging on glass transition temperature.

Figure 6.6: Glass Transition Temperature determined by Tan Delta Peak for Talc/Epoxy Composites

Table 6.3: Glass Transition Temperature determined by Tan Delta Peak for Talc/Epoxy Composites

Aging Time (hr)	Neat Epoxy T_g (°C)	10 wt% Talc T_g (°C)	20 wt% Talc T_g (°C)
0	250.6 ± 1.0 n=4	254.5 ± 2.0 n=4	250.7 ± 1.5 n=3
50	248.2 ± 0.9 n=4	252.0 ± 1.4 n=4	247.0 ± 1.9 n=3
100	248.6 ± 1.0 n=4	247.5 ± 1.7 n=3	247.0 ± 2.0 n=3
200	246.5 ± 1.0 n=4	249.4 ± 2.0 n=3	247.9 ± 0.5 n=4
400	245.8 ± 1.0 n=4	247.8 ± 2.0 n=4	247.2 ± 0.2 n=4

Figure 6.7: Glass Transition Temperature determined by Storage Modulus Onset for Talc/Epoxy Composites

6.5 Conclusions

For the talc/CE system examined in this study, the addition of talc reduced the water absorption into the composite at all aging times up to 398 hours. Talc also increased the flexural stiffness over the neat epoxy. Aging did not affect the stiffness for any of the talc loading levels, which indicates that the stiffness of the CE is resistant to hygrothermal aging. This combination of increased flexural stiffness and resistance to aging could be useful in the composite core of ACCC power transmission lines. Both the addition of talc and increased aging time decreased the flexural strength of the

composites. Talc did not appear to influence the negative effect of aging on the flexural strength of the composites, either positively through reduced water absorption or negatively through the increased number of interfaces. Talc also did not increase the T_g of the composites at any aging time. Per the authors' knowledge, aging studies of talc in CE have not been previously reported in the open literature.

6.6 Future Work

This study assessed the effects of hygrothermal aging at 100 %RH and 50 °C. For ACCC and other outdoor applications, other aging mechanisms also affect the properties and lifetime of the composite. It would be useful to test the effects of adding talc on the aging process for long-term mechanical fatigue, temperature cycling, ozone, and UV effects. The results could be compared to previous work done without particle fillers on the same or similar epoxy systems [1, 2, 4].

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7. Conclusions and Future Work

7.1 Conclusions

This work focuses on two separate applications and composite materials. A polyetheretherketone (PEEK) based composite is studied for aerospace applications, and a cycloaliphatic epoxy (CE) based composite is studied for high voltage applications. To the author's knowledge, properties of these composites had not been previously reported in the open literature prior to these experiments.

7.1.1 *PEEK Conclusions*

PEEK composites containing graphene nanoplatelets (GNP), carbon black (CB), and chopped carbon fiber (CF) fillers were produced and tested for their tensile, thermal, and electrical properties with the aim of developing conductive composites that could be used in aerospace applications. For the aerospace applications targeted here, thermal conductivity, electrical conductivity, and high stiffness and strength are desirable. The percolation threshold (the point at which electrical conductivity increases rapidly) was found to be around 13 wt% (9 vol%) GNP, around 3 wt% (2 vol%) CB, and between 10 wt% (7.4 vol%) and 20 wt% (15.3 vol%) CF for each single filler. The highly branched structure of the Akzo Nobel Ketjenblack CB used in this study likely causes the low percolation threshold for CB. The most conductive composite produced in this study was 7.5 wt% CB/30 wt% CF/PEEK which had an electrical resistivity (ER) of 0.56 ohm-cm.

This electrically conductive composite can serve in electromagnetic and radio frequency interference applications ($ER < 10^0$ ohm-cm), which is especially useful to protect electrical components from electromagnetic interference in aerospace structures. There were six composites produced that could be used for semiconductive applications ($ER 10^1$ to 10^2 ohm-cm):

- 30 wt% CF (23.6 vol% CF)
- 5 wt% CB (3.7 vol% CB)
- 7.5 wt% CB (5.5 vol% CB)
- 10 wt% CB (7.4 vol% CB)
- 2.5 wt% CB/30 wt% CF (2.0 vol% CB/23.8 vol% CF)
- 5.0 wt% CB/30 wt% CF (4.0 vol% CB/24.0 vol% CF)

Four composites were identified for electrostatic dissipative applications ($ER 10^3$ to 10^{10} ohm-cm), that can be used in aerospace applications to protect electrical components from static electricity:

- 15 wt% GNP (10.3 vol% GNP)
- 20 wt% CF (15.3 vol% CF)
- 2.5 wt% CB/10 wt% CF (1.9 vol% CB/7.5 vol% CF)
- 2.5 wt% CB/20 wt% CF (1.9 vol% CB/15.4 vol% CF)

The highest thermal conductivity reported in this article is 0.52 W/m-K for 7.5 wt% CB/30 wt% CF/PEEK, which is about double the thermal conductivity of the neat PEEK (0.25 W/m-K). Higher thermal conductivity causes a material to work as a better heat sink, rather than allowing hot spots to compromise the component. All fillers increased tensile modulus. The carbon fiber increased modulus and strength far more than GNP or CB, due to its higher aspect ratio. Composites containing ≤ 10 wt% GNP maintained their tensile strength and strain, while CB composites and higher wt% GNP composites experienced a small decrease in tensile strength. High specific stiffness and strength are important for lighter components, one of the main benefits of composite materials. The composites produced here with increased stiffness, strength, and electrical conductivity can be used in a variety of aerospace applications.

7.1.2 CE Conclusions

Fabrication methods were developed, and tensile, thermal, and electrical properties were tested for carbon black (CB), fumed silica, talc, and glass microsphere (glass MS) fillers in CE. High voltage applications such as power transmission line cores were targeted. For these applications, the composites should have a high electrical resistivity (ER), thermal conductivity, and tensile modulus. The current design for these power lines has a fiber and epoxy core without any particulate fillers, so any improvement in the properties due to the fillers indicates an improvement over the current design. The following fillers and amounts were made and tested in CE:

- Carbon Black (CB):
 - CB1 (low surface area): 0.5 (0.33), 1 (0.67), 2 (1.34) wt% (vol%)
 - CB2 (mid surface area): 1 (0.67) wt% (vol%)
 - CB3 (high surface area): 1 (0.67) wt% (vol%)
- Fumed Silica: 1 (0.55), 2 (1.1), 3 (1.65), 4 (2.2) wt% (vol%)
- Talc: 5 (2.2), 10 (4.6), 15 (7.1), 20 (9.8) wt% (vol%)
- Glass MS: 5 (2.5), 10 (5.1), 20 (10.9), 30 (17.3), 40 (24.5) wt% (vol%)

ER remained high for all filler levels tested in CE. In the targeted power transmission line applications, electrical resistivity is necessary so that the electricity flows through the aluminum conductors instead of the supporting core. Adding up to 2 wt% of Cabot CB1 or up to 4 wt% of Cabot fumed silica to this cycloaliphatic epoxy does not appreciably affect the ER, thermal conductivity, tensile properties, or glass transition temperature. For talc/CE and glass MS/CE composites, ER remained high as expected, and thermal conductivity increased from 0.15 W/m-K (neat CE) to about 0.25 W/m-K for 20 wt% talc or 40 wt% glass MS in CE. An elevated thermal conductivity is beneficial in these applications, as it allows heat to dissipate better, minimizing hot spots in the core. The tensile strength and strain of the talc/CE composites remained relatively constant up to 20 wt% talc at around 50 MPa and 2 %. Glass MS/CE composites had a reduced tensile strength and strain, dropping to around 40 MPa and 1 % for 5 to 40 wt% glass MS. Both talc and glass MS increased the composite tensile modulus from 2.7 GPa for the neat epoxy to 3.6 GPa for 20 wt% talc and 5.2 GPa for 40 wt% glass MS. An

increase in the tensile modulus is desired for improving the stiffness of the composite core in power transmission lines. These experimental values compared well with established micromechanical models. Specifically, the 3D Halpin-Tsai model fits the talc/CE tensile modulus data the best. The Nielsen model with modified ψ term fits the glass MS/CE results the best up to 30 wt% (17 vol%) glass MS. The modified ψ term was developed for glass sphere/epoxy composites [1]. All composites produced here maintained a high electrical resistivity, so all fillers and levels could be used in power transmission line cores. Talc and glass MS composites are especially promising, because they also increased thermal conductivity and tensile modulus.

Graphene nanoplatelet (GNP)/CE and talc/CE composites were aged at 50 °C and 100 % relative humidity for 400 hours, and tested for flexural properties and glass transition temperature (T_g). For power transmission line applications, high stiffness, high T_g , and resistance to hygrothermal aging are targeted. The following fillers and amounts were made and tested, along with the neat CE:

- GNP: 2 (1.2), 4 (2.4) wt% (vol%)
- Talc: 10 (4.6), 20 (9.8) wt% (vol%)

GNP slightly decreased the rate of water absorption, but had the same equilibrium value of about 2 wt% water gain for the neat CE up to 4 wt% GNP in CE. Up to 20 wt% talc reduced the water absorption at all aging times. Maintaining properties during aging

processes is important to a long product lifetime. Both GNP and talc increased the composite flexural stiffness at all aging levels. The higher stiffness from both fillers would improve the stiffness of the composite core in power transmission lines. The stiffness was not greatly affected by aging time in either study, which indicates that this CE is resistant to hygrothermal aging effects on modulus regardless of filler content. GNP increased the flexural strength for aging times less than 400 hours, while talc decreased the flexural strength at all aging times. The difference between the effects of the two fillers is likely due to the difference in interface quality and resulting load transfer capabilities. The flexural strength for both types of composites decreased slightly with aging time. In both studies, aging decreases the glass transition temperature (T_g) more than any positive effect from the fillers. In the GNP/CE composites, the presence of GNP in aged samples appears to slightly reduce the drop in T_g due to aging. A high glass transition temperature (T_g) is desired in order to support the power line at a high operating temperature, which is raised when transmitting more electricity through the same size line. The decrease noted for both fillers leaves the T_g (determined by tan delta peak) well above the current maximum operating temperature of 180 °C for composite core power transmission lines with no fillers. [2]. Both GNP and talc are good candidates for use in power transmission line cores.

7.2 Future Work

7.2.1 PEEK Future Work

The conductive PEEK composites with ER under 50 ohm-cm showed promise for use in shielding effectiveness applications. The shielding effectiveness should be tested for these conductive samples: 30 wt% CF/PEEK, 5 wt% CB/PEEK, 7.5 wt% CB/PEEK, 10 wt% CB/PEEK, 2.5 wt% CB/30 wt% CF/PEEK, 5 wt% CB/30 wt% CF/PEEK, 7.5 wt% CB/30 wt% CF/PEEK. Shielding effectiveness is measured as the difference between an electromagnetic signal's intensity before shielding and after shielding, as described in ASTM D4935. Conductive materials can shield electronics from electromagnetic and radio frequencies, which is beneficial in structural components that house electronic components.

In this study, the percolation threshold for CF in PEEK was determined to be between 10 and 20 wt%. By making composites between 10 and 20 wt% CF, a narrower range for the percolation threshold could be found.

It is possible that greater carbon fiber content would continue to increase the tensile modulus and strength, thermal conductivity, and electrical conductivity. The materials used here limited the composites to 30% CF or lower, but if more carbon fiber from a different source was added, improved properties might be achieved.

7.2.2 CE Future Work

Talc/CE and GNP/CE composites were tested when aged at elevated temperature and humidity. CB, Si, and glass MS could also be tested at elevated temperature and humidity, and compared with the talc and GNP composites. For outdoor applications like power transmission lines, other factors affect the properties and lifetime of the composites. UV radiation, ozone exposure, and temperature cycling effects could be investigated for these composites to determine further benefits or drawbacks of using these fillers. Some of these tests have already been done on the same or similar epoxy without fillers, and would be good to compare [3-7].

For these electrically resistive composites, many other tests could help to describe their electrical properties. Dielectric strength, dielectric constant, dissipation factor, capacitance, and arc resistance could provide more information on these composites and the electrical effects of the different filler types and amounts.

The target application for these composites is part of a power transmission line, used with carbon fiber and glass fiber as a support, and surrounded by aluminum conductors. Therefore, it would be useful to test the filled epoxy with the fibers and aluminum that will be used in the final power lines. Testing the mechanical, thermal, and electrical

properties of the system as a whole would be important before implementing it, so that the effects of the continuous glass and carbon fibers and the presence of an electric field on the composite can be understood.

7.3 References

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8. Appendix

8.1 PEEK Extrusion and Injection Molding Conditions

Michigan Technological University 2/5/2015

Figure 8.1: Extruder Screw Design named 2-5-15

Michigan Technological University July 8, 2015

Figure 8.2: Extruder Screw Design named 7-8-15

Table 8.2: Extrusion Conditions for Solvay KetaSpire KT-880 NT with Asbury Carbons T307

GNP (continued from Table 8.1)

Material Number	VD8K	VD10K	VD15K
Extrusion Date	3-19-15	3-19-15	8-25-15
Extruder Screw Name	2-5-15	2-5-15	7-8-15
Extruder RPM	120	120	150
Motor Amperage, %	56	60	62
Melt Temperature, °C	408	408	400
Melt Pressure, psig	90	120	220
#3 Feeder Setting, lb/hr	13.8	13.5	12.75
Material in Feeder #3	KetaSpire KT-880 NT	KetaSpire KT-880 NT	KetaSpire KT-880 NT
K-Tron Feeder Setting, lb/hr	1.2	1.5	2.25
Material in K-Tron Feeder	TC307 GNP	TC307 GNP	TC307 GNP
Vacuum Port	1 atm	1 atm	1 atm
Feed Section Temperature	H ₂ O Cooled	H ₂ O Cooled	H ₂ O Cooled
Zone 1 Temperature, °C	300	300	300
Zone 2 Temperature, °C	365	365	375
Zone 3 Temperature, °C	370	370	380
Zone 4 Temperature, °C	375	375	375
Zone 5 Temperature, °C	380	380	375
Zone 6 Temperature, °C	380	380	375
Zone 7 Temperature, °C	375	375	375
Zone 8 Temperature, °C	375	375	375
Zone 9 Temperature, °C	375	375	375
Zone 10 Temperature, °C	370	370	370 (actual 350C)
Die Type and Gap	2 x 3 mm dia	2 x 3 mm dia	2 x 3 mm dia
Pelletizer Setting	H ₂ O bath @80F	H ₂ O bath @80F	H ₂ O bath @80F
Output Rate, lb/hr	15	15	15

Notes:

- 1) The Asbury TC307 GNP was used as received.
- 2) Feeder 3 helix: 0.5" open helix with end stub and 0.75" nozzle side discharge: ran on automatic on loss in weight on laptop.
- 3) Dyna- Purge E used at end of the extrusion runs to clean out extruder.
- 4) Extruder was cleaned in sand bath before March 12, 2015 so we started out with a totally clean extruder screw.
- 5) Per Sabic, keep T melt < 405 °C.
- 6) Compressed air was used to cool zones 2 to 9. It was too hot to use water for cooling on these zones. Used fiberglass insulation on the die end of the extruder. Used on KTron feeder used 20 mm concave fine twin screws for 0.3 to 0.9 lb/hr TC307. Used 20 mm concave coarse twin screws for > 0.9 lb/hr TC 307. Concave coarse screws work fine also for 0.9 lb/hr TC307.
- 7) On July 7, 2015 we tried to extrude VD15K at 15 lb/h total flow of KT-880 NT and TC 307 and zone 1 'piled in to a hill of TC307 and polymer pellets. So VD10K is the max we can do with this 2-5-15 screw design and flow rates.
- 8) On Aug 25, 2015, we used extruder screw named 7-8-15 and all went well. There was no fiberglass insulation on the extruder die end.

Table 8.3: Extrusion Conditions for Solvay KetaSpire KT-880 NT with Akzo Nobel

Ketjenblack EC-600JD Carbon Black

Material Number	VA 2.5K	VA5K
Extruder Screw Design	8-27-15	8-27-15
Extrusion Date	2-16-16	2-16-16
Extruder RPM	250	250
Motor Amperage, %	73	64
Melt Temperature, °C	391	395
Melt Pressure, psig	260	300
#3 Feeder Setting, lb/hr	29.25	19.0
Material in Feeder #3	KetaSpire KT-880 NT	KetaSpire KT-880 NT
KTron Feeder Setting, lb/hr	0.75	1.0
Material in Feeder #1	EC-600 JD	EC-600 JD
Vacuum Port	1 atm	1 atm
Zone 5 side stuffer rpm	300	300
Material in Zone 5	EC-600 JD	EC-600 JD
Zone 7 side stuffer rpm	Closed port	Closed port
Material in Zone 7	nothing	nothing
Feed Section Temperature	H ₂ O Cooled	H ₂ O Cooled
Zone 1 Temperature, °C	300	300
Zone 2 Temperature, °C	375	375
Zone 3 Temperature, °C	380	380
Zone 4 Temperature, °C	375	375
Zone 5 Temperature, °C	375	375
Zone 6 Temperature, °C	375	375
Zone 7 Temperature, °C	375	375
Zone 8 Temperature, °C	375	375
Zone 9 Temperature, °C	375	375
Zone 10 Temperature, °C	370	370
Die Type and Gap	2 x 3 mm dia	2 x 3 mm dia
Pelletizer Setting	Water bath at 80F	Water bath at 80F
Output Rate, lb/hr	30 smooth surface	20 smooth surface

Notes:

- 1) The EC 600 JD carbon black was used as received.
- 2) Feeder 3 helix: 0.5" open helix with end stub and 0.75" nozzle side discharge: ran on automatic on loss in weight on laptop. Want mode = OP so can run on laptop. Toshiba laptop called feeder3zytel101jak5'02.par.
- 3) Extruder was taken apart and cleaned in sand bath before 2-16-16. Dyna- Purge E was not used at end of the extrusion runs to clean out extruder.
- 4) Extruder was cleaned in sand bath prior to 2-16-16 so we started out with a totally clean extruder screw. On 2-15-16, the screw was put back together and put into the extruder.
- 5) Per Sabic, keep T melt < 405°C.
- 6) Compressed air was used to cool zones 2 to 9. It was too hot to use water for cooling on these zones.
- 7) Did NOT use fiberglass insulation on the die end of the extruder (it got in the way of the extrudate).
- 8) Used Ktron twin screw feeder with 20 mm twin concave coarse screws for carbon black. Runs well 0.3 kg/h to 3.8kg/h. Worked great for 0.75 (0.34 kg/h) and 1.0 lb/hr (0.454 kg/h).
- 9) KT 880 NT was dried at 300F 4 hrs prior to extrusion and stored in moisture barrier bags. Extruded polymer was dried at 300F 4 hrs prior to injection molding and stored in moisture barrier bags.
- 10) Made about 10 lbs of VA2.5K and about 8 lbs of VA5K.
- 11) Used water bath at 80F. In bath for about 3 ft. Polymer did not work on the stainless steel conveyer- too hot- would not solidify.

Table 8.4: Extrusion Conditions for Solvay KetaSpire KT-880 NT with Akzo Nobel

Ketjenblack EC-600JD Carbon Black and/or Carbon Fiber from Solvay KT-880

CF30

Material Number	VA 7.5K	VA2.5F10K	VF10K
Extruder Screw Design	8-27-15	8-27-15	8-27-15
Extrusion Date	3-2-16	3-2-16	3-2-16
Extruder RPM	250	250	250
Motor Amperage, %	70	60	54
Melt Temperature, °C	400	395	395
Melt Pressure, psig	460	200	100
#3 Feeder Setting, lb/hr	18.5	20.0	20.0
Material in Feeder #3	KetaSpire KT-880 NT	KT880NT VA5K from 2-16-16 KT880 CF30	KetaSpire KT-880 NT KT880 CF30
KTron Feeder Setting, lb/hr	1.5	0	0
Material in Feeder #1	EC-600 JD	nothing	nothing
Vacuum Port	1 atm	1 atm	1 atm
Zone 5 side stuffer rpm	300	300	300
Material in Zone 5	EC-600 JD	nothing	nothing
Zone 7 side stuffer rpm	Closed port	Closed port	Closed port
Material in Zone 7	nothing	nothing	nothing
Feed Section Temperature	H ₂ O Cooled	H ₂ O Cooled	H ₂ O Cooled
Zone 1 Temperature, °C	300	300	300
Zone 2 Temperature, °C	375	375	375
Zone 3 Temperature, °C	380	380	380
Zone 4 Temperature, °C	380	380	380
Zone 5 Temperature, °C	380	380	380
Zone 6 Temperature, °C	380	380	380
Zone 7 Temperature, °C	380	380	380
Zone 8 Temperature, °C	380	380	380
Zone 9 Temperature, °C	380	380	380
Zone 10 Temperature, °C	370	370	370
Die Type and Gap	2 x 3 mm dia	2 x 3 mm dia	2 x 3 mm dia
Pelletizer Setting	Water bath at 80F	Water bath at 80F	Water bath at 80F
Output Rate, lb/hr	20 smooth surface	20	20 smooth surface

Notes:

- 1) The EC 600 JD carbon black was used as received.
- 2) Feeder 3 helix: 0.5" open helix with end stub and 0.75" nozzle side discharge: ran on automatic on loss in weight on laptop. Want mode = OP so can run on laptop. Toshiba laptop called feeder3zytel101jak5'02.par.
- 3) Dyna- Purge E was not used at end of the extrusion runs to clean out extruder.
- 4) Per Sabic, keep T melt < 405°C.
- 6) Compressed air was used to cool zones 2 to 9. It was too hot to use water for cooling on these zones.
- 7) Did NOT use fiberglass insulation on the die end of the extruder (it got in the way of the extrudate).
- 8) Used Ktron twin screw feeder with 20 mm twin concave coarse screws for carbon black. Runs well 0.3 kg/h to 3.8kg/h. Worked great for 1.5 lb/h (0.68 kg/h) for VA7.5K and all ran well.
- 9) KT 880 NT was dried at 300F 4 hrs prior to extrusion and stored in moisture barrier bags. Extruded polymer was dried at 300F 4 hrs prior to injection molding and stored in moisture barrier bags.
- 10) Made about 30 lbs of VA7.5K and about 7 lbs of VF10K.
- 11) Used water bath at 80F. In bath for about 3 ft. Polymer did not work on the stainless steel conveyor- too hot- would not solidify.
- 12) Extruded VA2.5F10K first, then VF10K, then VA7.5K on Mar 2 2016.
- 13) For VA2.5F10K and VF10K did blending of pellets from VA5K and Solvay KT880 CF30 and KT880NT in Vcone for 4 min at 24 rpm (max) in 3 lb batches (3 of so 9 lbs total). Then when extruding on 3-2-16, all went in zone 1.

Table 8.5: Extrusion Conditions for Solvay KetaSpire KT-880 NT with Akzo Nobel

Ketjenblack EC-600JD Carbon Black and Carbon Fiber from Solvay KT-880

CF30

Material Number	VA2.5KF20K	VA5F10K	VF20K
Extruder Screw Design	8-27-15	8-27-15	8-27-15
Extrusion Date	4-13-16	4-13-16	4-13-16
Extruder RPM	250	250	250
Motor Amperage, %	64	69	57
Melt Temperature, °C	396	395	395
Melt Pressure, psig	250	290	150
#3 Feeder Setting, lb/hr	20.0	20.0	20.0
Material in Feeder #3	KetaSpire KT-880 CF30 and VA7.5K from 3-2-16	KetaSpire KT-880 CF30 and VA7.5K from 3-2-16	KetaSpire KT-880 NT KT880 CF30
KTron Feeder Setting, lb/hr	0	0	0
Material in Feeder #1	nothing	nothing	nothing
Vacuum Port	1 atm	1 atm	1 atm
Zone 5 side stuffer rpm	300	300	300
Material in Zone 5	nothing	nothing	nothing
Zone 7 side stuffer rpm	Closed port	Closed port	Closed port
Material in Zone 7	nothing	nothing	nothing
Feed Section Temperature	H ₂ O Cooled	H ₂ O Cooled	H ₂ O Cooled
Zone 1 Temperature, °C	300	300	300
Zone 2 Temperature, °C	375	375	375
Zone 3 Temperature, °C	380	380	380
Zone 4 Temperature, °C	380	380	380
Zone 5 Temperature, °C	380	380	380
Zone 6 Temperature, °C	380	380	380
Zone 7 Temperature, °C	380	380	380
Zone 8 Temperature, °C	380	380	380
Zone 9 Temperature, °C	380	380	380
Zone 10 Temperature, °C	365	365	365
Die Type and Gap	2 x 3 mm dia	2 x 3 mm dia	2 x 3 mm dia
Pelletizer Setting	Water bath at 80F	Water bath at 80F	Water bath at 80F
Output Rate, lb/hr	20	20	20

Notes:

- 1) The EC 600 JD carbon black was used as received.
- 2) Feeder 3 helix: 0.5" open helix with end stub and 0.75" nozzle side discharge: ran on automatic on loss in weight on laptop. Want mode = OP so can run on laptop. Toshiba laptop called feeder3zytel101jak5'02.par.
- 3) Dyna- Purge E was not used at end of the extrusion runs to clean out extruder.
- 4) Per Sabic, keep T melt < 405°C.
- 6) Compressed air was used to cool zones 2 to 9. It was too hot to use water for cooling on these zones.
- 7) Did NOT use fiberglass insulation on the die end of the extruder (it got in the way of the extrudate).
- 8) Used Ktron twin screw feeder with 20 mm twin concave coarse screws for carbon black. Runs well 0.3 kg/h to 3.8kg/h. Worked great for 1.5 lb/h (0.68 kg/h) for VA7.5K and all ran well.
- 9) KT 880 NT was dried at 300F 4 hrs prior to extrusion and stored in moisture barrier bags. Extruded polymer was dried at 300F 4 hrs prior to injection molding and stored in moisture barrier bags.
- 10) Made about 8 lbs of VA2.5F20K, 8 lbs of VA5F10K, and about 6.5lbs of VF20K.
- 11) Used water bath at 80F. In bath for about 3 ft. Polymer did not work on the stainless steel conveyor- too hot- would not solidify.
- 12) Extruded VA2.5F20K first, then VA5F10K, then VF20K, then VD12.5K, then VA10K on April 13, 2016.
- 13) For VA2.5F20K, VA5F10K, and VF20K did blending of pellets from VA7.5K and Solvay KT880 CF30 and KT880NT in Vcone for 4 min at 24 rpm (max) in 3 lb batches (3 of so 9 lbs total). Then when extruding on 4-13-16, all went in zone 1.
- 14) For VD12.5K did blending of pellets from VD15K and Solvay KT880NT in Vcone for 4 min at 24 rpm (max) in 2.5lb batches (2 of so 5 lbs total). Then when extruding on 4-13-16, all went in zone 1. We used all the VD15K we had left over from prior extrusion work.

Table 8.6: Extrusion Conditions for Solvay KetaSpire KT-880 NT with Akzo Nobel

Ketjenblack EC-600JD Carbon Black or Carbon Fiber from Solvay KT-880 CF30

Material Number	VD12.5K	VA10K
Extruder Screw Design	8-27-15	8-27-15
Extrusion Date	4-13-16	4-13-16
Extruder RPM	150	250
Motor Amperage, %	49	78
Melt Temperature, °C	396	395
Melt Pressure, psig	170	700
#3 Feeder Setting, lb/hr	15.0	18.0
Material in Feeder #3	KetaSpire KT-880 NT and VD15K	KetaSpire KT-880 NT
KTron Feeder Setting, lb/hr	0	2.0
Material in Feeder #1	nothing	EC 600 JD carbon black
Vacuum Port	1 atm	1 atm
Zone 5 side stuffer rpm	300	300
Material in Zone 5	nothing	EC- 600 JD
Zone 7 side stuffer rpm	Closed port	Closed port
Material in Zone 7	nothing	nothing
Feed Section Temperature	H ₂ O Cooled	H ₂ O Cooled
Zone 1 Temperature, °C	300	300
Zone 2 Temperature, °C	375	375
Zone 3 Temperature, °C	380	380
Zone 4 Temperature, °C	380	380
Zone 5 Temperature, °C	380	380
Zone 6 Temperature, °C	380	380
Zone 7 Temperature, °C	380	380
Zone 8 Temperature, °C	380	380
Zone 9 Temperature, °C	380	380
Zone 10 Temperature, °C	365	365
Die Type and Gap	2 x 3 mm dia	2 x 3 mm dia
Pelletizer Setting	Water bath at 80F	Water bath at 80F
Output Rate, lb/hr	15	20

Notes:

- 1) The EC 600 JD carbon black was used as received.
- 2) Feeder 3 helix: 0.5" open helix with end stub and 0.75" nozzle side discharge: ran on automatic on loss in weight on laptop. Want mode = OP so can run on laptop. Toshiba laptop called feeder3zytel101jak5'02.par.
- 3) Dyna- Purge E was not used at end of the extrusion runs to clean out extruder.
- 4) Per Sabic, keep T melt < 405°C.
- 6) Compressed air was used to cool zones 2 to 9. It was too hot to use water for cooling on these zones.
- 7) Did NOT use fiberglass insulation on the die end of the extruder (it got in the way of the extrudate).
- 8) Used Ktron twin screw feeder with 20 mm twin concave coarse screws for carbon black. Runs well 0.3 kg/h to 3.8kg/h. Worked great for 2.0 lb/h (0.91 kg/h) for VA10K and all ran well but we are close to the maximum limit for carbon black content (getting very brittle and higher motor amps and pressures).
- 9) KT 880 NT was dried at 300F 4 hrs prior to extrusion and stored in moisture barrier bags. Extruded polymer was dried at 300F 4 hrs prior to injection molding and stored in moisture barrier bags.
- 10) Made about 4 lbs of VD12.5K and about 7.4 kg of VA10K.
- 11) Used water bath at 80F. In bath for about 3 ft. Polymer did not work on the stainless steel conveyor- too hot- would not solidify.
- 12) Extruded VA2.5F20K first, then VA5F10K, then VF20K, then VD12.5K, then VA10K on April 13, 2016.
- 13) For VA2.5F20K, VA5F10K, and VF20K did blending of pellets from VA7.5K and Solvay KT880 CF30 and KT880NT in Vcone for 4 min at 24 rpm (max) in 3 lb batches (3 of so 9 lbs total). Then when extruding on 4-13-16, all went in zone 1.
- 14) For VD12.5K did blending of pellets from VD15K and Solvay KT880NT in Vcone for 4 min at 24 rpm (max) in 2.5lb batches (2 of so 5 lbs total). Then when extruding on 4-13-16, all went in zone 1. We used all the VD15K we had left over from prior extrusion work.

Table 8.7: Extrusion Conditions for Solvay KetaSpire KT-880 NT with Akzo Nobel

Ketjenblack EC-600 JD Carbon Black and Carbon Fiber from Solvay KT-880

CF30

Material Number	VA5F30K	VA7.5F30K	VA2.5F30K
Extruder Screw Design	8-27-15	8-27-15	8-27-15
Extrusion Date	5-30-16	5-30-16	6-19-16
Extruder RPM	250	250	250
Motor Amperage, %	78	93	89
Melt Temperature, °C	396	400	400
Melt Pressure, psig	640	1040	540
#3 Feeder Setting, lb/hr	19.0	18.5	29.25
Material in Feeder #3	KetaSpire KT-880 CF30	KetaSpire KT-880 CF30	KetaSpire KT-880 CF30
KTron Feeder Setting, lb/hr	1.0	1.5	0.75
Material in Feeder #1	EC 600 JD carbon black	EC 600 JD carbon black	EC 600 JD carbon black
Vacuum Port	1 atm	1 atm	1 atm
Zone 5 side stuffer rpm	300	300	300
Material in Zone 5	EC- 600 JD	EC- 600 JD	EC- 600 JD
Zone 7 side stuffer rpm	Closed port	Closed port	Closed port
Material in Zone 7	nothing	nothing	nothing
Feed Section Temperature	H ₂ O Cooled	H ₂ O Cooled	H ₂ O Cooled
Zone 1 Temperature, °C	300	300	300
Zone 2 Temperature, °C	375	375	375
Zone 3 Temperature, °C	380	380	380
Zone 4 Temperature, °C	380	380	380
Zone 5 Temperature, °C	380	380	380
Zone 6 Temperature, °C	380	380	380
Zone 7 Temperature, °C	380	380	385
Zone 8 Temperature, °C	380	380	385
Zone 9 Temperature, °C	380	390	385
Zone 10 Temperature, °C	370	370	370
Die Type and Gap	2 x 3 mm dia	2 x 3 mm dia	2 x 3 mm dia
Pelletizer Setting	Water bath at 80F	Water bath at 80F	Water bath at 80F
Output Rate, lbs/hr	20	20	30

Notes:

- 1) The EC 600 JD carbon black was used as received.
- 2) Feeder 3 helix: 0.5" open helix with end stub and 0.75" nozzle side discharge: ran on automatic on loss in weight on laptop. Want mode = OP so can run on laptop. NEC laptop called feeder3zytel101jak5'02.par.
- 3) Extruder screws pulled and cleaned at end of day on May 30, 2015 since the extruder plugged when we tried to do VA10F27K- can not run anything with the VA10K! Took 5 hrs to clean it up, sand bath, etc.
- 4) Per Sabic, keep T melt < 405°C.
- 6) Compressed air was used to cool zones 2 to 9. It was too hot to use water for cooling on these zones.
- 7) Did NOT use fiberglass insulation on the die end of the extruder (it got in the way of the extrudate).
- 8) Used Ktron twin screw feeder with 20 mm twin concave coarse screws for carbon black. Runs well 0.3 kg/h to 3.8kg/h. Worked great for 2.0 lb/h (0.91 kg/h) for VA10K and all ran well but we are close to the maximum limit for carbon black content (getting very brittle and higher motor amps and pressures).
- 9) KT 880 NT was dried at 300F 4 hrs prior to extrusion and stored in moisture barrier bags. Extruded polymer was dried at 300F 4 hrs prior to injection molding and stored in moisture barrier bags.
- 10) Made about 7 lbs of VA5F30K and 7 lbs of VA7.5F30K.
- 11) Used water bath at 80F. In bath for about 3 ft. Polymer did not work on the stainless steel conveyor- too hot- would not solidify.
- 12) Next time make VA2.5F30K use concave fine screws in KTron feeder and then go at 20 lb/hr total flow. High on motor amps with 30 lb/h total flow.

Table 8.8: Injection Molding Conditions for Asbury TC307 GNP in KT-880 NT

Notation	Injection Molding Conditions	KetaSpire KT-880 NT (VK1) 6-30-15	VD2K Tensile only 7-15-15	VD4K Tensile only 7-15-15	VD6K Tensile only 7-15-15
T _{mold}	Mold Temperature (F)	400	400	400	400
EO	Zone 0 Temperature (F) (additional nozzle)	600 PV, SP at 700	600 PV, SP at 700	600 PV, SP at 700	600 PV, SP at 700
E1	Zone 1 Temperature (F) (nozzle)	720	745	745	745
E2	Zone 2 Temperature (F)	720	745	745	745
E3	Zone 3 Temperature (F)	720	745	745	745
E4	Zone 4 Temperature (F) (feed zone)	690	735	735	735
P1	Injection pressure (psi)	99% =22,384	99% =22,384	99% =22,384	99% =22,384
P2	Hold Pressure (psi)	70% =15,827	70% =15,827	70% =15,827	70% =15,827
P7	Back Pressure (psi)	2.2% = 500	2.2% = 500	2.2% = 500	2.2% = 500
S1	Shot size (mm)	22 tensile, 18.5 flex, 16.0 disk	21.5 for tensile	21.5 tensile	21.5 tensile
S2	Pullback before (mm)	0	0	0	0
S3	Pullback after (mm)	0.1	0.1	0.1	0.1
S6	Width of mold (mm)	202	202	202	202
S8	Screw Position to Switch from P1 to P2 (mm)	8 tensile, 10 flex, 12 disk	8 for tensile	8 for tensile	8 for tensile
T1	Injection Time (s)	12	12	12	12
T2	Cool Time (s)	35	35	35	35
T3	Interval Time (s)	1.0	15.0	15.0	15.0
T6	Retraction Time (s)	0	0	0	0
T7	Nozzle Retraction Delay Time (s)	0	0	0	0
T8	Injection Delay Time (s)	0	0	0	0
T9	Charge Delay Time (s)	0	0	0	0
V1	Injection Velocity (in ³ /s)	30% = 1.866	30% = 1.866	30% = 1.866	30% = 1.866
V6	Screw Rotation (rpm)	25% = 80	25% = 80	19% = 60	19% = 60
V9	Retraction Velocity (%)	90	90	20	20
V10	Advance Velocity (%)	90	90	50	50
V11	Retraction Velocity (%)	90	90	90	90
CF	Clamp Force (US tons)	80	80	80	80
P-5-1	Actual Cycle Time, sec	56 tensile, 52 flex, 51 disk	64 tensile	64 tensile	64 tensile

P-5-1	Inj Start, mm	22.2 tensile, 19.4 flex, 17.0 disk	23.6 tensile	23.6 tensile	23.6 tensile
P-5-1	Cushion, mm	0.1	3.6 tensile	3.6 tensile	3.6 tensile
P-5-1	P1 → P2 Fill time, sec	0.73 tensile, 0.53 flex, 0.34 disk	0.85 tensile	0.85 tensile	0.85 tensile
P-5-1	P1 Actual Pressure P1 → P2, kg/cm ²	44 tensile, 46 flex, 39 disk	70 tensile	84 tensile	89 tensile
P-5-1	Charge time, sec	3.9 tensile, 2.7 flex, 2.9 disk	2.5 tensile	2.5 tensile	2.5 tensile
P-5-1	Injection Peak Pressure P1, kg/cm ²	44 tensile, 46 flex, 39 disk	70 tensile	84 tensile	89 tensile

Table 8.9: Injection Molding Conditions for Asbury TC307 GNP in KT-880 NT (continued from Table 8.8)

Notation	Injection Molding Conditions	VD8K Tensile only 7-15-15	VD10K Tensile only 7-15-15
T _{mold}	Mold Temperature (F)	400	400
EO	Zone 0 Temperature (F) (additional nozzle)	600, SP at 700	600 PV, SP at 700
E1	Zone 1 Temperature (F) (nozzle)	745	745
E2	Zone 2 Temperature (F)	745	745
E3	Zone 3 Temperature (F)	745	745
E4	Zone 4 Temperature (F) (feed zone)	735	735
P1	Injection pressure (psi)	99% =22,384	99% =22,384
P2	Hold Pressure (psi)	70% =15,827	70% =15,827
P7	Back Pressure (psi)	2.2% = 500	2.2% = 500
S1	Shot size (mm)	21.5 tensile	21.5 tensile
S2	Pullback before (mm)	0	0
S3	Pullback after (mm)	0.1	0.1
S6	Width of mold (mm)	202	202
S8	Screw Position to Switch from P1 to P2 (mm)	8 for tensile	8 for tensile
T1	Injection Time (s)	12	12
T2	Cool Time (s)	35	35
T3	Interval Time (s)	15.0	15.0
T6	Retraction Time (s)	0	0

T7	Nozzle Retraction Delay Time (s)	0	0
T8	Injection Delay Time (s)	0	0
T9	Charge Delay Time (s)	0	0
V1	Injection Velocity (in ³ /s)	30% = 1.866	30% = 1.866
V6	Screw Rotation (rpm)	19% = 60	19% = 60
V9	Retraction Velocity (%)	20	20
V10	Advance Velocity (%)	50	50
V11	Retraction Velocity (%)	90	90
CF	Clamp Force (US tons)	80	80
P-5-1	Actual Cycle Time, sec	64 tensile	64 tensile
P-5-1	Inj Start, mm	23.6 tensile	23.6 tensile
P-5-1	Cushion, mm	3.6 tensile	3.6 tensile
P-5-1	P1→ P2 Fill time, sec	0.85 tensile	0.85 tensile
P-5-1	P1 Actual Pressure P1→ P2, kg/cm ²	96 tensile	98 tensile
P-5-1	Charge time, sec	2.5 tensile	2.5 tensile
P-5-1	Injection Peak Pressure P1, kg/cm ²	96 tensile	98 tensile

Notes:

1. On June 30 and July 15, 2015, the nozzle was not retracted so it stayed at ‘nozzle touch’.
2. After each formulation, the feed hopper was run until it was empty and no material was in screw. Then we discarded 5 transition shots, and then started collecting the next formulation.
3. For VD10K next time try to increase temperatures and/or P2 (hold pressure).

Table 8.10: Injection Molding Conditions for Asbury TC307 GNP in KT-880 NT

		VD15K
Notation	Injection Molding Conditions	9-10-15
T _{mold}	Mold Temperature (F)	400
EO	Zone 0 Temperature (F) (additional nozzle)	630 PV, SP at 700
E1	Zone 1 Temperature (F) (nozzle)	780
E2	Zone 2 Temperature (F)	780
E3	Zone 3 Temperature (F)	780
E4	Zone 4 Temperature (F) (feed zone)	790
P1	Injection pressure (psi)	99% = 22,384
P2	Hold Pressure (psi)	90% = 20,349
P7	Back Pressure (psi)	2.2% = 500

S1	Shot size (mm)	21.2 tensile, 18.5 flex, 16.0 disk
S2	Pullback before (mm)	0
S3	Pullback after (mm)	0.1
S6	Width of mold (mm)	202
S8	Screw Position to Switch from P1 to P2 (mm)	8 for tensile, 10 for flex, 12 disk
T1	Injection Time (s)	12
T2	Cool Time (s)	20
T3	Interval Time (s)	15.0
T6	Retraction Time (s)	0
T7	Nozzle Retraction Delay Time (s)	0
T8	Injection Delay Time (s)	0
T9	Charge Delay Time (s)	0
V1	Injection Velocity (in ³ /s)	30% = 1.866
V6	Screw Rotation (rpm)	19% = 60
V9	Retraction Velocity (%)	20
V10	Advance Velocity (%)	50
V11	Retraction Velocity (%)	90
CF	Clamp Force (US tons)	80
P-5-1	Actual Cycle Time, sec	48.6 tensile 48.6 flex 48.6 disk
P-5-1	Inj Start, mm	21.8 tensile 18.6 flex 16.1 disk
P-5-1	Cushion, mm	0.9 tensile 0.1 flex 0.1 disk
P-5-1	P1→ P2 Fill time, sec	0.77 tensile 0.55 flex 0.34 disk
P-5-1	P1 Actual Pressure P1→ P2, kg/cm ²	102 tensile 64 flex 45 disk
P-5-1	Charge time, sec	4 tensile 4 flex 4 disk
P-5-1	Injection Peak Pressure P1, kg/cm ²	94 tensile 64 flex 45 disk

Notes:

1. The nozzle was not retracted so it stayed at 'nozzle touch'.

2. After each formulation, the feed hopper was run until it was empty and no material was in screw. Then we discarded 5 transition shots, and then started collecting the next formulation.
3. For tensiles for VD15K, we had problems with tensile bars- stuck in sprue in mold near injection end- on every other sample. Flex and disks ran OK.
4. $1\text{ kgf/cm}^2=14.22\text{ psi}$ for unit conversion

Table 8.11: Injection Molding Conditions for Akzo Nobel Ketjenblack EC-600 JD Carbon

Black and/or Carbon Fiber from KT-880 CF30 in KT-880 NT

Notation	Injection Molding Conditions	VA2.5K 2-24-16	VA5K 2-24-16	VA2.5F10K Extr 3-2-16 3-23-16	VF10K Extr 3-2-16 3-23-16
T _{mold}	Mold Temperature (F)	400	400	400	400
E0	Zone 0 Temperature (F) (additional nozzle)	650 PV, SP at 750	650 PV, SP at 750	650 PV, SP at 750	650 PV, SP at 750
E1	Zone 1 Temperature (F) (nozzle)	750	750 tensile 780 flex disk	780	780
E2	Zone 2 Temperature (F)	750	790 tens 800 disk flex	790	790
E3	Zone 3 Temperature (F)	750	790 tens 800 disk flex	790	790
E4	Zone 4 Temperature (F) (feed zone)	750	790 tens 800 disk flex	790	790
P1	Injection pressure (psi)	99% =22,384 for tensile and flex, 90% for disks	99% =22,384 for tensile and flex, 90% for disks	99% =22,384 for tensile and flex, and disks	99% =22,384 for tensile and flex, and disks
P2	Hold Pressure (psi)	80% =need to calc	80% = need to calc	80% =need calc	80% =need calc
P7	Back Pressure (psi)	2.2% = 500	2.2% = 500	2.2% = 500	2.2% = 500
S1	Shot size (mm)	21.2 tensile, 18.5 flex, 16.0 disk	21.2 tensile, 18.5 flex, 16.0 disk	21.2 tensile, 18.5 flex, 16.0 disk	21.2 tensile, 18.5 flex, 16.0 disk
S2	Pullback before (mm)	0	0	0	0
S3	Pullback after (mm)	0.5	0.5	0.5	0.5
S6	Width of mold (mm)	220= SP PV=207	220= SP PV=207	220= SP PV=207	220= SP PV=207
S8	Screw Position to Switch from P1 to P2 (mm)	8 tensile, 10 flex, 12 disk	8 tensile, 10 flex, 12 disk	8 tensile, 10 flex, 12 disk	8 tensile, 10 flex, 12 disk

T1	Injection Time (s)	12	12	12	12
T2	Cool Time (s)	20	20	20	20
T3	Interval Time (s)	40	40	30	30
T6	Retraction Time (s)	0	0	0	0
T7	Nozzle Retraction Delay Time (s)	0	0	0	0
T8	Injection Delay Time (s)	0	0	0	0
T9	Charge Delay Time (s)	0	0	0	0
V1	Injection Velocity (in ³ /s)	30% = 1.866	30% = 1.866	30% = 1.866	30% = 1.866
V6	Screw Rotation (rpm)	25% = 80	25% = 80	25% = 80	25% = 80
V9	Retraction Velocity (%)	90	90	90	90
V10	Advance Velocity (%)	90	90	90	90
V11	Retraction Velocity (%)	90	90	90	90
CF	Clamp Force (US tons)	80	80	80	80
P-5-1	Actual Cycle Time, sec	74 tensile 74 flex 74 disk	74 tensile 74 flex 74 disk	64 tensile 64 flex 64 disk	64 tensile 64 flex 64 disk
P-5-1	Inj Start, mm	21.9 tensile 19.7 flex 17.2 disk	21.5 tensile 19.0 flex 16.6 disk	22 tensile 19.3 flex 16.9 disk	22 tensile 19.3 flex 16.9 disk
P-5-1	Cushion, mm	2.4 tensile 0.7 flex 0.1 disk	0.2 tensile 0.1 flex 0.1 disk	0.3 tensile 0.1 flex 0.1 disk	0.3 tensile 0.1 flex 0.1 disk
P-5-1	P1→ P2 Fill time, sec	0.8 tensile 0.6 flex 0.34 disk	0.8 tensile 0.5 flex 0.3 disk	0.8 tensile 0.5 flex 0.4 disk	0.8 tensile 0.6 flex 0.4 disk
P-5-1	P1 Actual Pressure P1→ P2, kg/cm ²	74 tensile 48 flex 30 disk	74 tensile 56 flex 39 disk	64 tensile 47 flex 29 disk	67 tensile 49 flex 29 disk
P-5-1	Charge time, sec	3 tensile 3 flex 3 disk	3.3 tensile 6.4 flex 6.8 disk	3.3 tensile 3.1 flex 2.9 disk	3.3 tensile 3.1 flex 2.9 disk
P-5-1	Injection Peak Pressure P1, kg/cm ²	74 tensile 48 flex 30 disk	74 tensile 56 flex 39 disk	64 tensile 47 flex 29 disk	67 tensile 49 flex 29 disk

Notes:

1. On Mar 23, 2016, the nozzle was not retracted so it stayed at 'nozzle touch'.
2. After each formulation, the feed hopper was run until it was empty and no material was in screw. Then we discarded 5 transition shots, and then started collecting the next formulation.
3. 1kgf/cm²=14.22 psi for unit conversion

**Table 8.12: Injection Molding Conditions for Akzo Nobel Ketjenblack EC-600 JD Carbon
Black and/or Carbon Fiber from KT-880 CF30 in KT-880 NT**

Notation	Injection Molding Conditions	VF20K Ex 4-13-16 4-20-16	VA2.5F20K Ex 4-14-16 4-20-16	VA5F10K Ex 4-13-16 4-20-16	VD12.5K Ex4-13-16 4-20-16
T _{mold}	Mold Temperature (F)	400	400	400	400
E0	Zone 0 Temperature (F) (additional nozzle)	700 PV, SP at 750	700 PV, SP at 750	650 PV, SP at 750	650 PV, SP at 750
E1	Zone 1 Temperature (F) (nozzle)	740	780	780	780
E2	Zone 2 Temperature (F)	740	790	800	780
E3	Zone 3 Temperature (F)	740	790	800	780
E4	Zone 4 Temperature (F) (feed zone)	740	790	800	790
P1	Injection pressure (psi)	99% =22,384 for tensile and flex, 90% for disks	99% =22,384 for tensile and flex, 90% for disks	99% =22,384 for tensile and flex, and disks	99% =22,384 for tensile and flex, and disks
P2	Hold Pressure (psi)	75% =need to calc	75% = need to calc	80% =need calc	80% =need calc
P7	Back Pressure (psi)	2.2% = 500	2.2% = 500	2.2% = 500	2.2% = 500
S1	Shot size (mm)	21.2 tensile, 18.5 flex, 16.0 disk	21.2 tensile, 18.5 flex, 16.0 disk	21.2 tensile, 18.5 flex, 16.0 disk	21.2 tensile, 18.5 flex, 16.0 disk
S2	Pullback before (mm)	0	0	0	0
S3	Pullback after (mm)	0.5	0.5	0.5	0.5
S6	Width of mold (mm)	220= SP PV=207	220= SP PV=207	220= SP PV=207	220= SP PV=207
S8	Screw Position to Switch from P1 to P2 (mm)	8 for tensile, 10 for flex, 12 for disk	8 tensile, 10 for flex, 12 for disk	8 for tensile, 10 for flex, 12 for disk	8 tensile, 10 flex, 12 disk
T1	Injection Time (s)	12	12	12	12
T2	Cool Time (s)	20	20	20	20
T3	Interval Time (s)	40	40	40	40
T6	Retraction Time (s)	0	0	0	0
T7	Nozzle Retraction Delay Time (s)	0	0	0	0
T8	Injection Delay Time (s)	0	0	0	0
T9	Charge Delay Time (s)	0	0	0	0
V1	Injection Velocity (in ³ /s)	30% = 1.866	30% = 1.866	30% = 1.866	30% = 1.866

V6	Screw Rotation (rpm)	25% = 80	25% = 80	25% = 80	25% = 80
V9	Retraction Velocity (%)	90	90	90	90
V10	Advance Velocity (%)	90	90	90	90
V11	Retraction Velocity (%)	90	90	90	90
CF	Clamp Force (US tons)	80	80	80	80
P-5-1	Actual Cycle Time, sec	74 tensile 74 flex 74 disk	74 tensile 74 flex 74 disk	74 tensile 74 flex 74 disk	74 tensile 74 flex 74 disk
P-5-1	Inj Start, mm	22.3 tensile 19.4 flex 16.9 disk	22.3 tensile 19.4 flex 16.9 disk	22.3 tensile 19.4 flex 16.9 disk	22.3 tensile 19.4 flex 16.9 disk
P-5-1	Cushion, mm	1.6 tensile 1.1 flex 0.1 disk	1.1 tensile 1. flex 0.1 disk	1.2 tensile 0.1 flex 0.1 disk	0.7 tensile 0.7 flex 0.1 disk
P-5-1	P1→ P2 Fill time, sec	0.8 tensile 0.6 flex 0.4 disk	0.8 tensile 0.8 flex 0.4 disk	0.8 tensile 0.6 flex 0.4 disk	0.8 tensile 0.6 flex 0.4 disk
P-5-1	P1 Actual Pressure P1→ P2, kg/cm ²	78 tensile 57 flex 41 disk	69 tensile 56 flex 35 disk	78 tensile 55 flex 33 disk	78 tensile 60 flex 46 disk
P-5-1	Charge time, sec	3 tensile 3 flex 3 disk	3.6 tensile 2.7 flex 2.9 disk	3.3 tensile 3.1 flex 2.9 disk	3.3 tensile 3.1 flex 2.9 disk
P-5-1	Injection Peak Pressure P1, kg/cm ²	78 tensile 57 flex 41 disk	69 tensile 56 flex 35 disk	78 tensile 55 flex 33 disk	78 tensile 60 flex 46 disk

Notes:

1. On 4-20-16, the nozzle was not retracted so it stayed at 'nozzle touch'.
2. After each formulation, the feed hopper was run until it was empty and no material was in screw. Then we discarded 5 transition shots, and then started collecting the next formulation.
3. 1kg/cm²=14.22 psi for unit conversion
4. VA5F10K is getting close to the limit of what we can inj mold- starting to get more viscous

Table 8.13: Injection Molding Conditions for Akzo Nobel Ketjenblack EC-600 JD Carbon

Black in KT-880 NT

Notation	Injection Molding Conditions	VA7.5K Ex 3-2-16 5-10-16	VA10K Ex 4-13-16 5-10-16
T _{mold}	Mold Temperature (F)	400	400
E0	Zone 0 Temperature (F) (additional nozzle)	700 PV, SP at 750	700 PV, SP at 750
E1	Zone 1 Temperature (F) (nozzle)	780	790
E2	Zone 2 Temperature (F)	800	830
E3	Zone 3 Temperature (F)	800	830
E4	Zone 4 Temperature (F) (feed zone)	800	830
P1	Injection pressure (psi)	99% =22,384 for tensile and flex, 99% for disks	99% =22,384 for tensile and flex, 99% for disks
P2	Hold Pressure (psi)	90% =need to calc	90% = need to calc
P7	Back Pressure (psi)	2.2% = 500	2.2% = 500
S1	Shot size (mm)	21.2 for tensile, 18.5 for flex, 16.0 for disk	21.2 for tensile, 18.5 for flex, 16.0 for disk
S2	Pullback before (mm)	0	0
S3	Pullback after (mm)	0.5	0.5
S6	Width of mold (mm)	220= SP PV=207	220= SP PV=207
S8	Screw Position to Switch from P1 to P2 (mm)	8 for tensile, 10 for flex, 12 for disk	8 for tensile, 10 for flex, 12 for disk
T1	Injection Time (s)	12	12
T2	Cool Time (s)	20	20
T3	Interval Time (s)	40	40
T6	Retraction Time (s)	0	0
T7	Nozzle Retraction Delay Time (s)	0	0
T8	Injection Delay Time (s)	0	0
T9	Charge Delay Time (s)	0	0
V1	Injection Velocity (in ³ /s)	30% = 1.866	30% = 1.866
V6	Screw Rotation (rpm)	25% = 80	25% = 80
V9	Retraction Velocity (%)	90	90

V10	Advance Velocity (%)	90	90
V11	Retraction Velocity (%)	90	90
CF	Clamp Force (US tons)	80	80
P-5-1	Actual Cycle Time, sec	74 tensile 74 flex 74 disk	74 tensile 74 flex 74 disk
P-5-1	Inj Start, mm	22.3 tensile 19.4 flex 16.9 disk	22.3 tensile 19.4 flex 16.9 disk
P-5-1	Cushion, mm	0.1 mm tensile 0.1 flex 0.1 disk	0.1 mm tensile 0.1 flex 0.1 disk
P-5-1	P1→ P2 Fill time, sec	0.8 tensile 0.6 flex 0.4 disk	0.8 tensile 0.5 flex 0.4 disk
P-5-1	P1 Actual Pressure P1→ P2, kg/cm ²	88 tensile 62 flex 41 disk	91 tensile 54 flex 43 disk
P-5-1	Charge time, sec	3 tensile 3 flex 3 disk	3 tensile 3 flex 3 disk
P-5-1	Injection Peak Pressure P1, kg/cm ²	88 tensile 62 flex 41 disk	91 tensile 54 flex 43 disk

Notes:

1. On 5-10-16, the nozzle was not retracted so it stayed at 'nozzle touch'.
2. After each formulation, the feed hopper was run until it was empty and no material was in screw. Then we discarded 5 transition shots, and then started collecting the next formulation.
3. 1kgf/cm²=14.22 psi for unit conversion
4. VA7.5K and VA10K inj molded OK so can likely add some carbon fiber (maybe 10 wt%) likely and be able to inj mold it

Table 8.14: Injection Molding Conditions for Akzo Nobel Ketjenblack EC-600 JD Carbon

Black and Carbon Fiber from KT-880 CF30 in KT-880 NT

Notation	Injection Molding Conditions	VA5F30K Ex 5-30-16 6-7-16	VA7.5F30K Ex 5-30-16 6-7-16	VA2.5F30K Ex 6-19-16 6-23-16
T _{mold}	Mold Temperature (F)	400	400	400
E0	Zone 0 Temperature (F) (additional nozzle)	700 PV, SP at 750	700 PV, SP at 750	700 PV, SP at 750
E1	Zone 1 Temperature (F) (nozzle)	780	830F tensile, 840 F flex, 860 disc	790
E2	Zone 2 Temperature (F)	800	845 ten 845 flex 850 disc	800
E3	Zone 3 Temperature (F)	800	845 ten 845 flex 850 disc	800
E4	Zone 4 Temperature (F) (feed zone)	800	845 ten 845 flex 850 disc	800
P1	Injection pressure (psi)	99% =22,384 for tensile and flex, 99% for disks	99% =22,384 for tensile and flex, 99% for disks	99%
P2	Hold Pressure (psi)	90% =need to calc	99% = need to calc	99%
P7	Back Pressure (psi)	2.2% = 500	2.2% = 500	2.2% = 500
S1	Shot size (mm)	21.2 tensile, 18.5 for flex, 16.0 for disk	21.2 for tensile, 18.5 for flex, 16.0 for disk	21.2 for tensile, 18.5 for flex, 16.0 for disk
S2	Pullback before (mm)	0	0	0
S3	Pullback after (mm)	0.5	0.5	0.5
S6	Width of mold (mm)	220= SP PV=207	220= SP PV=207	220= SP PV=207
S8	Screw Position to Switch from P1 to P2 (mm)	8 for tensile, 10 for flex, 12 for disk	8 for tensile, 10 for flex, 12 for disk	8 for tensile, 10 for flex, 12 for disk
T1	Injection Time (s)	12	12	12
T2	Cool Time (s)	20	20	20
T3	Interval Time (s)	40	40	20
T6	Retraction Time (s)	0	0	0

T7	Nozzle Retraction Delay Time (s)	0	0	0
T8	Injection Delay Time (s)	0	0	0
T9	Charge Delay Time (s)	0	0	0
V1	Injection Velocity (in ³ /s)	30% = 1.866	30% = 1.866	30% = 1.866
V6	Screw Rotation (rpm)	25% = 80	25% = 80	25% = 80
V9	Retraction Velocity (%)	90	90	90
V10	Advance Velocity (%)	90	90	90
V11	Retraction Velocity (%)	90	90	90
CF	Clamp Force (US tons)	80	80	80
P-5-1	Actual Cycle Time, sec	74 tensile 74 flex 74 disk	74 tensile 74 flex 74 disk	53 tensile 53 flex 53 disk
P-5-1	Inj Start, mm	22.3 tensile 19.4 flex 16.9 disk	22.3 tensile 19.4 flex 16.9 disk	22.3 tensile 19.4 flex 16.9 disk
P-5-1	Cushion, mm	0.1 mm tensile 0.1 flex 0.1 disk	0.1 mm tensile 0.1 flex 0.1 disk	0.9 mm tensile 0.1 flex 0.1 disk
P-5-1	P1→ P2 Fill time, sec	0.8 tensile 0.6 flex 0.4 disk	0.8 tensile 0.5 flex 0.4 disk	0.8 tensile 0.6 flex 0.4 disk
P-5-1	P1 Actual Pressure P1→ P2, kg/cm ²	127 tensile 85 flex 50 disk	134 tensile 79 flex 40 disk	112 tensile 73 flex 48 disk
P-5-1	Charge time, sec	3 tensile 3 flex 3 disk	3 tensile 3 flex 3 disk	3 tensile 3 flex 3 disk
P-5-1	Injection Peak Pressure P1, kg/cm ²	127 tensile 85 flex 50 disk	134 tensile 79 flex 40 disk	112 tensile 73 flex 48 disk

Notes:

1. On 6-7-16 and June 23 2016, the nozzle was not retracted so it stayed at 'nozzle touch'.
2. After each formulation, the feed hopper was run until it was empty and no material was in screw. Then we discarded 5 transition shots, and then started collecting the next formulation.
3. 1kg_f/cm²=14.22 psi for unit conversion
4. VA5F30K molded OK. VA7.5F30K was hard to inj mold but it ran- higher temps. VA2.5F30K molded OK.

8.2 PEEK Electrical Resistivity Data

Table 8.15: ASTM D257 Through-Plane Electrical Resistivity Results for KetaSpire KT 880

NT injection molded at MTU 6-30-15

Replicates 1 and 2 combined			
Test Date	Sample Number	Applied Voltage (V)	Through-Plane Volume Electrical Resistivity (Ω -cm)
1/15/2015	KT880-ER-26	100	2.8811E+17
1/15/2015	KT880-ER-27	100	2.4145E+17
1/15/2015	KT880-ER-10	100	2.4512E+17
1/15/2015	KT880-ER-24	100	2.9261E+17
1/15/2015	KT880-ER-8	100	2.8528E+17
1/15/2015	KT880-ER-21	100	3.1196E+17
1/15/2015	KT880-ER-42	100	2.9018E+17
1/15/2015	KT880-ER-22	100	2.9108E+17
		Average	2.8072E+17
		Standard Deviation	2.4492E+16
		Number of Samples	8
Replicate 1			
Test Date	Sample Number	Applied Voltage (V)	Through-Plane Volume Electrical Resistivity (Ω -cm)
1/15/2015	KT880-ER-26	100	2.8811E+17
1/15/2015	KT880-ER-27	100	2.4145E+17
1/15/2015	KT880-ER-10	100	2.4512E+17
1/15/2015	KT880-ER-24	100	2.9261E+17
		Average	2.6682E+17
		Standard Deviation	2.7282E+16
		Number of Samples	4
Replicate 2			
Test Date	Sample Number	Applied Voltage (V)	Through-Plane Volume Electrical Resistivity (Ω -cm)
1/15/2015	KT880-ER-21	100	3.1196E+17
1/15/2015	KT880-ER-42	100	2.9018E+17
1/15/2015	KT880-ER-8	100	2.8528E+17
1/15/2015	KT880-ER-22	100	2.9108E+17
		Average	2.9463E+17
		Standard Deviation	1.1834E+16
		Number of Samples	4

Table 8.16: ASTM D257 Through-Plane Electrical Resistivity Results for VD2K: 2 wt%

**TC307 GNP in KetaSpire KT880 NT: Extruded 3-17-15 at MTU, 2-5-15 extruder
screw: Injection Molded at Solvay**

Test Date	Sample Number	Applied Voltage (V)	Through-Plane Surface Electrical Resistivity (Ω /square)	Through-Plane Volume Electrical Resistivity (Ω -cm)
6/10/2015	VD2K-TC-8	100	4.2272E+17	2.3617E+17
6/10/2015	VD2K-TC-3	100	3.1078E+17	2.2167E+17
6/10/2015	VD2K-TC-9	100	5.6441E+17	2.3074E+17
6/10/2015	VD2K-TC-5	100	4.2440E+17	2.4358E+17
6/10/2015	VD2K-TC-6	100	4.8474E+17	2.3934E+17
6/10/2015	VD2K-TC-10	100	4.4964E+17	2.3325E+17
		Average	4.4278E+17	2.3413E+17
		Standard Deviation	8.3386E+16	7.5911E+15
		Number of Samples	6	6

Table 8.17: ASTM D257 Through-Plane Electrical Resistivity Results for VD4K: 4 wt%

**TC307 GNP in KetaSpire KT880 NT: Extruded 3-17-15 at MTU, 2-5-15 extruder
screw: Injection Molded at Solvay**

Test Date	Sample Number	Applied Voltage (V)	Through-Plane Surface Electrical Resistivity (Ω /square)	Through-Plane Volume Electrical Resistivity (Ω -cm)
6/10/2015	VD4K-TC-1	100	5.1389E+17	7.4611E+16
6/10/2015	VD4K-TC-2	100	3.7642E+17	7.7257E+16
6/10/2015	VD4K-TC-3	100	4.8222E+17	7.5588E+16
6/10/2015	VD4K-TC-4	100	4.2495E+17	7.8087E+16
6/10/2015	VD4K-TC-5	100	4.4073E+17	8.0982E+16
6/10/2015	VD4K-TC-6	100	5.7769E+17	7.7558E+16
		Average	4.6932E+17	7.7347E+16
		Standard Deviation	7.1153E+16	2.2088E+15
		Number of Samples	6	6

Table 8.18: ASTM D257 Through-Plane Electrical Resistivity Results for VD6K: 6 wt%

**TC307 GNP in KetaSpire KT880 NT: Extruded 3-17-15 at MTU, 2-5-15 extruder
screw: Injection Molded at Solvay**

Test Date	Sample Number	Applied Voltage (V)	Through-Plane Surface Electrical Resistivity (Ω /square)	Through-Plane Volume Electrical Resistivity (Ω -cm)
6/10/2015	VD6K-TC-1	100	7.1849E+16	5.3028E+16
6/10/2015	VD6K-TC-2	100	4.6627E+17	5.0400E+16
6/10/2015	VD6K-TC-3	100	1.0692E+17	4.9988E+16
6/10/2015	VD6K-TC-4	100	5.1119E+17	4.8412E+16
6/10/2015	VD6K-TC-5	100	2.3004E+17	4.6813E+16
6/10/2015	VD6K-TC-6	100	8.7719E+16	3.9782E+16
		Average	2.4566E+17	4.8071E+16
		Standard Deviation	1.9689E+17	4.5624E+15
		Number of Samples	6	6

Table 8.19: ASTM D257 Through-Plane Electrical Resistivity Results for VD8K: 8 wt%

**TC307 GNP in KetaSpire KT880 NT: Extruded 3-19-15 at MTU, 2-5-15 extruder
screw: Injection Molded at Solvay**

Test Date	Sample Number	Applied Voltage (V)	Through-Plane Surface Electrical Resistivity (Ω /square)	Through-Plane Volume Electrical Resistivity (Ω -cm)
6/10/2015	VD8K-TC-1	100	2.1507E+17	2.9801E+16
6/10/2015	VD8K-TC-2	100	2.2766E+17	2.8370E+16
6/10/2015	VD8K-TC-4	100	2.5920E+17	2.5964E+16
6/10/2015	VD8K-TC-5	100	3.0221E+17	2.9601E+16
6/10/2015	VD8K-TC-6	100	3.2955E+17	3.0450E+16
6/10/2015	VD8K-TC-7	100	2.6826E+17	2.7357E+16
		Average	2.6699E+17	2.8591E+16
		Standard Deviation	4.3495E+16	1.6978E+15
		Number of Samples	6	6

Table 8.20: ASTM D257 Through-Plane Electrical Resistivity Results for VD10K: 10 wt%

TC307 GNP in KetaSpire KT880 NT: Extruded 3-19-15 at MTU, 2-5-15 extruder screw: Injection Molded at Solvay

Test Date	Sample Number	Applied Voltage (V)	Through-Plane Surface Electrical Resistivity (Ω /square)	Through-Plane Volume Electrical Resistivity (Ω -cm)
6/10/2015	VD10K-TC-1	100	1.1210E+17	1.3324E+16
6/10/2015	VD10K-TC-2	100	8.6467E+16	1.5243E+16
6/10/2015	VD10K-TC-3	100	1.1441E+17	1.3609E+16
6/10/2015	VD10K-TC-4	100	9.9446E+16	1.5672E+16
6/10/2015	VD10K-TC-5	100	1.1635E+17	1.4351E+16
6/10/2015	VD10K-TC-6	100	1.1988E+17	1.4946E+16
		Average	1.0811E+17	1.4524E+16
		Standard Deviation	1.2693E+16	9.2953E+14
		Number of Samples	6	6

Table 8.21: ASTM D257 Through-Plane Electrical Resistivity Results for VD12.5K: 12.5 wt%

TC307 GNP in KetaSpire KT880 NT: Extruded 4-13-16 at MTU, 78-27-15 extruder screw: Injection Molded at MTU 4-20-16

Test Date	Sample Number	Applied Voltage (V)	Through-Plane Surface Electrical Resistivity (Ω /square)	Through-Plane Volume Electrical Resistivity (Ω -cm)
4/27/2016	VD12.5K-TC-9	100	1.2086E+15	6.9041E+13
4/27/2016	VD12.5K-TC-14	100	2.4557E+15	5.2435E+13
4/27/2016	VD12.5K-TC-12	100	2.7070E+15	1.2373E+14
4/27/2016	VD12.5K-TC-16	100	1.4185E+15	6.0024E+13
4/27/2016	VD12.5K-TC-11	100	1.6577E+15	1.8693E+14
4/27/2016	VD12.5K-TC-18	100	4.2090E+16	9.4352E+13
		Average	8.5896E+15	9.7752E+13
		Standard Deviation	1.6422E+16	5.0873E+13
		Number of Samples	6	6

Table 8.22: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for VD15K: 15

wt% TC307 in KT880 NT: extruded 8-25-15 at MTU, 7-8-15 screw, Injection

Molded at MTU 9-10-15

Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
9/18/2015	VD15K-T-41	6.02	0.34	1.26	2.57E-08	10	2.769E+7
9/18/2015	VD15K-T-49	5.95	0.34	1.26	2.30E-08	10	3.130E+7
9/18/2015	VD15K-T-52	5.84	0.34	1.26	2.27E-08	10	3.232E+7
9/18/2015	VD15K-T-53	6	0.34	1.26	2.34E-08	10	3.051E+7
9/18/2015	VD15K-T-57	5.95	0.34	1.26	2.73E-08	10	2.637E+7
					Average		2.964E+7
					Std Dev		2.508E+6
					Number		5

Table 8.23: ASTM D257 Through-Plane Electrical Resistivity Results for 10 wt% Carbon

Fiber from KT-880 CF30 in KetaSpire KT 880 NT Extruded 3-2-2016, 8-27-15

screw, Injection Molded at MTU 3-23-16

Test Date	Sample Number	Applied Voltage (V)	Through-Plane Surface Electrical Resistivity (Ω /square)	Through-Plane Volume Electrical Resistivity (Ω -cm)
3/28/2016	VF10K-ER-13	100	9.3917E+15	1.2618E+16
3/29/2016	VF10K-ER-12	100	2.6040E+16	1.4042E+16
3/30/2016	VF10K-ER-16	100	2.6230E+16	1.2457E+16
3/31/2016	VF10K-ER-20	100	2.5870E+16	1.1400E+16
4/1/2016	VF10K-ER-11	100	2.1218E+16	1.2035E+16
4/2/2016	VF10K-ER-3	100	2.3076E+15	1.2424E+16
		Average	1.8510E+16	1.2496E+16
		Standard Deviation	1.0232E+16	8.7469E+14
		Number of Samples	6	6

Table 8.24: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for VF20K: 20

wt% Carbon Fiber from KT 880 CF30 with KT880 NT: Extruded 4-13-16 at MTU,

8-27-15 screw, Injection Molded at MTU 4-20-16

Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
4/28/2016	VF20K-T-10	6.16	0.34	1.25	2.31E-08	10	2.9867E+07
4/28/2016	VF20K-T-11	6.02	0.34	1.25	2.61E-08	10	2.7049E+07
4/28/2016	VF20K-T-12	5.99	0.34	1.25	3.25E-08	10	2.1831E+07
4/28/2016	VF20K-T-16	6.04	0.34	1.25	6.74E-08	10	1.0440E+07
4/28/2016	VF20K-T-26	6.01	0.34	1.25	7.20E-08	10	9.8216E+06
					Average		1.9802E+07
					Std Dev		9.2899E+06
					Number		5

Table 8.25: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for KT880 CF30:

30 wt% Carbon Fiber in KetaSpire KT880 NT: From Solvay: Injection Molded at Solvay

Replicates 1 and 2 combined							
Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
6/11/15	KT880 CF30-T-12	5.9	0.32	1.25	1.85E-05	0.01	36.647
6/11/15	KT880 CF30-T-13	6.01	0.32	1.25	2.86E-05	0.01	23.271
6/11/15	KT880 CF30-T-14	6.05	0.32	1.25	2.86E-05	0.01	23.117
6/11/15	KT880 CF30-T-15	5.91	0.32	1.25	1.97E-05	0.01	34.356
6/11/15	KT880 CF30-T-1	5.91	0.32	1.25	3.93E-05	0.02	34.444
6/11/15	KT880 CF30-T-17	6.05	0.32	1.25	2.01E-05	0.01	32.893
6/11/15	KT880 CF30-T-18	6.08	0.32	1.25	1.59E-05	0.01	41.377
6/11/15	KT880 CF30-T-19	6.05	0.32	1.25	2.20E-05	0.01	30.053
					Average		32.020
					Std Dev		6.333
					Number		8
Replicate 1							
Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
6/11/15	KT880 CF30-T-12	5.9	0.32	1.25	1.85E-05	0.01	36.647
6/11/15	KT880 CF30-T-13	6.01	0.32	1.25	2.86E-05	0.01	23.271
6/11/15	KT880 CF30-T-14	6.05	0.32	1.25	2.86E-05	0.01	23.117
6/11/15	KT880 CF30-T-15	5.91	0.32	1.25	1.97E-05	0.01	34.356
					Average		29.348
					Std Dev		7.167
					Number		4
Replicate 2							
Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
6/11/15	KT880CF30-T-1	5.91	0.32	1.25	3.93E-05	0.02	34.444
6/11/15	KT880 CF30-T-17	6.05	0.32	1.25	2.01E-05	0.01	32.893
6/11/15	KT880 CF30-T-18	6.08	0.32	1.25	1.59E-05	0.01	41.377
6/11/15	KT880 CF30-T-19	6.05	0.32	1.25	2.20E-05	0.01	30.053
					Average		34.692
					Std Dev		4.814
					Number		4

Table 8.26: ASTM D257 Through-Plane Electrical Resistivity Results for 2.5 wt% Ketjenblack

EC-600 JD Carbon Black in KetaSpire KT 880 NT Extruded 2-16-16, 8-27-15

screw, Injection Molded at MTU 2-24-16

Replicates 1 and 2 combined				
Test Date	Sample Number	Applied Voltage (V)	Through-Plane Surface Electrical Resistivity (Ω/square)	Through-Plane Volume Electrical Resistivity (Ω-cm)
2/26/2016	VA2.5K- ER-24	100	3.2634E+16	1.5914E+15
2/26/2016	VA2.5K- ER-25	100	1.6329E+16	1.8780E+15
2/26/2016	VA2.5K- ER-21	100	6.9398E+15	1.7996E+15
2/26/2016	VA2.5K- ER-19	100	6.1077E+15	2.0908E+15
2/26/2016	VA2.5K- ER-13	100	2.8839E+16	1.7655E+15
2/26/2016	VA2.5K- ER-11	100	2.4346E+16	1.8525E+15
2/26/2016	VA2.5K- ER-12	100	2.6530E+16	1.9960E+15
2/26/2016	VA2.5K- ER-10	100	2.5103E+16	2.3850E+15
	Average		2.0854E+16	1.9199E+15
	Standard Deviation		9.9752E+15	2.4019E+14
	Number of Samples		8	8
Replicate 1				
Test Date	Sample Number	Applied Voltage (V)	Through-Plane Surface Electrical Resistivity (Ω/square)	Through-Plane Volume Electrical Resistivity (Ω-cm)
2/26/2016	VA2.5K- ER-19	100	6.1077E+15	2.0908E+15
2/26/2016	VA2.5K- ER-13	100	2.8839E+16	1.7655E+15
2/26/2016	VA2.5K- ER-11	100	2.4346E+16	1.8525E+15
2/26/2016	VA2.5K- ER-10	100	2.5103E+16	2.3850E+15
	Average		2.1099E+16	2.0235E+15
	Standard Deviation		1.0185E+16	2.7750E+14
	Number of Samples		4	4
Replicate 2				
Test Date	Sample Number	Applied Voltage (V)	Through-Plane Surface Electrical Resistivity (Ω/square)	Through-Plane Volume Electrical Resistivity (Ω-cm)
2/26/2016	VA2.5K- ER-24	100	3.2634E+16	1.5914E+15
2/26/2016	VA2.5K- ER-25	100	1.6329E+16	1.8780E+15
2/26/2016	VA2.5K- ER-21	100	6.9398E+15	1.7996E+15
2/26/2016	VA2.5K- ER-12	100	2.6530E+16	1.9960E+15
	Average		2.0608E+16	1.8163E+15
	Standard Deviation		1.1326E+16	1.7025E+14
	Number of Samples		4	4

Table 8.27: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for VA5K: 5 wt%

Ketjenblack EC-600 JD in KT880 NT: Extruded 2-16-16 at MTU, 8-27-15 screw,

Injection Molded at MTU 2-24-16

Replicates 1 and 2 combined							
Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
2/26/2016	VA5K-T-10	5.44	0.34	1.26	1.58E-05	0.005	24.92
2/26/2016	VA5K-T-21	5.89	0.34	1.26	1.32E-05	0.005	27.55
2/26/2016	VA5K-T-22	5.7	0.34	1.26	1.05E-05	0.005	35.79
2/26/2016	VA5K-T-23	4.8	0.34	1.26	1.55E-05	0.005	28.79
2/26/2016	VA5K-T-24	5.78	0.34	1.26	1.01E-05	0.005	36.69
2/26/2016	VA5K-T-25	5.84	0.34	1.26	1.52E-05	0.005	24.13
2/26/2016	VA5K-T-28	5.9	0.34	1.26	1.07E-05	0.005	33.93
2/26/2016	VA5K-T-29	5.98	0.34	1.26	1.24E-05	0.005	28.89
					Average		30.09
					Std Dev		4.82
					Number		8
Replicate 1							
Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
2/26/2016	VA5K-T-10	5.44	0.34	1.26	1.58E-05	0.005	24.921
2/26/2016	VA5K-T-21	5.89	0.34	1.26	1.32E-05	0.005	27.551
2/26/2016	VA5K-T-22	5.7	0.34	1.26	1.05E-05	0.005	35.789
2/26/2016	VA5K-T-23	4.8	0.34	1.26	1.55E-05	0.005	28.790
					Average		29.263
					Std Dev		4.641
					Number		4
Replicate 2							
Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
2/26/2016	VA5K-T-24	5.78	0.34	1.26	1.01E-05	0.005	36.692
2/26/2016	VA5K-T-25	5.84	0.34	1.26	1.52E-05	0.005	24.130
2/26/2016	VA5K-T-28	5.9	0.34	1.26	1.07E-05	0.005	33.930
2/26/2016	VA5K-T-29	5.98	0.34	1.26	1.24E-05	0.005	28.887
					Average		30.910
					Std Dev		5.556
					Number		4

Table 8.28: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for VA7.5K: 7.5

wt% Ketjenblack EC-600 JD in KetaSpire KT 880 NT, Extruded 3-2-16 with 8-27-15 screw, Injection Molded at MTU 5-10-16

Replicates 1 and 2 combined							
Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
5/12/2016	VA7.5K-T-24	6.1	0.34	1.26	1.05E-05	0.001	6.69
5/12/2016	VA7.5K-T-28	6.04	0.34	1.26	1.10E-05	0.001	6.45
5/12/2016	VA7.5K-T-31	6.1	0.34	1.26	1.04E-05	0.001	6.75
5/12/2016	VA7.5K-T-32	6	0.34	1.26	1.06E-05	0.001	6.74
5/12/2016	VA7.5K-T-36	6.14	0.34	1.26	1.00E-05	0.001	6.98
5/12/2016	VA7.5K-T-37	5.84	0.34	1.26	1.04E-05	0.001	7.05
5/12/2016	VA7.5K-T-34	6.15	0.34	1.26	1.00E-05	0.001	6.97
5/12/2016	VA7.5K-T-38	6.1	0.34	1.26	9.80E-06	0.001	7.17
5/12/2016	VA7.5K-T-35	6.14	0.34	1.26	1.02E-05	0.001	6.84
					Average		6.85
					Std Dev		0.22
					Number		9
Replicate 1							
Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
5/12/2016	VA7.5K-T-36	6.14	0.34	1.26	1.00E-05	0.001	6.977
5/12/2016	VA7.5K-T-37	5.84	0.34	1.26	1.04E-05	0.001	7.053
5/12/2016	VA7.5K-T-34	6.15	0.34	1.26	1.00E-05	0.001	6.966
5/12/2016	VA7.5K-T-38	6.1	0.34	1.26	9.80E-06	0.001	7.166
5/12/2016	VA7.5K-T-35	6.14	0.34	1.26	1.02E-05	0.001	6.840
					Average		7.001
					Std Dev		0.120
					Number		5
Replicate 2							
Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
5/12/2016	VA7.5K-T-24	6.1	0.34	1.26	1.05E-05	0.001	6.689
5/12/2016	VA7.5K-T-28	6.04	0.34	1.26	1.10E-05	0.001	6.448
5/12/2016	VA7.5K-T-31	6.1	0.34	1.26	1.04E-05	0.001	6.753
5/12/2016	VA7.5K-T-32	6	0.34	1.26	1.06E-05	0.001	6.736
					Average		6.656
					Std Dev		0.142
					Number		4

Table 8.29: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for VA10K: 10

wt% Ketjenblack EC-600 JD in KetaSpire KT 880 NT, Extruded 4-13-16 with 8-27-15 screw, Injection Molded at MTU 5-10-16

Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
5/12/2016	VA10K-T-31	5.74	0.34	1.25	2.60E-05	0.001	2.85
5/12/2016	VA10K-T-32	5.96	0.34	1.25	2.65E-05	0.001	2.69
5/12/2016	VA10K-T-33	5.98	0.34	1.25	2.45E-05	0.001	2.90
5/12/2016	VA10K-T-34	6	0.34	1.25	2.50E-05	0.001	2.83
5/12/2016	VA10K-T-35	5.72	0.34	1.25	2.52E-05	0.001	2.95
5/12/2016	VA10K-T-36	6.04	0.34	1.25	2.55E-05	0.001	2.76
5/12/2016	VA10K-T-37	6	0.34	1.25	2.56E-05	0.001	2.77
5/12/2016	VA10K-T-38	5.98	0.34	1.25	2.53E-05	0.001	2.81
5/12/2016	VA10K-T-39	5.93	0.34	1.25	2.55E-05	0.001	2.81
5/12/2016	VA10K-T-40	6	0.34	1.25	2.55E-05	0.001	2.78
					Average		2.81
					Std Dev		0.07
					Number		10

Table 8.30: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for VA2.5F30K:

2.5 wt% Ketjenblack EC-600 JD Carbon Black, 30 wt% Carbon Fiber from KT-880

CF30 with KT-880 NT: Extruded 6-19-16 at MTU, 8-27-15 screw, Injection

Molded at MTU 6-23-16

Replicates 1 and 2 combined							
Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
6/25/2016	VA2.5F30K-T-22	6	0.33	1.24	2.53E-05	0.01	26.96
6/25/2016	VA2.5F30K-T-26	6	0.34	1.25	2.60E-05	0.01	27.24
6/25/2016	VA2.5F30K-T-27	6.10	0.34	1.25	2.47E-05	0.01	28.21
6/25/2016	VA2.5F30K-T-28	6.30	0.34	1.25	2.41E-05	0.01	27.99
6/25/2016	VA2.5F30K-T-30	5.80	0.34	1.25	2.66E-05	0.01	27.55
6/25/2016	VA2.5F30K-T-33	6.3	0.34	1.25	2.48E-05	0.01	27.20
6/25/2016	VA2.5F30K-T-41	6.2	0.34	1.25	2.42E-05	0.01	28.33
6/25/2016	VA2.5F30K-T-43	6.2	0.34	1.25	2.40E-05	0.01	28.56
6/25/2016	VA2.5F30K-T-45	6.3	0.34	1.25	2.36E-05	0.01	28.58
					Average		27.85
					Std Dev		0.62
					Number		9
Replicate 1							
Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
6/25/2016	VA2.5F30K-T-22	6	0.33	1.24	2.53E-05	0.01	26.957
6/25/2016	VA2.5F30K-T-33	6.3	0.34	1.25	2.48E-05	0.01	27.202
6/25/2016	VA2.5F30K-T-41	6.2	0.34	1.25	2.42E-05	0.01	28.326
6/25/2016	VA2.5F30K-T-43	6.2	0.34	1.25	2.40E-05	0.01	28.562
6/25/2016	VA2.5F30K-T-45	6.3	0.34	1.25	2.36E-05	0.01	28.585
					Average		27.926
					Std Dev		0.785
					Number		5
Replicate 2							
Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
6/25/2016	VA2.5F30K-T-26	6	0.34	1.25	2.60E-05	0.01	27.244
6/25/2016	VA2.5F30K-T-27	6.10	0.34	1.25	2.47E-05	0.01	28.207
6/25/2016	VA2.5F30K-T-28	6.30	0.34	1.25	2.41E-05	0.01	27.992
6/25/2016	VA2.5F30K-T-30	5.80	0.34	1.25	2.66E-05	0.01	27.547
					Average		27.748
					Std Dev		0.434
					Number		4

Table 8.31: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for VA5F30K: 5

wt% Ketjenblack EC-600 JD Carbon Black, 30 wt% Carbon Fiber from KT-880

CF30 with KT-880 NT: Extruded 5-30-16 at MTU, 8-27-15 screw, Injection

Molded at MTU 6-7-16

Replicates 1 and 2 combined							
Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
6/9/2016	VA5F30K-T-20	6	0.34	1.25	4.20E-05	0.001	1.69
6/9/2016	VA5F30K-T-24	6.1	0.34	1.25	3.60E-05	0.001	1.94
6/9/2016	VA5F30K-T-29	6.1	0.34	1.25	3.60E-05	0.001	1.94
6/9/2016	VA5F30K-T-31	6.00	0.34	1.25	3.50E-05	0.001	2.02
6/9/2016	VA5F30K-T-40	6.00	0.34	1.25	3.20E-05	0.001	2.21
6/9/2016	VA5F30K-T-41	6.00	0.34	1.25	3.30E-05	0.001	2.15
6/9/2016	VA5F30K-T-44	6.1	0.34	1.25	3.50E-05	0.001	1.99
6/9/2016	VA5F30K-T-45	6.1	0.34	1.25	3.30E-05	0.001	2.11
6/9/2016	VA5F30K-T-46	6.1	0.34	1.25	3.60E-05	0.001	1.94
6/9/2016	VA5F30K-T-50	6	0.34	1.25	3.30E-05	0.001	2.15
					Average		2.01
					Std Dev		0.15
					Number		10
Replicate 1							
Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
6/9/2016	VA5F30K-T-41	6.00	0.34	1.25	3.30E-05	0.001	2.146
6/9/2016	VA5F30K-T-44	6.1	0.34	1.25	3.50E-05	0.001	1.991
6/9/2016	VA5F30K-T-45	6.1	0.34	1.25	3.30E-05	0.001	2.111
6/9/2016	VA5F30K-T-46	6.1	0.34	1.25	3.60E-05	0.001	1.935
6/9/2016	VA5F30K-T-50	6	0.34	1.25	3.30E-05	0.001	2.146
					Average		2.066
					Std Dev		0.097
					Number		5
Replicate 2							
Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
6/9/2016	VA5F30K-T-20	6	0.34	1.25	4.20E-05	0.001	1.687
6/9/2016	VA5F30K-T-24	6.1	0.34	1.25	3.60E-05	0.001	1.935
6/9/2016	VA5F30K-T-29	6.1	0.34	1.25	3.60E-05	0.001	1.935
6/9/2016	VA5F30K-T-31	6.00	0.34	1.25	3.50E-05	0.001	2.024
6/9/2016	VA5F30K-T-40	6.00	0.34	1.25	3.20E-05	0.001	2.214
					Average		1.959
					Std Dev		0.190
					Number		5

Table 8.32: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for VA7.5F30K:

7.5 wt% Ketjenblack EC-600 JD Carbon Black, 30 wt% Carbon Fiber from KT-880

CF30 with KT-880 NT: Extruded 5-30-16 at MTU, 8-27-15 screw, Injection

Molded at MTU 6-7-16

Replicates 1 and 2 combined							
Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
6/9/2016	VA7.5F30K-T-36	6.2	0.34	1.25	6.20E-05	0.0005	0.55
6/9/2016	VA7.5F30K-T-37	6.2	0.34	1.25	6.00E-05	0.0005	0.57
6/9/2016	VA7.5F30K-T-41	6.2	0.34	1.25	6.30E-05	0.0005	0.54
6/9/2016	VA7.5F30K-T-42	6.00	0.34	1.25	6.00E-05	0.0005	0.59
6/9/2016	VA7.5F30K-T-43	6.10	0.34	1.25	6.10E-05	0.0005	0.57
6/9/2016	VA7.5F30K-T-44	6.00	0.34	1.25	7.00E-05	0.0005	0.51
6/9/2016	VA7.5F30K-T-45	6.2	0.34	1.25	5.50E-05	0.0005	0.62
6/9/2016	VA7.5F30K-T-46	6.1	0.34	1.25	6.30E-05	0.0005	0.55
6/9/2016	VA7.5F30K-T-48	6.1	0.34	1.25	7.00E-05	0.0005	0.50
6/9/2016	VA7.5F30K-T-49	6	0.34	1.25	6.00E-05	0.0005	0.59
					Average		0.56
					Std Dev		0.04
					Number		10
Replicate 1							
Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
6/9/2016	VA7.5F30K-T-36	6.2	0.34	1.25	6.2E-05	0.0005	0.553
6/9/2016	VA7.5F30K-T-37	6.2	0.34	1.25	0.00006	0.0005	0.571
6/9/2016	VA7.5F30K-T-41	6.2	0.34	1.25	6.3E-05	0.0005	0.544
6/9/2016	VA7.5F30K-T-44	6	0.34	1.25	0.00007	0.0005	0.506
6/9/2016	VA7.5F30K-T-45	6.2	0.34	1.25	5.5E-05	0.0005	0.623
					Average		0.559
					Std Dev		0.043
					Number		5
Replicate 2							
Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
6/9/2016	VA7.5F30K-T-42	6	0.34	1.25	0.00006	0.0005	0.590
6/9/2016	VA7.5F30K-T-43	6.1	0.34	1.25	6.1E-05	0.0005	0.571
6/9/2016	VA7.5F30K-T-46	6.1	0.34	1.25	6.3E-05	0.0005	0.553
6/9/2016	VA7.5F30K-T-48	6.1	0.34	1.25	0.00007	0.0005	0.498
6/9/2016	VA7.5F30K-T-49	6	0.34	1.25	0.00006	0.0005	0.590
					Average		0.560
					Std Dev		0.038
					Number		5

Table 8.33: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for VA2.5F10K:

2.5 wt% Ketjenblack EC-600 JD Carbon Black, 10 wt% Carbon Fiber from KT-880

**CF30 with KT-880 NT: Extruded 3-2-16 at MTU, 8-27-15 screw, Injection Molded
at MTU 3-23-16**

Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
3/29/16	VA2.5F10K-T-8	5.93	0.35	1.26	2.13E-06	10	3.4914E+05
3/29/16	VA2.5F10K-T-9	5.89	0.35	1.26	8.75E-07	10	8.5569E+05
3/29/16	VA2.5F10K-T-19	5.74	0.35	1.26	4.26E-07	10	1.8035E+06
3/29/16	VA2.5F10K-T-7	7.30	0.35	1.26	2.08E-06	10	2.9044E+05
3/29/16	VA2.5F10K-T-18	5.82	0.35	1.26	3.70E-07	10	2.0479E+06
					Average		1.0693E+06
					Std Dev		8.1665E+05
					Number		5

Table 8.34: ASTM D4496 Two Probe In-Plane Electrical Resistivity Results for VA2.5F20K:

2.5 wt% Ketjenblack EC-600 JD Carbon Black, 20 wt% Carbon Fiber from KT-880

**CF30 with KT-880 NT: Extruded 4-13-16 at MTU, 8-27-15 screw, Injection
Molded at MTU 4-20-16**

Test Date	Sample Number	Length (cm)	Thickness (cm)	Width (cm)	Amps (A)	Volts (V)	Volume ER (ohm-cm)
5/12/16	VA2.5F20K-T-21	5.83	0.34	1.25	4.78E-05	5	7.6254E+03
5/12/16	VA2.5F20K-T-14	6.05	0.34	1.25	5.09E-05	5	6.9006E+03
5/12/16	VA2.5F20K-T-16	5.95	0.34	1.25	5.31E-05	5	6.7259E+03
5/12/16	VA2.5F20K-T-18	5.93	0.34	1.25	5.02E-05	5	7.1384E+03
5/12/16	VA2.5F20K-T-23	6.02	0.34	1.25	4.40E-05	5	8.0225E+03
					Average		7.2825E+03
					Std Dev		5.3424E+02
					Number		5

8.3 PEEK Thermal Conductivity Data

Table 8.35: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300

Guarded Heat Flow Meter Method for KetaSpire KT-880 NT: Injection Molded at

MTU June 30, 2015

Replicates 1 and 2 combined		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
7/9/2015	KT880-TC-5	0.2579
7/9/2015	KT880-TC-2	0.2535
7/9/2015	KT880-TC-3	0.2469
7/9/2015	KT880-TC-4	0.2515
7/9/2015	KT880-TC-28	0.2533
7/9/2015	KT880-TC-12	0.2558
7/9/2015	KT880-TC-15	0.2569
7/9/2015	KT880-TC-1	0.261
	Average	0.2546
	Standard Deviation	0.0043
	Number of Samples	8
Replicate 1		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
7/9/2015	KT880-TC-5	0.2579
7/9/2015	KT880-TC-2	0.2535
7/9/2015	KT880-TC-3	0.2469
7/9/2015	KT880-TC-4	0.2515
	Average	0.2525
	Standard Deviation	0.0046
	Number of Samples	4
Replicate 2		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
7/9/2015	KT880-TC-28	0.2533
7/9/2015	KT880-TC-12	0.2558
7/9/2015	KT880-TC-15	0.2569
7/9/2015	KT880-TC-1	0.261
	Average	0.2568
	Standard Deviation	0.0032
	Number of Samples	4

Table 8.36: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300

Guarded Heat Flow Meter Method for 2 wt% TC307 GNP in KetaSpire KT-880 NT

(VD2K): Extruded at MTU 3-17-15, 2-5-15 extruder screw, Injection Molded May

14, 2015 by Solvay

Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
6/11/2015	VD2K-TC-1	0.2731
6/11/2015	VD2K-TC-2	0.2738
6/11/2015	VD2K-TC-3	0.2723
6/11/2015	VD2K-TC-4	0.2731
	Average	0.2731
	Standard Deviation	0.0006
	Number of Samples	4

Table 8.37: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300

Guarded Heat Flow Meter Method for 4 wt% TC307 GNP in KetaSpire KT-880 NT

(VD4K): Extruded at MTU 3-17-15, 2-5-15 extruder screw: Injection Molded May

14, 2015 by Solvay

Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
6/12/2015	VD4K-TC-1	0.2863
6/12/2015	VD4K-TC-2	0.2858
6/12/2015	VD4K-TC-3	0.2855
6/12/2015	VD4K-TC-4	0.2874
	Average	0.2863
	Standard Deviation	0.0008
	Number of Samples	4

Table 8.38: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300

Guarded Heat Flow Meter Method for 6 wt% TC307 GNP in KetaSpire KT-880 NT

(VD6K): Extruded at MTU 3-17-15, 2-5-15 extruder screw: Injection Molded May

14, 2015 by Solvay

Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
6/12/2015	VD6K-TC-1	0.3056
6/12/2015	VD6K-TC-2	0.305
6/12/2015	VD6K-TC-3	0.3045
6/12/2015	VD6K-TC-4	0.3057
	Average	0.3052
	Standard Deviation	0.0006
	Number of Samples	4

Table 8.39: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300

Guarded Heat Flow Meter Method for 8 wt% TC307 GNP in KetaSpire KT-880 NT

(VD8K): Extruded at MTU 3-19-15, 2-5-15 extruder screw: Injection Molded May

14, 2015 by Solvay

Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
6/12/2015	VD8K-TC-1	0.3237
6/12/2015	VD8K-TC-2	0.3261
6/12/2015	VD8K-TC-3	0.3253
6/15/2015	VD8K-TC-5	0.3178
	Average	0.3232
	Standard Deviation	0.0038
	Number of Samples	4

Table 8.40: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300

Guarded Heat Flow Meter Method for 10 wt% TC307 GNP in KetaSpire KT-880

NT (VD10K): Extruded at MTU 3-19-15, 2-5-15 extruder screw: Injection Molded

May 14, 2015 by Solvay

Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
6/15/2015	VD10K-TC-1	0.3474
6/15/2015	VD10K-TC-2	0.3509
6/15/2015	VD10K-TC-3	0.3430
6/15/2015	VD10K-TC-4	0.3521
	Average	0.3484
	Standard Deviation	0.0041
	Number of Samples	4

Table 8.41: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300

Guarded Heat Flow Meter Method for 12.5 wt% TC307 GNP in KetaSpire KT-880

NT (VD12.5K): Extruded at MTU 4-13-16 all in zone 1, 8-27-15 extruder screw:

Injection Molded 4-20-16 at MTU

Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
4/25/2016	VD12.5K-TC-6	0.3717
4/25/2016	VD12.5K-TC-15	0.3781
4/25/2016	VD12.5K-TC-17	0.374
4/25/2016	VD12.5K-TC-20	0.3874
4/25/2016	VD12.5K-TC-13	0.3673
	Average	0.3757
	Standard Deviation	0.0076
	Number of Samples	5

Table 8.42: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300

Guarded Heat Flow Meter Method for 15 wt% TC307 GNP in KetaSpire KT-880

NT (VD10K): Extruded at MTU 8-25-15, 7-8-15 extruder screw: Injection Molded

Sept 10, 2015 at MTU

Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
9/17/2015	VD15K-TC-24	0.4268
9/17/2015	VD15K-TC-27	0.4259
9/17/2015	VD15K-TC-28	0.4281
9/17/2015	VD15K-TC-33	0.4276
	Average	0.4271
	Standard Deviation	0.0010
	Number of Samples	4

Table 8.43: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300

Guarded Heat Flow Meter Method for 2.5 wt% Ketjenblack EC-600 JD in

KetaSpire KT-880 NT (VA2.5K): Extruded at MTU 2-16-16, 8-27-15 extruder

screw: Injection Molded at MTU 2-24-16

Replicates 1 and 2 combined		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
3/2/2016	VA2.5K-TC-10	0.2789
3/2/2016	VA2.5K-TC-11	0.2782
3/2/2016	VA2.5K-TC-13	0.2806
3/2/2016	VA2.5K-TC-24	0.2806
7/12/2016	VA2.5K-TC-7	0.2788
7/12/2016	VA2.5K-TC-14	0.2826
3/2/2016	VA2.5K-TC-25	0.2785
3/2/2016	VA2.5K-TC-19	0.2735
	Average	0.2790
	Standard Deviation	0.0027
	Number of Samples	8
Replicate 1		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
3/2/2016	VA2.5K-TC-10	0.2789
3/2/2016	VA2.5K-TC-11	0.2782
3/2/2016	VA2.5K-TC-13	0.2806
3/2/2016	VA2.5K-TC-24	0.2806
	Average	0.2796
	Standard Deviation	0.0012
	Number of Samples	4
Replicate 2		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
7/12/2016	VA2.5K-TC-7	0.2788
7/12/2016	VA2.5K-TC-14	0.2826
3/2/2016	VA2.5K-TC-25	0.2785
3/2/2016	VA2.5K-TC-19	0.2735
	Average	0.2784
	Standard Deviation	0.0037
	Number of Samples	4

Table 8.44: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300

Guarded Heat Flow Meter Method for 5 wt% Ketjenblack EC-600 JD in KetaSpire

KT-880 NT (VA5K): Extruded at MTU 2-16-16, 8-27-15 extruder screw: Injection

Molded at MTU 2-24-16

Replicates 1 and 2 combined		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
3/2/2016	VA5K-TC-12	0.3064
3/2/2016	VA5K-TC-8	0.2984
3/2/2016	VA5K-TC-13	0.312
3/2/2016	VA5K-TC-13	0.3094
7/12/2016	VA5K-TC-10	0.3084
7/12/2016	VA5K-TC-18	0.3049
3/2/2016	VA5K-TC-16	0.3021
3/2/2016	VA5K-TC-9	0.3103
	Average	0.3065
	Standard Deviation	0.0045
	Number of Samples	8
Replicate 1		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
3/2/2016	VA5K-TC-12	0.3064
3/2/2016	VA5K-TC-8	0.2984
3/2/2016	VA5K-TC-13	0.312
3/2/2016	VA5K-TC-13	0.3094
	Average	0.3066
	Standard Deviation	0.0059
	Number of Samples	4
Replicate 2		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
7/12/2016	VA5K-TC-10	0.3084
7/12/2016	VA5K-TC-18	0.3049
3/2/2016	VA5K-TC-16	0.3021
3/2/2016	VA5K-TC-9	0.3103
	Average	0.3064
	Standard Deviation	0.0036
	Number of Samples	4

Table 8.45: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300

Guarded Heat Flow Meter Method for 7.5 wt% Ketjenblack EC-600 JD in

KetaSpire KT-880 NT (VA7.5K): Extruded at MTU 3-2-16, 8-27-15 extruder screw:

Injection Molded at MTU 5-10-16

Replicates 1 and 2 combined		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
5/12/2016	VA7.5K-TC-8	0.3192
5/12/2016	VA7.5K-TC-10	0.3266
7/12/2016	VA7.5K-TC-5	0.329
7/12/2016	VA7.5K-TC-13	0.333
7/13/2016	VA7.5K-TC-7	0.3352
5/12/2016	VA7.5K-TC-11	0.3317
5/12/2016	VA7.5K-TC-15	0.3262
5/12/2016	VA7.5K-TC-16	0.3245
	Average	0.3282
	Standard Deviation	0.0052
	Number of Samples	8
Replicate 1		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
5/12/2016	VA7.5K-TC-8	0.3192
5/12/2016	VA7.5K-TC-10	0.3266
7/12/2016	VA7.5K-TC-5	0.329
7/12/2016	VA7.5K-TC-13	0.333
	Average	0.3270
	Standard Deviation	0.0058
	Number of Samples	4
Replicate 2		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
7/13/2016	VA7.5K-TC-7	0.3352
5/12/2016	VA7.5K-TC-11	0.3317
5/12/2016	VA7.5K-TC-15	0.3262
5/12/2016	VA7.5K-TC-16	0.3245
	Average	0.3294
	Standard Deviation	0.0049
	Number of Samples	4

Table 8.46: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300

Guarded Heat Flow Meter Method for 10 wt% Ketjenblack EC-600 JD in

KetaSpire KT-880 NT (VA10K): Extruded at MTU 4-13-16, 8-27-15 extruder

screw: Injection Molded at MTU 5-10-16

Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
5/12/2016	VA10K-TC-10	0.3625
5/12/2016	VA10K-TC-4	0.347
5/12/2016	VA10K-TC-14	0.3509
5/12/2016	VA10K-TC-12	0.3524
	Average	0.3532
	Standard Deviation	0.0066
	Number of Samples	4

Table 8.47: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300

Guarded Heat Flow Meter Method for 10 wt% carbon fiber from KT-880 CF30 in

KetaSpire KT-880 NT (VF10K): Extruded at MTU 3-2-16, 8-27-15 extruder screw:

Injection Molded at MTU 3-23-16

Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
3/30/2016	VF10K-TC-5	0.2893
3/30/2016	VF10K-TC-6	0.2871
3/30/2016	VF10K-TC-7	0.2888
3/30/2016	VF10K-TC-8	0.2781
3/30/2016	VF10K-TC-10	0.2745
3/30/2016	VF10K-TC-18	0.2885
	Average	0.2844
	Standard Deviation	0.0064
	Number of Samples	6

Table 8.48: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300

Guarded Heat Flow Meter Method for 20 wt% carbon fiber from KT-880 CF30 in

KetaSpire KT-880 NT (VF10K): Extruded at MTU 4-13-16, 8-27-15 extruder

screw: Injection Molded at MTU 4-20-16

Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
4/25/2016	VF20K-TC-9	0.3357
4/25/2016	VF20K-TC-10	0.3313
4/25/2016	VF20K-TC-14	0.3489
4/25/2016	VF20K-TC-16	0.3379
4/25/2016	VF20K-TC-17	0.3352
	Average	0.3378
	Standard Deviation	0.0066
	Number of Samples	5

Table 8.49: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300

Guarded Heat Flow Meter Method for KetaSpire KT-880 CF30 (30 wt% Carbon

Fiber): From Solvay: Injection Molded May 14, 2015 by Solvay

Replicates 1 and 2 combined		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
1/21/2016	KT880 CF30-TC-1	0.4018
1/21/2016	KT880 CF30-TC-2	0.4273
1/21/2016	KT880 CF30-TC-3	0.4271
1/21/2016	KT880 CF30-TC-4	0.3947
7/12/2016	KT880 CF30-TC-13	0.421
7/12/2016	KT880 CF30-TC-14	0.4034
7/12/2016	KT880 CF30-TC-19	0.4042
7/12/2016	KT880 CF30-TC-20	0.4367
	Average	0.4145
	Standard Deviation	0.0153
	Number of Samples	8
Replicate 1		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
1/21/2016	KT880 CF30-TC-1	0.4018
1/21/2016	KT880 CF30-TC-2	0.4273
1/21/2016	KT880 CF30-TC-3	0.4271
1/21/2016	KT880 CF30-TC-4	0.3947
	Average	0.4127
	Standard Deviation	0.0170
	Number of Samples	4
Replicate 2		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
7/12/2016	KT880 CF30-TC-13	0.421
7/12/2016	KT880 CF30-TC-14	0.4034
7/12/2016	KT880 CF30-TC-19	0.4042
7/12/2016	KT880 CF30-TC-20	0.4367
	Average	0.4163
	Standard Deviation	0.0158
	Number of Samples	4

Table 8.50: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300

Guarded Heat Flow Meter Method for 2.5 wt% Ketjenblack EC-600 JD, 30 wt% carbon fiber from KT-880 CF30 in KetaSpire KT-880 NT (VA2.5F30K): Extruded at MTU 6-19-16, 8-27-15 extruder screw: Injection Molded at MTU 6-23-16

Replicates 1 and 2 combined		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
7/6/2016	VA2.5F30K-TC-7	0.4216
7/6/2016	VA2.5F30K-TC-13	0.4273
7/6/2016	VA2.5F30K-TC-16	0.4341
7/6/2016	VA2.5F30K-TC-15	0.4231
7/6/2016	VA2.5F30K-TC-8	0.4211
7/6/2016	VA2.5F30K-TC-16	0.4341
7/14/2016	VA2.5F30K-TC-10	0.4294
7/14/2016	VA2.5F30K-TC-16	0.4217
	Average	0.4266
	Standard Deviation	0.0055
	Number of Samples	8
Replicate 1		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
7/6/2016	VA2.5F30K-TC-7	0.4216
7/6/2016	VA2.5F30K-TC-13	0.4273
7/6/2016	VA2.5F30K-TC-16	0.4341
7/6/2016	VA2.5F30K-TC-15	0.4231
	Average	0.4265
	Standard Deviation	0.0056
	Number of Samples	4
Replicate 2		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
7/6/2016	VA2.5F30K-TC-8	0.4211
7/6/2016	VA2.5F30K-TC-16	0.4341
7/14/2016	VA2.5F30K-TC-10	0.4294
7/14/2016	VA2.5F30K-TC-16	0.4217
	Average	0.4266
	Standard Deviation	0.0063
	Number of Samples	4

Table 8.51: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300

Guarded Heat Flow Meter Method for 5 wt% Ketjenblack EC-600 JD, 30 wt%

carbon fiber from KT-880 CF30 in KetaSpire KT-880 NT (VA2.5F20K): Extruded

at MTU 5-30-16, 8-27-15 extruder screw: Injection Molded at MTU 6-7-16

Replicates 1 and 2 combined		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
6/9/2016	VA5F30K-TC-6	0.4876
6/9/2016	VA5F30K-TC-11	0.4731
6/9/2016	VA5F30K-TC-12	0.4709
6/9/2016	VA5F30K-TC-13	0.4754
7/14/2016	VA5F30K-TC-5	0.4627
7/13/2016	VA5F30K-TC-2	0.4796
7/13/2016	VA5F30K-TC-7	0.4674
7/14/2016	VA5F30K-TC-9	0.4692
	Average	0.4732
	Standard Deviation	0.0077
	Number of Samples	8
Replicate 1		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
6/9/2016	VA5F30K-TC-6	0.4876
6/9/2016	VA5F30K-TC-11	0.4731
6/9/2016	VA5F30K-TC-12	0.4709
6/9/2016	VA5F30K-TC-13	0.4754
	Average	0.4768
	Standard Deviation	0.0075
	Number of Samples	4
Replicate 2		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
7/14/2016	VA5F30K-TC-5	0.4627
7/13/2016	VA5F30K-TC-2	0.4796
7/13/2016	VA5F30K-TC-7	0.4674
7/14/2016	VA5F30K-TC-9	0.4692
	Average	0.4697
	Standard Deviation	0.0071
	Number of Samples	4

Table 8.52: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300

Guarded Heat Flow Meter Method for 7.5 wt% Ketjenblack EC-600 JD, 30 wt% carbon fiber from KT-880 CF30 in KetaSpire KT-880 NT (VA2.5F20K): Extruded at MTU 5-30-16, 8-27-15 extruder screw: Injection Molded at MTU 6-7-16

Replicates 1 and 2 combined		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
6/13/2016	VA7.5F30K-TC-14	0.5232
6/13/2016	VA7.5F30K-TC-16	0.5153
6/13/2016	VA7.5F30K-TC-17	0.518
6/13/2016	VA7.5F30K-TC-18	0.5257
7/12/2016	VA7.5F30K-TC-28	0.5167
7/12/2016	VA7.5F30K-TC-19	0.5029
7/12/2016	VA7.5F30K-TC-15	0.5155
6/13/2016	VA7.5F30K-TC-12	0.5015
	Average	0.5149
	Standard Deviation	0.0086
	Number of Samples	8
Replicate 1		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
6/13/2016	VA7.5F30K-TC-14	0.5232
6/13/2016	VA7.5F30K-TC-16	0.5153
6/13/2016	VA7.5F30K-TC-17	0.518
6/13/2016	VA7.5F30K-TC-18	0.5257
	Average	0.5206
	Standard Deviation	0.0047
	Number of Samples	4
Replicate 2		
Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
7/12/2016	VA7.5F30K-TC-28	0.5167
7/12/2016	VA7.5F30K-TC-19	0.5029
7/12/2016	VA7.5F30K-TC-15	0.5155
6/13/2016	VA7.5F30K-TC-12	0.5015
	Average	0.5092
	Standard Deviation	0.0081
	Number of Samples	4

Table 8.53: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300

Guarded Heat Flow Meter Method for 2.5 wt% Ketjenblack EC-600 JD, 10 wt% carbon fiber from KT-880 CF30 in KetaSpire KT-880 NT (VA2.5F10K): Extruded at MTU 3-2-16, 8-27-15 extruder screw: Injection Molded at MTU 3-23-16

Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
4/4/2016	VA2.5F10K-TC-3	0.3058
4/4/2016	VA2.5F10K-TC-6	0.3063
4/4/2016	VA2.5F10K-TC-8	0.3112
4/4/2016	VA2.5F10K-TC-9	0.3239
4/4/2016	VA2.5F10K-TC-10	0.3114
4/4/2016	VA2.5F10K-TC-14	0.3255
	Average	0.3140
	Standard Deviation	0.0086
	Number of Samples	6

Table 8.54: Through-Plane Thermal Conductivity at 55°C using Holometrix TCA-300

Guarded Heat Flow Meter Method for 2.5 wt% Ketjenblack EC-600 JD, 20 wt% carbon fiber from KT-880 CF30 in KetaSpire KT-880 NT (VA2.5F20K): Extruded at MTU 4-13-16, 8-27-15 extruder screw: Injection Molded at MTU 4-20-16

Test Date	Sample Number	Through Plane Thermal Conductivity (W/m•K)
4/29/2016	VA2.5F20K-TC-15	0.3651
4/29/2016	VA2.5F20K-TC-18	0.366
4/29/2016	VA2.5F20K-TC-19	0.3737
4/29/2016	VA2.5F20K-TC-22	0.3738
4/29/2016	VA2.5F20K-TC-8	0.3673
	Average	0.3692
	Standard Deviation	0.0042
	Number of Samples	5

8.4 PEEK Tensile Data

Figure 8.4: Tensile Results for KetaSpire KT-880 NT Injection Molded 6-30-15at MTU

Table 8.55: Tensile Results for KetaSpire KT-880 NT Injection Molded 6-30-15 at MTU

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
17	95.499	4.814	73.116	37.150	3.745
28	95.297	4.766	72.444	34.893	3.804
18	95.275	5.057	72.751	34.125	3.573
20	95.353	4.701	73.418	33.636	3.850
21	95.366	4.882	72.877	34.117	3.590
25	95.381	4.889	72.239	34.892	3.650
Average	95.36	4.85	72.81	34.80	3.70
Std Dev	0.08	0.12	0.43	1.25	0.11
Count	6	6	6	6	6

Figure 8.5 Tensile Results for VD2K: 2 wt% TC307 GNP in KetaSpire KT-880 NT: Injection

Molded at Solvay 5-14-15, Extruded March 2015 at MTU

Table 8.56: Tensile Results for VD2K: 2 wt% TC307 GNP in KetaSpire KT-880 NT: Injection

Molded at Solvay 5-14-15, Extruded March 2015 at MTU

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
16	94.764	5.711	78.374	25.313	3.770
17	94.166	5.614	75.883	24.319	3.796
20	95.162	5.410	76.150	21.580	3.858
22	95.622	5.418	77.257	26.221	3.878
23	95.122	5.979	77.310	33.590	3.691
24	95.243	5.344	76.304	21.200	3.888
25	94.914	5.484	75.906	32.136	3.757
Average	95.00	5.57	76.74	26.34	3.81
Std Dev	0.46	0.22	0.93	4.84	0.07
Count	7	7	7	7	7

Figure 8.6: Tensile Results for VD4K: 4 wt% TC307 GNP in KetaSpire KT-880 NT: Injection

Molded at Solvay 5-14-15, Extruded March 2015 at MTU

Table 8.57: Tensile Results for VD4K: 4 wt% TC307 GNP in KetaSpire KT-880 NT: Injection

Molded at Solvay 5-14-15, Extruded March 2015 at MTU

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
16	95.345	5.273	87.502	9.483	4.076
17	95.825	5.329	80.597	16.100	4.000
19	94.800	5.616	79.139	23.271	3.874
20	95.352	5.300	79.630	15.334	3.931
21	95.575	5.492	80.388	19.770	3.976
Average	95.38	5.40	81.45	16.79	3.97
Std Dev	0.38	0.15	3.43	5.17	0.08
Count	5	5	5	5	5

Figure 8.7: Tensile Results for VD6K: 6 wt% TC307 GNP in KetaSpire KT-880 NT: Injection

Molded at Solvay 5-14-15, Extruded March 2015 at MTU

Table 8.58: Tensile Results for VD6K: 6 wt% TC307 GNP in KetaSpire KT-880 NT: Injection

Molded at Solvay 5-14-15, Extruded March 2015 at MTU

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
16	94.470	5.279	90.361	10.184	4.269
17	95.192	5.416	87.736	13.605	4.235
18	93.944	5.291	87.590	11.647	4.227
19	95.110	5.156	85.940	16.237	4.254
21	95.542	5.104	87.480	14.153	4.163
Average	94.85	5.25	87.82	13.17	4.23
Std Dev	0.64	0.12	1.59	2.33	0.04
Count	5	5	5	5	5

Figure 8.8: Tensile Results for VD8K: 8 wt% TC307 GNP in KetaSpire KT-880 NT: Injection

Molded at Solvay 5-14-15, Extruded March 2015 at MTU

Table 8.59: Tensile Results for VD8K: 8 wt% TC307 GNP in KetaSpire KT-880 NT: Injection

Molded at Solvay 5-14-15, Extruded March 2015 at MTU

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
16	95.477	5.147	93.216	8.489	4.478
19	95.598	4.897	93.781	6.779	4.513
20	94.958	5.351	90.661	11.125	4.250
21	95.271	5.458	93.020	8.996	4.297
22	95.504	5.268	93.657	6.988	4.376
Average	95.36	5.22	92.87	8.48	4.38
Std Dev	0.26	0.22	1.27	1.76	0.11
Count	5	5	5	5	5

Figure 8.9: Tensile Results for VD10K: 10 wt% TC307 GNP in KetaSpire KT-880 NT:

Injection Molded at Solvay 5-14-15, Extruded March 2015 at MTU

Table 8.60: Tensile Results for VD10K: 10 wt% TC307 GNP in KetaSpire KT-880 NT:

Injection Molded at Solvay 5-14-15, Extruded March 2015 at MTU

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
21	94.512	4.883	94.165	5.321	4.545
22	95.018	4.909	93.355	7.166	4.587
24	95.189	4.819	94.252	6.217	4.594
25	95.682	4.951	95.642	5.189	4.567
26	95.377	5.108	94.815	6.164	4.480
Average	95.16	4.93	94.45	6.01	4.55
Std Dev	0.44	0.11	0.85	0.80	0.05
Count	5	5	5	5	5

Figure 8.10: Tensile Results for VD12.5K: 12.5 wt% TC307 GNP in KetaSpire KT-880 NT:

Extruded April 13 2016 at MTU, IM at MTU April 20 2016

Table 8.61: Tensile Results for VD12.5K: 12.5 wt% TC307 GNP in KetaSpire KT-880 NT:

Extruded April 13 2016 at MTU, IM at MTU April 20 2016

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
9	90.837	2.903	90.837	2.903	4.548
8	89.284	2.523	89.284	2.523	4.942
6	93.020	2.758	93.020	2.758	4.878
13	87.557	2.538	87.557	2.538	4.647
17	87.021	2.449	87.021	2.449	4.951
14	92.488	3.012	92.488	3.012	4.866
Average	90.03	2.70	90.03	2.70	4.81
Std Dev	2.50	0.23	2.50	0.23	0.17
Count	6	6	6	6	6

Figure 8.11: Tensile Results for VD15K: 15 wt% TC307 GNP in KetaSpire KT-880 NT:

Extruded 8-25-15 at MTU screw 7-8-15, IM at MTU Sept 10 2015

Table 8.62: Tensile Results for VD15K: 15 wt% TC307 GNP in KetaSpire KT-880 NT:

Extruded 8-25-15 at MTU screw 7-8-15, IM at MTU Sept 10 2015

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
23	81.378	2.279	81.378	2.279	4.621
28	85.252	2.183	85.252	2.183	5.147
30	77.590	2.054	77.590	2.054	4.731
34	84.465	2.219	84.465	2.219	5.067
38	79.272	2.034	79.272	2.034	5.119
44	80.142	2.022	80.142	2.022	4.935
54	78.896	2.112	78.896	2.112	4.759
56	81.854	2.216	81.854	2.216	4.825
Average	81.11	2.14	81.11	2.14	4.90
Std Dev	2.69	0.10	2.69	0.10	0.20
Count	8	8	8	8	8

Figure 8.12: Tensile Results for VF10K: 10 wt% Carbon Fiber from KT-880 CF30 in KetaSpire KT-880 NT: Extruded 3-2-16 at MTU screw 8-27-15, IM at MTU 3-23-16

Table 8.63: Tensile Results for VF10K: 10 wt% Carbon Fiber from KT-880 CF30 in KetaSpire KT-880 NT: Extruded 3-2-16 at MTU screw 8-27-15, IM at MTU 3-23-16

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
8	117.240	3.678	117.240	3.678	6.118
7	117.316	3.826	117.316	3.826	5.980
5	118.943	3.699	118.943	3.699	5.998
11	116.982	4.257	115.324	5.284	5.815
19	114.093	3.026	114.093	3.026	6.296
27	113.694	2.928	113.694	2.928	6.412
28	116.122	3.148	116.122	3.148	6.769
31	117.129	4.394	115.450	5.494	5.980
Average	116.44	3.62	116.02	3.89	6.17
Std Dev	1.76	0.55	1.76	0.99	0.31
Count	8	8	8	8	8

Figure 8.13: Tensile Results for VF20K: 20 wt% Carbon Fiber from KT-880 CF30 in KetaSpire KT-880 NT: Extruded 4-13-16 at MTU screw 8-27-15, IM at MTU April 20 2016

Table 8.64: Tensile Results for VF20K: 20 wt% Carbon Fiber from KT-880 CF30 in KetaSpire KT-880 NT: Extruded 4-13-16 at MTU screw 8-27-15, IM at MTU April 20 2016

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
13	143.640	3.108	143.640	3.108	10.164
20	143.721	2.771	143.721	2.771	10.369
19	143.952	2.850	143.952	2.850	10.694
21	144.708	3.092	144.708	3.092	10.863
22	143.937	2.958	143.937	2.958	11.422
Average	143.99	2.96	143.99	2.96	10.70
Std Dev	0.42	0.15	0.42	0.15	0.49
Count	5	5	5	5	5

Figure 8.14: Tensile Results for VF30K: 30 wt% Carbon Fiber from KT-880 CF30, IM at Solvay May 2015

Table 8.65: Tensile Results for VF30K: 30 wt% Carbon Fiber from KT-880 CF30, IM at Solvay May 2015

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
12	229.298	1.841	229.298	1.841	23.535
13	230.504	1.933	230.504	1.933	24.755
14	225.018	2.052	225.018	2.052	22.465
16	231.392	1.945	231.392	1.945	23.733
17	230.978	1.895	230.978	1.895	23.196
21	233.573	2.074	233.573	2.074	23.310
24	230.848	1.936	230.848	1.936	22.576
26	230.904	1.938	230.904	1.938	23.43
27	231.213	1.915	231.213	1.915	24.231
Average	230.41	1.95	230.41	1.95	23.47
Std Dev	2.31	0.07	2.31	0.07	0.73
Count	9	9	9	9	9

Figure 8.15: Tensile Results for VA2.5K: 2.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black in Solvay KetaSpire KT-880 NT: Extruded 2-16-16 at MTU screw 8-27-15, IM at MTU 2-24-16

Table 8.66: Tensile Results for VA2.5K: 2.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black in Solvay KetaSpire KT-880 NT: Extruded 2-16-16 at MTU screw 8-27-15, IM at MTU 2-24-16

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
19	85.704	2.879	85.704	2.879	4.084
23	92.646	3.390	92.646	3.390	3.938
26	85.139	2.749	85.139	2.749	4.187
24	92.404	3.196	92.404	3.196	4.324
40	86.569	3.083	86.569	3.083	3.885
47	89.839	3.185	89.839	3.185	3.988
28	90.828	2.967	90.828	2.967	4.217
25	90.335	3.191	90.335	3.191	3.821
Average	89.18	3.08	89.18	3.08	4.06
Std Dev	2.98	0.21	2.98	0.21	0.18
Count	8	8	8	8	8

**Figure 8.16: Tensile Results for VA5K: 5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon
Black in Solvay KetaSpire KT-880 NT: Extruded 2-16-16 at MTU screw 8-27-15,
IM at MTU 2-24-16**

**Table 8.67: Tensile Results for VA5K: 5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon
Black in Solvay KetaSpire KT-880 NT: Extruded 2-16-16 at MTU screw 8-27-15,
IM at MTU 2-24-16**

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
1	89.290	3.348	89.290	3.348	3.876
8	86.602	3.073	86.602	3.073	3.894
15	85.406	2.440	85.406	2.440	4.400
19	92.217	3.535	92.217	3.535	4.014
13	92.216	3.426	92.216	3.426	4.062
11	90.631	2.885	90.631	2.885	4.053
Average	89.39	3.12	89.39	3.12	4.06
Std Dev	2.87	0.41	2.87	0.41	0.19
Count	6	6	6	6	6

Figure 8.17: Tensile Results for VA7.5K: 7.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black in Solvay KetaSpire KT-880 NT: Extruded 3-2-16 at MTU screw 8-27-15, IM at MTU 5-10-16

Table 8.68: Tensile Results for VA7.5K: 7.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black in Solvay KetaSpire KT-880 NT: Extruded 3-2-16 at MTU screw 8-27-15, IM at MTU 5-10-16

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
23	91.974	2.876	91.974	2.876	4.197
21	90.975	2.653	90.975	2.653	4.227
66	87.124	2.622	87.124	2.622	4.281
64	93.454	2.892	93.454	2.892	4.199
65	89.772	2.632	89.772	2.632	4.432
69	84.275	2.507	84.275	2.507	4.396
62	88.144	2.604	88.144	2.604	4.104
Average	89.39	2.68	89.39	2.68	4.26
Std Dev	3.13	0.14	3.13	0.14	0.12
Count	7	7	7	7	7

Figure 8.18: Tensile Results for VA10K: 10 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black in Solvay KetaSpire KT-880 NT: Extruded 3-2-16 at MTU screw 8-27-15, IM at MTU 5-10-16

Table 8.69: Tensile Results for VA10K: 10 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black in Solvay KetaSpire KT-880 NT: Extruded 3-2-16 at MTU screw 8-27-15, IM at MTU 5-10-16

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
20	78.214	1.966	78.214	1.966	4.483
23	82.024	2.150	82.024	2.150	4.545
20	78.214	1.966	78.214	1.966	4.487
21	70.757	1.669	70.757	1.669	4.845
24	83.709	2.140	83.709	2.140	4.467
25	85.087	2.243	85.087	2.243	4.557
26	83.612	2.300	83.612	2.300	4.530
28	79.385	1.947	79.385	1.947	4.775
29	78.883	1.952	78.883	1.952	4.597
Average	79.99	2.04	79.99	2.04	4.59
Std Dev	4.33	0.19	4.33	0.19	0.13
Count	9	9	9	9	9

Figure 8.19: Tensile Results for VA2.5F30K: 2.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black and 30 wt% Carbon Fiber from Solvay KT-880 CF30 in Solvay KetaSpire KT-880 NT: Extruded 6-19-16 at MTU screw 8-27-15, IM at MTU 6-23-16

Table 8.70: Tensile Results for VA2.5F30K: 2.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black and 30 wt% Carbon Fiber from Solvay KT-880 CF30 in Solvay KetaSpire KT-880 NT: Extruded 6-19-16 at MTU screw 8-27-15, IM at MTU 6-23-16

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
37	166.971	1.964	166.971	1.964	16.289
32	166.655	2.022	166.655	2.022	15.830
24	164.341	1.869	164.341	1.869	17.021
14	164.867	2.008	164.867	2.008	15.235
17	166.266	2.025	166.266	2.025	16.014
34	166.400	1.844	166.400	1.844	17.409
31	165.586	1.858	165.586	1.858	17.363
16	163.876	1.764	163.876	1.764	17.663
12	163.105	2.061	163.105	2.061	16.042
15	163.725	1.776	163.725	1.776	17.339
23	163.164	1.942	163.164	1.942	16.723
Average	165.00	1.92	165.00	1.92	16.63
Std Dev	1.44	0.10	1.44	0.10	0.79
Count	11	11	11	11	11

Figure 8.20: Tensile Results for VA5F30K: 5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black and 30 wt% Carbon Fiber from Solvay KT-880 CF30 in Solvay KetaSpire KT-880 NT: Extruded 5-30-16 at MTU screw 8-27-15, IM at MTU 6-7-16

Table 8.71: Tensile Results for VA5F30K: 5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black and 30 wt% Carbon Fiber from Solvay KT-880 CF30 in Solvay KetaSpire KT-880 NT: Extruded 5-30-16 at MTU screw 8-27-15, IM at MTU 6-7-16

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
14	145.141	1.558	145.141	1.558	16.083
12	142.854	1.488	142.854	1.488	16.626
26	144.529	1.534	144.529	1.534	15.527
28	144.644	1.517	144.644	1.517	15.941
27	143.675	1.518	143.675	1.518	16.717
Average	144.17	1.52	144.17	1.52	16.18
Std Dev	0.90	0.03	0.90	0.03	0.50
Count	5	5	5	5	5

Figure 8.21: Tensile Results for VA7.5F30K: 7.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black and 30 wt% Carbon Fiber from Solvay KT-880 CF30 in Solvay KetaSpire KT-880 NT: Extruded 5-30-16 at MTU screw 8-27-15, IM at MTU 6-7-16

Table 8.72: Tensile Results for VA7.5F30K: 7.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black and 30 wt% Carbon Fiber from Solvay KT-880 CF30 in Solvay KetaSpire KT-880 NT: Extruded 5-30-16 at MTU screw 8-27-15, IM at MTU 6-7-16

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
16	139.446	1.189	139.446	1.189	17.711
18	149.973	1.397	149.973	1.397	19.548
19	149.126	1.388	149.126	1.388	18.537
30	142.699	1.257	142.699	1.257	17.012
34	142.529	1.188	142.529	1.188	17.564
31	145.727	1.296	145.727	1.296	20.175
Average	144.92	1.29	144.92	1.29	18.42
Std Dev	4.11	0.09	4.11	0.09	1.23
Count	6	6	6	6	6

Figure 8.22: Tensile Results for VA2.5F10K: 2.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black and 10 wt% Carbon Fiber from Solvay KT-880 CF30 in Solvay KetaSpire KT-880 NT: Extruded 3-2-16 at MTU screw 8-27-15, IM at MTU 3-23-16

Table 8.73: Tensile Results for VA2.5F10K: 2.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black and 10 wt% Carbon Fiber from Solvay KT-880 CF30 in Solvay KetaSpire KT-880 NT: Extruded 3-2-16 at MTU screw 8-27-15, IM at MTU 3-23-16

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
13	118.571	3.945	117.495	4.680	6.450
14	118.229	3.705	118.229	3.705	6.277
15	118.196	3.464	118.196	3.464	6.370
28	118.936	4.194	117.471	5.108	6.208
30	117.924	3.317	117.924	3.317	6.853
25	114.281	2.815	114.281	2.815	6.449
10	113.733	2.771	113.733	2.771	6.819
35	119.754	4.044	118.728	4.934	6.352
34	117.100	3.104	117.100	3.104	6.776
3	119.161	4.354	117.695	5.294	6.151
Average	117.59	3.57	117.09	3.92	6.47
Std Dev	2.02	0.57	1.69	0.98	0.26
Count	10	10	10	10	10

Figure 8.23: Tensile Results for VA2.5F20K: 2.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black and 20 wt% Carbon Fiber from Solvay KT-880 CF30 in Solvay KetaSpire KT-880 NT: Extruded 4-13-16 at MTU screw 8-27-15, IM at MTU 4-20-16

Table 8.74: Tensile Results for VA2.5F20K: 2.5 wt% Akzo Nobel Ketjenblack EC-600 JD Carbon Black and 20 wt% Carbon Fiber from Solvay KT-880 CF30 in Solvay KetaSpire KT-880 NT: Extruded 4-13-16 at MTU screw 8-27-15, IM at MTU 4-20-16

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
7	145.766	2.830	145.766	2.830	10.944
10	145.188	2.924	145.188	2.924	11.729
20	144.963	2.586	144.963	2.586	10.112
17	145.625	2.795	145.625	2.795	10.381
19	144.572	2.636	144.572	2.636	10.981
29	145.834	2.766	145.834	2.766	11.429
27	144.949	2.670	144.949	2.670	11.004
24	144.547	2.539	144.547	2.539	11.416
Average	145.18	2.72	145.18	2.72	11.00
Std Dev	0.51	0.13	0.51	0.13	0.54
Count	8	8	8	8	8

8.5 Carbon Black and Fumed Silica Electrical Resistivity Data

Table 8.75: Electrical Resistivity Results for Neat Cycloaliphatic Epoxy

Sample Number	Applied Voltage (V)	Through-Plane Volume Electrical Resistivity (Ω -cm)
I310-3-25-15-1	100	1.1172E+16
I310-3-25-15-2	100	1.1916E+16
I310-3-25-15-3	100	6.7905E+15
I310-3-25-15-4	100	8.7036E+15
Average		9.65E+15
Standard Deviation		2.35E+15
Number of Samples		4

Table 8.76: Electrical Resistivity Results for 1 wt% Cabot Carbon Black 2 in Cycloaliphatic Epoxy

Sample Number	Applied Voltage (V)	Through-Plane Volume Electrical Resistivity (Ω -cm)
I310-X-1-4-27-15-1	10	4.3634E+10
I310-X-1-4-27-15-2	10	3.2553E+10
I310-X-1-4-27-15-3	10	8.0132E+10
I310-X-1-4-27-15-4	10	5.4832E+10
I310-X-1-4-27-15-5	10	7.9531E+10
Average		5.81E+10
Standard Deviation		2.13E+10
Number of Samples		5

Table 8.77: Electrical Resistivity Results for 1 wt% Cabot Carbon Black 3 in Cycloaliphatic

Epoxy

Sample Number	Applied Voltage (V)	Through-Plane Volume Electrical Resistivity (Ω -cm)
I310-Y-1-4-2-15-1	10	1.2018E+12
I310-Y-1-4-2-15-2	10	5.1735E+11
I310-Y-1-4-2-15-3	10	3.4702E+11
I310-Y-1-4-2-15-4	10	1.1602E+12
I310-Y-1-4-2-15-5	10	6.4284E+11
Average		7.74E+11
Standard Deviation		3.87E+11
Number of Samples		5

Table 8.78: Electrical Resistivity Results for 0.5 wt% Cabot Carbon Black 1 in Cycloaliphatic

Epoxy

Sample Number	Applied Voltage (V)	Through-Plane Surface Electrical Resistivity (Ω /square)	Through-Plane Volume Electrical Resistivity (Ω -cm)
I310-W-0.5-10-1-15-1	100	3.68E+15	4.94E+14
I310-W-0.5-10-1-15-2	100	6.87E+15	9.94E+14
I310-W-0.5-10-1-15-3	100	4.29E+15	4.13E+14
I310-W-0.5-10-1-15-4	100	5.62E+15	1.12E+15
Average		5.11E+15	7.55E+14
Standard Deviation		1.42E+15	3.53E+14
Number of Samples		4	4

Table 8.79: Electrical Resistivity Results for 1 wt% Cabot Carbon Black 1 in Cycloaliphatic Epoxy

Sample Number	Applied Voltage (V)	Through-Plane Surface Electrical Resistivity (Ω/square)	Through-Plane Volume Electrical Resistivity ($\Omega\text{-cm}$)
I-310-W-1-10-22-15-1	100	2.65E+15	1.78E+14
I-310-W-1-10-22-15-2	100	3.41E+15	2.34E+14
I-310-W-1-10-22-15-3	100	3.27E+15	2.29E+14
I-310-W-1-10-22-15-4	100	3.65E+15	2.07E+14
Average		3.25E+15	2.12E+14
Standard Deviation		4.26E+14	2.54E+13
Number of Samples		5	5

Table 8.80: Electrical Resistivity Results for 2 wt% Carbon Black 1 in Cycloaliphatic Epoxy

Sample Number	Applied Voltage (V)	Through-Plane Surface Electrical Resistivity (Ω/square)	Through-Plane Volume Electrical Resistivity ($\Omega\text{-cm}$)
I310-W-2-10-29-15-1	100	7.89E+14	1.02E+14
I310-W-2-10-29-15-2	100	4.60E+14	2.17E+13
I310-W-2-10-29-15-3	100	6.70E+14	6.82E+13
I310-W-2-10-29-15-4	100	3.22E+14	1.99E+13
Average		5.60E+14	5.28E+13
Standard Deviation		2.09E+14	3.94E+13
Number of Samples		4	4

Table 8.81: Electrical Resistivity Results for 1 wt% Cabot Silica1 in Cycloaliphatic Epoxy

Sample Number	Applied Voltage (V)	Through-Plane Surface Electrical Resistivity (Ω /square)	Through-Plane Volume Electrical Resistivity (Ω -cm)
I310-Si-1-2-18-16-1	100	3.22E+15	8.33E+14
I310-Si-1-2-18-16-2	100	3.35E+15	2.00E+15
I310-Si-1-2-18-16-3	100	2.40E+15	8.62E+14
I310-Si-1-2-18-16-4	100	4.46E+15	2.85E+15
Average		3.36E+15	1.64E+15
Standard Deviation		8.45E+14	9.76E+14
Number of Samples		4	4

Table 8.82: Electrical Resistivity Results for 2 wt% Cabot Silica1 in Cycloaliphatic Epoxy

Sample Number	Applied Voltage (V)	Through-Plane Surface Electrical Resistivity (Ω /square)	Through-Plane Volume Electrical Resistivity (Ω -cm)
I310-Si-2-2-16-16-1	100	2.96E+15	4.88E+14
I310-Si-2-2-16-16-2	100	4.89E+15	5.95E+14
I310-Si-2-2-16-16-3	100	4.39E+15	7.55E+14
Average		4.08E+15	6.13E+14
Standard Deviation		1.00E+15	1.34E+14
Number of Samples		3	3

Table 8.83: Electrical Resistivity Results for 3 wt% Cabot Silica1 in Cycloaliphatic Epoxy

Sample Number	Applied Voltage (V)	Through-Plane Surface Electrical Resistivity (Ω /square)	Through-Plane Volume Electrical Resistivity (Ω -cm)
I310-Si-3-2-23-16-1	100	1.04E+16	1.60E+15
I310-Si-3-2-23-16-2	100	4.69E+15	4.11E+14
I310-Si-3-2-23-16-3	100	5.80E+15	6.77E+14
I310-Si-3-2-23-16-4	100	5.22E+15	5.93E+14
I310-Si-3-2-23-16-5	100	8.17E+15	1.22E+15
Average		6.85E+15	9.00E+14
Standard Deviation		2.37E+15	4.94E+14
Number of Samples		5	5

Table 8.84: Electrical Resistivity Results for 4 wt% Cabot Silica1 in Cycloaliphatic Epoxy

Sample Number	Applied Voltage (V)	Through-Plane Surface Electrical Resistivity (Ω /square)	Through-Plane Volume Electrical Resistivity (Ω -cm)
I310-Si-4-2-29-16-1	100	4.90E+15	5.88E+14
I310-Si-4-2-29-16-2	100	7.17E+15	1.24E+15
I310-Si-4-2-29-16-3	100	5.27E+15	7.94E+14
I310-Si-4-2-29-16-4	100	7.12E+15	8.12E+14
I310-Si-4-2-29-16-5	100	4.52E+15	4.48E+14
Average		5.80E+15	7.76E+14
Standard Deviation		1.26E+15	3.00E+14
Number of Samples		5	5

8.6 Carbon Black and Fumed Silica Thermal Conductivity Data

Table 8.85: Thermal Conductivity Results for Neat Cycloaliphatic Epoxy

Sample Number	Through Plane Thermal Conductivity (W/m•K)
I310-12-8-15-1	0.1483
I310-12-8-15-2	0.1528
I310-12-8-15-3	0.1490
I310-12-8-15-4	0.1482
Average	0.1496
Standard Deviation	0.0022
Number of Samples	4

Table 8.86: Thermal Conductivity Results for 1 wt% Cabot Carbon Black 2 in Cycloaliphatic Epoxy

Sample Number	Through Plane Thermal Conductivity (W/m•K)
I310-X-1-4-27-15-1	0.1620
I310-X-1-4-27-15-2	0.1626
I310-X-1-4-27-15-3	0.1597
I310-X-1-4-27-15-4	0.1577
I310-X-1-4-27-15-5	0.1582
Average	0.1600
Standard Deviation	0.0022
Number of Samples	5

Table 8.87: Thermal Conductivity Results for 1 wt% Cabot Carbon Black 3 in Cycloaliphatic

Epoxy

Sample Number	Through Plane Thermal Conductivity (W/m•K)
I310-Y-1-4-2-15-1	0.1543
I310-Y-1-4-2-15-2	0.1556
I310-Y-1-4-2-15-3	0.1543
I310-Y-1-4-2-15-4	0.1563
I310-Y-1-4-2-15-5	0.1550
Average	0.1551
Standard Deviation	0.0009
Number of Samples	5

Table 8.88: Thermal Conductivity Results for 0.5 wt% Cabot Carbon Black 1 in Cycloaliphatic

Epoxy

Sample Number	Through Plane Thermal Conductivity (W/m•K)
I310-W-1-10-1-15-1	0.1508
I310-W-1-10-1-15-2	0.1556
I310-W-1-10-1-15-3	0.1536
I310-W-1-10-1-15-4	0.1551
Average	0.1538
Standard Deviation	0.0022
Number of Samples	4

Table 8.89: Thermal Conductivity Results for 1 wt% Cabot Carbon Black 1 in Cycloaliphatic

Epoxy

Sample Number	Through Plane Thermal Conductivity (W/m•K)
I310-W-1-10-22-15-1	0.1507
I310-W-1-10-22-15-2	0.1535
I310-W-1-10-22-15-3	0.1550
I310-W-1-10-22-15-4	0.1525
Average	0.1529
Standard Deviation	0.0018
Number of Samples	4

Table 8.90: Thermal Conductivity Results for 2 wt% Cabot Carbon Black 1 in Cycloaliphatic

Epoxy

Sample Number	Through Plane Thermal Conductivity (W/m•K)
I310-W-2-10-29-15-1	0.1483
I310-W-2-10-29-15-2	0.1518
I310-W-2-10-29-15-3	0.1513
I310-W-2-10-29-15-4	0.1553
Average	0.1517
Standard Deviation	0.0029
Number of Samples	4

Table 8.91: Thermal Conductivity Results for 1 wt% Silica1 in Cycloaliphatic Epoxy

Sample Number	Through Plane Thermal Conductivity (W/m•K)
I310-Si-1-2-18-16-1	0.1523
I310-Si-1-2-18-16-2	0.1514
I310-Si-1-2-18-16-3	0.1538
I310-Si-1-2-18-16-4	0.1561
Average	0.1534
Standard Deviation	0.0020
Number of Samples	4

Table 8.92: Thermal Conductivity Results for 2 wt% Silica1 in Cycloaliphatic Epoxy

Sample Number	Through Plane Thermal Conductivity (W/m•K)
I310-Si-2-2-16-16-1	0.1586
I310-Si-2-2-16-16-2	0.1583
I310-Si-2-2-16-16-3	0.1584
Average	0.1584
Standard Deviation	0.0001
Number of Samples	3

Table 8.93: Thermal Conductivity Results for 3 wt% Silica1 in Cycloaliphatic Epoxy

Sample Number	Through Plane Thermal Conductivity (W/m•K)
I310-Si-3-2-23-16-1	0.1583
I310-Si-3-2-23-16-2	0.1592
I310-Si-3-2-23-16-3	0.1580
I310-Si-3-2-23-16-4	0.1602
I310-Si-3-2-23-16-5	0.1611
Average	0.1594
Standard Deviation	0.0013
Number of Samples	5

Table 8.94: Thermal Conductivity Results for 4 wt% Silica1 in Cycloaliphatic Epoxy

Sample Number	Through Plane Thermal Conductivity (W/m•K)
I310-Si-4-2-29-16-1	0.1630
I310-Si-4-2-29-16-2	0.1643
I310-Si-4-2-29-16-3	0.1593
I310-Si-4-2-29-16-4	0.1610
I310-Si-4-2-29-16-5	0.1593
Average	0.1614
Standard Deviation	0.0022
Number of Samples	5

8.7 Carbon Black and Fumed Silica Tensile Data

Table 8.95 Tensile Results Neat Cycloaliphatic Epoxy

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (MPa)
2	49.397	2.983	49.397	2.983	2.072
3	47.495	2.740	47.495	2.740	2.108
4	56.631	3.843	56.631	3.843	2.122
8	52.889	3.411	52.889	3.411	2.047
9	53.356	3.476	53.356	3.476	2.006
10	57.179	3.980	57.179	3.980	2.014
Average	52.82	3.41	52.82	3.41	2.06
Std Dev	3.84	0.48	3.84	0.48	0.05
Number	6	6	6	6	6

Figure 8.24 Tensile Results 1 wt% Cabot Carbon Black 2 in Cycloaliphatic Epoxy

Table 8.96 Tensile Results 1 wt% Cabot Carbon Black 2 in Cycloaliphatic Epoxy

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
4	48.628	3.613	48.628	3.613	2.068
6	51.777	4.483	51.777	4.483	1.851
7	51.605	4.604	51.605	4.604	2.020
9	50.946	3.988	50.946	3.988	2.130
10	51.947	3.856	51.947	3.856	2.140
12	48.444	3.492	48.444	3.492	2.230
14	49.639	4.035	49.639	4.035	2.068
Average	50.43	4.01	50.43	4.01	2.07
Std Dev	1.50	0.41	1.50	0.41	0.12
Count	7	7	7	7	7

Figure 8.25 Tensile Results 1 wt% Cabot Carbon Black 3 in Cycloaliphatic Epoxy

Table 8.97 Tensile Results 1 wt% Cabot Carbon Black 3 in Cycloaliphatic Epoxy

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
2	49.807	3.929	49.807	3.929	1.936
3	48.098	3.330	48.098	3.330	2.084
6	44.669	3.076	44.669	3.076	1.912
7	48.863	3.510	48.863	3.510	1.950
9	45.812	2.951	45.812	2.951	2.126
11	46.110	3.195	46.110	3.195	1.985
14	44.669	3.076	44.669	3.076	1.912
15	46.705	3.121	46.705	3.121	2.068
Average	46.84	3.27	46.84	3.27	2.00
Std Dev	1.91	0.32	1.91	0.32	0.08
Count	8	8	8	8	8

Figure 8.26 Tensile Results 0.5 wt% Cabot Carbon Black 1 in Cycloaliphatic Epoxy

Table 8.98 Tensile Results 0.5 wt% Cabot Carbon Black 1 in Cycloaliphatic Epoxy

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
7	52.641	4.533	52.641	4.533	1.897
8	51.421	4.396	51.421	4.396	1.862
10	49.246	3.264	49.246	3.264	1.984
11	52.697	4.427	52.697	4.427	1.881
15	43.898	2.850	43.898	2.850	1.909
18	48.224	3.296	48.224	3.296	2.023
19	51.431	4.021	51.431	4.021	1.905
Average	49.94	3.83	49.94	3.83	1.92
Std Dev	3.14	0.68	3.14	0.68	0.06
Count	7	7	7	7	7

Figure 8.27 Tensile Results 1 wt% Cabot Carbon Black 1 in Cycloaliphatic Epoxy

Table 8.99 Tensile Results 1 wt% Cabot Carbon Black 1 in Cycloaliphatic Epoxy

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
1	50.998	3.778	50.998	3.778	2.014
5	54.827	4.095	54.827	4.095	2.126
8	48.206	3.450	48.206	3.450	1.894
9	48.845	3.283	48.845	3.283	2.006
12	55.417	4.284	55.417	4.284	2.079
14	51.879	3.423	51.879	3.423	2.189
23	54.872	4.453	54.872	4.453	2.054
Average	52.15	3.82	52.15	3.82	2.05
Std Dev	2.98	0.46	2.98	0.46	0.09
Count	7	7	7	7	7

Figure 8.28 Tensile Results 2 wt% Cabot Carbon Black 1 in Cycloaliphatic Epoxy

Table 8.100 Tensile Results 2 wt% Cabot Carbon Black 1 in Cycloaliphatic Epoxy

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
3	52.318	3.538	52.318	3.538	2.082
5	52.204	3.558	52.204	3.558	2.081
9	47.575	2.993	47.575	2.993	2.096
11	47.358	2.844	47.358	2.844	2.179
15	51.613	3.430	51.613	3.430	2.086
19	48.411	3.113	48.411	3.113	1.999
10	50.828	3.307	50.828	3.307	2.084
25	53.056	3.948	53.056	3.948	1.992
Average	50.42	3.34	50.42	3.34	2.07
Std Dev	2.29	0.36	2.29	0.36	0.06
Count	8	8	8	8	8

Figure 8.29 Tensile Results 1 wt% Silica1 in Cycloaliphatic Epoxy

Table 8.101 Tensile Results 1 wt% Silica1 in Cycloaliphatic Epoxy

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
9	45.829	2.900	45.829	2.900	2.038
10	46.985	3.045	46.985	3.045	2.027
13	46.078	2.920	46.078	2.920	2.021
14	46.238	3.102	46.238	3.102	1.912
17	45.031	2.872	45.031	2.872	1.977
19	45.460	2.919	45.460	2.919	1.993
21	48.084	3.477	48.084	3.477	1.807
23	46.279	3.154	46.279	3.154	1.903
24	44.471	2.801	44.471	2.801	1.991
Average	46.05	3.02	46.05	3.02	1.96
Std Dev	1.06	0.21	1.06	0.21	0.08
Count	9	9	9	9	9

Figure 8.30 Tensile Results 2 wt% Silica1 in Cycloaliphatic Epoxy

Table 8.102 Tensile Results 2 wt% Silica1 in Cycloaliphatic Epoxy

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
4	55.094	4.104	55.094	4.104	2.078
5	47.029	3.138	47.029	3.138	1.885
6	50.595	3.783	50.595	3.783	1.816
7	46.367	2.985	46.367	2.985	1.931
9	52.932	4.137	52.932	4.137	1.915
11	52.104	4.335	52.104	4.335	1.887
13	47.143	3.238	47.143	3.238	1.895
14	49.996	3.345	49.996	3.345	1.976
Average	50.16	3.63	50.16	3.63	1.92
Std Dev	3.15	0.52	3.15	0.52	0.08
Count	8	8	8	8	8

Figure 8.31 Tensile Results 3 wt% Silica1 in Cycloaliphatic Epoxy

Table 8.103 Tensile Results 3 wt% Silica1 in Cycloaliphatic Epoxy

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
4	53.580	4.214	53.580	4.214	1.919
5	56.469	5.237	56.469	5.237	1.842
6	55.252	4.521	55.252	4.521	2.039
7	55.493	3.995	55.493	3.995	2.219
8	54.085	4.237	54.085	4.237	1.949
19	55.296	4.460	55.296	4.460	1.962
21	56.387	4.947	56.387	4.947	1.947
20	56.099	5.151	56.099	5.151	1.897
Average	55.33	4.60	55.33	4.60	1.97
Std Dev	1.05	0.46	1.05	0.46	0.11
Count	8	8	8	8	8

Figure 8.32 Tensile Results 4 wt% Silica1 in Cycloaliphatic Epoxy

Table 8.104 Tensile Results 4 wt% Silica1 in Cycloaliphatic Epoxy

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
2	51.160	3.548	51.160	3.548	2.041
6	55.250	3.993	55.250	3.993	2.180
7	50.839	3.215	50.839	3.215	2.212
8	50.427	3.339	50.427	3.339	2.084
9	55.128	4.334	55.128	4.334	2.064
10	54.440	4.162	54.440	4.162	2.026
11	55.427	4.568	55.427	4.568	2.006
13	55.373	4.154	55.373	4.154	2.131
14	52.201	3.866	52.201	3.866	1.917
19	51.979	3.190	51.979	3.190	2.329
20	53.248	3.882	53.248	3.882	2.029
Average	53.22	3.84	53.22	3.84	2.09
Std Dev	1.98	0.46	1.98	0.46	0.11
Count	11	11	11	11	11

8.8 Carbon Black and Fumed Silica DMA Data

Table 8.105: DMA results for Neat Cycloaliphatic Epoxy made 12-4-15, tested 5-2-16 and 5-3-

16

Run	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	198.75	219.35	233.74
2	200.38	218.92	233.16
3	198.87	218.93	233.17
4	198.18	219.07	233.46
Average	199.05	219.07	233.38
Std Dev	0.94	0.20	0.28

Table 8.106: DMA results for 0.5 wt% Carbon Black 1 in Cycloaliphatic Epoxy made 10-1-15,

tested 12-15-15

Run	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	205.47	221.14	236.59
3	204.28	222.16	235.51
Average	204.88	221.65	236.05
Std Dev	0.84	0.72	0.76

***Table 8.107: DMA results for 1 wt% Carbon Black 1 in Cycloaliphatic Epoxy made 10-22-15,
tested 12-11-15***

Run	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	199.00	222.01	236.56
2	199.11	221.95	236.34
3	198.56	223.13	235.88
Average	198.89	222.36	236.26
Std Dev	0.29	0.66	0.35

***Table 8.108: DMA results for 2 wt% Carbon Black 1 in Cycloaliphatic Epoxy made 10-29-15,
tested 12-14-15***

Run	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	201.52	217.31	232.15
2	201.61	217.88	232.57
3	199.24	217.53	232.38
Average	200.79	217.57	232.37
Std Dev	1.34	0.29	0.21

Figure 8.33: Loss Modulus for Carbon Black 1 in Cycloaliphatic Epoxy

Figure 8.34: Storage Modulus for Carbon Black 1 in Cycloaliphatic Epoxy

Figure 8.35: Tan Delta for Carbon Black 1 in Cycloaliphatic Epoxy

Table 8.109: DMA results for 1 wt% Silica1 in Cycloaliphatic Epoxy made 3-15-16, tested 4-5-16

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	199.10	219.81	234.05
2	189.44	217.68	234.18
3	200.74	219.85	234.24
Average	196.43	219.11	234.16
Std Dev	6.11	1.24	0.10

Table 8.110: DMA results for 2 wt% Silica1 in Cycloaliphatic Epoxy made 2-16-16, tested 4-19-16

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	199.20	221.10	235.49
2	198.27	220.42	235.11
3	185.88	219.49	234.33
Average	194.45	220.34	234.98
Std Dev	7.44	0.81	0.59

Table 8.111: DMA results for 3 wt% Silica1 in Cycloaliphatic Epoxy made 2-23-16, tested 3-31-16

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	200.01	219.87	235.17
2	196.61	219.52	234.36
3	198.59	219.37	234.52
Average	198.40	219.59	234.68
Std Dev	1.71	0.26	0.43

Table 8.112: DMA results for 4 wt% Silica1 in Cycloaliphatic Epoxy made 02-29-16, tested 4-12-16

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	195.94	219.94	236.89
2	196.47	220.82	236.71
3	195.47	220.94	236.53
Average	195.96	220.57	236.71
Std Dev	0.50	0.55	0.18

Figure 8.36: Loss Modulus for Silica1 in Cycloaliphatic Epoxy

Figure 8.37: Storage Modulus for Silica1 in Cycloaliphatic Epoxy

Figure 8.38: Tan Delta for Silica 1 in Cycloaliphatic Epoxy

8.9 Talc and Glass Microspheres Electrical Resistivity Data

Table 8.113: Electrical Resistivity Results for Neat Cycloaliphatic Epoxy

Sample Number	Applied Voltage (V)	Through-Plane Volume Electrical Resistivity (Ω /square)	Through-Plane Surface Electrical Resistivity (Ω -cm)
I022-TT-138-2-16-17-1	100	3.60E+16	2.19E+15
I022-TT-138-2-16-17-2	100	3.645E+16	4.03E+15
I022-TT-138-2-16-17-3	100	3.77E+16	3.45E+15
I022-TT-138-2-16-17-4	100	3.74E+16	2.42E+15
I022-TT-138-2-16-17-5	100	3.51E+16	1.53E+15
Average		3.65E+16	2.72E+15
Standard Deviation		1.05E+15	1.00E+15
Number of Samples		5	5

Table 8.114: Electrical Resistivity Results for 5 wt% Talc in Cycloaliphatic Epoxy

Sample Number	Applied Voltage (V)	Through-Plane Volume Electrical Resistivity (Ω /square)
I022-TT-N-5-2-10-17-1	100	3.46E+16
I022-TT-N-5-2-10-17-2	100	3.69E+16
I022-TT-N-5-2-10-17-3	100	3.46E+16
I022-TT-N-5-2-10-17-4	100	3.66E+16
I022-TT-N-5-2-10-17-5	100	3.39E+16
Average		3.53E+16
Standard Deviation		1.43E+15
Number of Samples		5

Table 8.115: Electrical Resistivity Results for 10 wt% Talc in Cycloaliphatic Epoxy

Sample Number	Applied Voltage (V)	Through-Plane Volume Electrical Resistivity (Ω/square)	Through-Plane Surface Electrical Resistivity ($\Omega\text{-cm}$)
I022-TT-N-10-1-26-17-1	100	2.21E+16	3.45E+16
I022-TT-N-10-1-26-17-2	100	2.17E+16	1.32E+17
I022-TT-N-10-1-26-17-3	100	2.16E+16	1.02E+17
I022-TT-N-10-1-26-17-3	100	1.93E+16	1.63E+17
Average		2.12E+16	1.08E+17
Standard Deviation		1.26E+15	5.48E+16
Number of Samples		4	4

Table 8.116: Electrical Resistivity Results for 15 wt% Talc in Cycloaliphatic Epoxy

Sample Number	Applied Voltage (V)	Through-Plane Volume Electrical Resistivity (Ω/square)	Through-Plane Surface Electrical Resistivity ($\Omega\text{-cm}$)
I022-TT-N-15-1-24-17-1	100	1.71E+16	4.34E+16
I022-TT-N-15-1-24-17-2	100	2.06E+16	6.41E+16
I022-TT-N-15-1-24-17-3	100	2.03E+16	5.85E+16
I022-TT-N-15-1-24-17-4	100	1.87E+16	4.73E+16
I022-TT-N-15-1-24-17-5	100	1.68E+16	4.32E+16
Average		1.87E+16	5.13E+16
Standard Deviation		1.74E+15	9.48E+15
Number of Samples		5	5

Table 8.117: Electrical Resistivity Results for 20 wt% Talc in Cycloaliphatic Epoxy

Sample Number	Applied Voltage (V)	Through-Plane Volume Electrical Resistivity (Ω /square)
I022-TT-N-20-1-31-17-1	100	1.87E+16
I022-TT-N-20-1-31-17-2	100	2.02E+16
I022-TT-N-20-1-31-17-3	100	2.02E+16
I022-TT-N-20-1-31-17-4	100	2.02E+16
I022-TT-N-20-1-31-17-5	100	1.78E+16
Average		1.94E+16
Standard Deviation		1.11E+15
Number of Samples		5

Table 8.118: Electrical Resistivity Results for 5 wt% Glass Microspheres in Cycloaliphatic Epoxy

Sample Number	Applied Voltage (V)	Through-Plane Volume Electrical Resistivity (Ω /square)	Through-Plane Surface Electrical Resistivity (Ω -cm)
I022-TT-SG-5-11-15-16-1	100	9.99E+15	1.65E+16
I022-TT-SG-5-11-15-16-2	100	9.99E+15	1.09E+16
I022-TT-SG-5-11-15-16-3	100	1.11E+16	1.07E+16
I022-TT-SG-5-11-15-16-4	100	1.07E+16	1.16E+16
I022-TT-SG-5-11-15-16-5	100	1.06E+16	1.16E+16
Average		1.05E+16	1.23E+16
Standard Deviation		4.88E+14	2.41E+15
Number of Samples		5	5

Table 8.119: Electrical Resistivity Results for 10 wt% Glass Microspheres in Cycloaliphatic

Epoxy

Sample Number	Applied Voltage (V)	Through-Plane Volume Electrical Resistivity (Ω /square)	Through-Plane Surface Electrical Resistivity (Ω -cm)
I022-TT-SG-10-11-3-16-1	100	4.79E+15	7.93E+15
I022-TT-SG-10-11-3-16-2	100	5.16E+15	6.33E+15
I022-TT-SG-10-11-3-16-3	100	4.99E+15	7.00E+15
I022-TT-SG-10-11-3-16-4	100	5.05E+15	6.80E+16
I022-TT-SG-10-11-3-16-5	100	4.44E+15	6.91E+15
Average		4.89E+15	6.99E+15
Standard Deviation		2.84E+14	5.83E+14
Number of Samples		5	5

Table 8.120: Electrical Resistivity Results for 20 wt% Glass Microspheres in Cycloaliphatic

Epoxy

Sample Number	Applied Voltage (V)	Through-Plane Volume Electrical Resistivity (Ω /square)	Through-Plane Surface Electrical Resistivity (Ω -cm)
I022-TT-SG-20-9-16-16-1	100	1.68E+15	5.48E+15
I022-TT-SG-20-9-16-16-2	100	1.73E+15	5.41E+15
I022-TT-SG-20-9-16-16-3	100	1.69E+15	6.57E+15
I022-TT-SG-20-9-16-16-4	100	1.72E+15	5.96E+15
I022-TT-SG-20-9-16-16-5	100	1.90E+15	6.05E+15
Average		1.74E+15	5.89E+15
Standard Deviation		8.86E+13	4.69E+14
Number of Samples		5	5

Table 8.121: Electrical Resistivity Results for 30 wt% Glass Microspheres in Cycloaliphatic

Epoxy

Sample Number	Applied Voltage (V)	Through-Plane Volume Electrical Resistivity (Ω/square)	Through-Plane Surface Electrical Resistivity ($\Omega\text{-cm}$)
I022-TT-SG-30-10-27-16-1	100	1.37E+15	7.67E+15
I022-TT-SG-30-10-27-16-2	100	1.26E+15	6.26E+15
I022-TT-SG-30-10-27-16-3	100	1.12E+15	5.30E+15
I022-TT-SG-30-10-27-16-4	100	1.18E+15	6.18E+15
I022-TT-SG-30-10-27-16-5	100	1.21E+15	6.41E+15
Average		1.23E+15	6.36E+15
Standard Deviation		9.42E+13	8.47E+14
Number of Samples		5	5

Table 8.122: Electrical Resistivity Results for 40 wt% Glass Microspheres in Cycloaliphatic

Epoxy

Sample Number	Applied Voltage (V)	Through-Plane Volume Electrical Resistivity (Ω/square)	Through-Plane Surface Electrical Resistivity ($\Omega\text{-cm}$)
I022-TT-SG-40-10-3-16-1	100	5.56E+14	4.44E+15
I022-TT-SG-40-10-3-16-2	100	5.68E+14	3.47E+15
I022-TT-SG-40-10-3-16-3	100	5.81E+14	4.32E+15
I022-TT-SG-40-10-3-16-4	100	5.63E+14	4.48E+15
I022-TT-SG-40-10-3-16-5	100	6.26E+14	3.53E+15
Average		5.79E+14	4.05E+15
Standard Deviation		2.79E+13	5.03E+14
Number of Samples		5	5

8.10 Talc and Glass Microspheres Thermal Conductivity Data

Table 8.123: Thermal Conductivity Results for Neat Cycloaliphatic Epoxy

Sample Number	Through Plane Thermal Conductivity (W/m•K)
I022-TT-138-7-11-16-1	0.1480
I022-TT-138-7-11-16-2	0.1499
I022-TT-138-7-11-16-3	0.1502
I022-TT-138-7-11-16-4	0.1466
I022-TT-138-7-11-16-5	0.1494
I022-TT-138-7-11-16-6	0.1481
Average	0.1486
Standard Deviation	0.0013
Number of Samples	6

Table 8.124: Thermal Conductivity Results for 5 wt% Talc in Cycloaliphatic Epoxy

Sample Number	Through Plane Thermal Conductivity (W/m•K)
I022-TT-N-5-7-18-16-1	0.1682
I022-TT-N-5-7-18-16-2	0.1689
I022-TT-N-5-7-18-16-3	0.1675
I022-TT-N-5-7-18-16-4	0.1663
I022-TT-N-5-7-18-16-5	0.1673
Average	0.1676
Standard Deviation	0.0010
Number of Samples	5

Table 8.125: Thermal Conductivity Results for 10 wt% Talc in Cycloaliphatic Epoxy

Sample Number	Through Plane Thermal Conductivity (W/m•K)
I022-TT-N-10-6-15-16-1	0.1877
I022-TT-N-10-6-15-16-2	0.1889
I022-TT-N-10-6-15-16-3	0.1876
I022-TT-N-10-6-15-16-4	0.1871
I022-TT-N-10-6-15-16-5	0.1862
Average	0.1875
Standard Deviation	0.0010
Number of Samples	5

Table 8.126: Thermal Conductivity Results for 15 wt% Talc in Cycloaliphatic Epoxy

Sample Number	Through Plane Thermal Conductivity (W/m•K)
I022-TT-N15-7-19-16-1	0.2173
I022-TT-N-15-7-19-16-2	0.2235
I022-TT-N-15-7-19-16-3	0.2197
I022-TT-N-15-7-19-16-4	0.2249
I022-TT-N-15-7-19-16-5	0.2255
Average	0.2222
Standard Deviation	0.0035
Number of Samples	5

Table 8.127: Thermal Conductivity Results for 20 wt% Talc in Cycloaliphatic Epoxy

Sample Number	Through Plane Thermal Conductivity (W/m•K)
I022-TT-N-20-6-27-16-1	0.2547
I022-TT-N-20-6-27-16-2	0.2559
I022-TT-N-20-6-27-16-3	0.2549
I022-TT-N-20-6-27-16-4	0.2574
I022-TT-N-20-6-27-16-5	0.2548
I022-TT-N-20-6-27-16-6	0.2539
Average	0.2553
Standard Deviation	0.0012
Number of Samples	6

Table 8.128: Thermal Conductivity Results for 5 wt% Glass Microspheres in Cycloaliphatic Epoxy

Sample Number	Through Plane Thermal Conductivity (W/m•K)
I022-TT-SG-5-11-15-16-1	0.1484
I022-TT-SG-5-11-15-16-2	0.1497
I022-TT-SG-5-11-15-16-3	0.1471
I022-TT-SG-5-11-15-16-4	0.1475
I022-TT-SG-5-11-15-16-5	0.1478
Average	0.1481
Standard Deviation	0.0010
Number of Samples	5

Table 8.129: Thermal Conductivity Results for 10 wt% Glass Microspheres in Cycloaliphatic Epoxy

Sample Number	Through Plane Thermal Conductivity (W/m•K)
I022-TT-SG-10-11-3-16-1	0.1601
I022-TT-SG-10-11-3-16-2	0.1594
I022-TT-SG-10-11-3-16-3	0.1602
I022-TT-SG-10-11-3-16-4	0.1598
I022-TT-SG-10-11-3-16-5	0.1590
Average	0.1597
Standard Deviation	0.0005
Number of Samples	5

Table 8.130: Thermal Conductivity Results for 20 wt% Glass Microspheres in Cycloaliphatic Epoxy

Sample Number	Through Plane Thermal Conductivity (W/m•K)
I022-TT-SG-20-9-16-16-1	0.1822
I022-TT-SG-20-9-16-16-2	0.1841
I022-TT-SG-20-9-16-16-3	0.1880
I022-TT-SG-20-9-16-16-4	0.1837
I022-TT-SG-20-9-16-16-5	0.1846
Average	0.1845
Standard Deviation	0.0022
Number of Samples	5

Table 8.131: Thermal Conductivity Results for 30 wt% Glass Microspheres in Cycloaliphatic Epoxy

Sample Number	Through Plane Thermal Conductivity (W/m•K)
I022-TT-SG-30-10-27-16-1	0.1991
I022-TT-SG-30-10-27-16-2	0.2001
I022-TT-SG-30-10-27-16-3	0.2042
I022-TT-SG-30-10-27-16-4	0.2082
I022-TT-SG-30-10-27-16-5	0.2083
Average	0.2040
Standard Deviation	0.0043
Number of Samples	5

Table 8.132: Thermal Conductivity Results for 40 wt% Glass Microspheres in Cycloaliphatic Epoxy

Sample Number	Through Plane Thermal Conductivity (W/m•K)
I022-TT-SG-40-10-3-16-1	0.2394
I022-TT-SG-40-10-3-16-2	0.2498
I022-TT-SG-40-10-3-16-3	0.2396
I022-TT-SG-40-10-3-16-4	0.2390
I022-TT-SG-40-10-3-16-5	0.2354
Average	0.2406
Standard Deviation	0.0054
Number of Samples	5

8.11 Talc and Glass Microspheres Tensile Data

Figure 8.39: Tensile Results for Neat Cycloaliphatic Epoxy

Table 8.133: Tensile Results for Neat Cycloaliphatic Epoxy

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
1	43.343	1.764	43.343	1.764	2.549
5	44.424	1.624	44.424	1.624	2.841
6	53.306	2.130	53.306	2.130	2.665
10	43.420	1.569	43.420	1.569	2.836
11	50.035	1.861	50.035	1.861	2.859
14	43.781	1.713	43.781	1.713	2.645
15	52.103	2.152	52.103	2.152	2.576
4	47.206	1.760	47.206	1.760	2.840
Average	47.20	1.82	47.20	1.82	2.73
Std Dev	4.10	0.22	4.10	0.22	0.13
Count	8	8	8	8	8

Figure 8.40: Tensile Results for 5 wt% Talc in Cycloaliphatic Epoxy

Table 8.134: Tensile Results for 5 wt% Talc in Cycloaliphatic Epoxy

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
1	55.443	2.105	55.443	2.105	2.980
2	54.135	1.997	54.135	1.997	3.037
3	54.330	2.132	54.330	2.132	2.809
5	58.668	2.327	58.668	2.327	2.863
12	56.853	2.159	56.853	2.159	2.992
14	51.508	1.924	51.508	1.924	2.919
19	53.364	2.115	53.364	2.115	2.799
Average	54.90	2.11	54.90	2.11	2.91
Std Dev	2.35	0.13	2.35	0.13	0.09
Count	7	7	7	7	7

Figure 8.41: Tensile Results for 10 wt% Talc in Cycloaliphatic Epoxy

Table 8.135: Tensile Results for 10 wt% Talc in Cycloaliphatic Epoxy

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
4	53.799	1.892	53.799	1.892	3.267
5	53.374	2.031	53.374	2.031	2.997
7	46.051	1.617	46.051	1.617	3.091
8	50.580	1.805	50.580	1.805	3.153
9	52.887	1.876	52.887	1.876	3.246
13	50.245	1.791	50.245	1.791	3.144
16	45.216	1.551	45.216	1.551	3.175
Average	50.31	1.79	50.31	1.79	3.15
Std Dev	3.47	0.16	3.47	0.16	0.09
Count	7	7	7	7	7

Figure 8.42: Tensile Results for 15 wt% Talc in Cycloaliphatic Epoxy

Table 8.136: Tensile Results for 15 wt% Talc in Cycloaliphatic Epoxy

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
1	52.817	1.909	52.817	1.909	3.2382
3	49.351	1.748	49.351	1.748	3.2441
5	47.047	1.551	47.047	1.551	3.3876
6	54.152	2.056	54.152	2.056	3.1491
8	53.692	1.995	53.692	1.995	3.1558
9	49.363	1.744	49.363	1.744	3.22
11	52.579	1.886	52.579	1.886	3.3235
12	49.85	1.726	49.85	1.726	3.3145
15	48.753	1.691	48.753	1.691	3.2461
Average	50.85	1.81	50.85	1.81	3.25
Std Dev	2.50	0.16	2.50	0.16	0.08
Count	9	9	9	9	9

Figure 8.43: Tensile Results for 20 wt% Talc in Cycloaliphatic Epoxy

Table 8.137: Tensile Results for 20 wt% Talc in Cycloaliphatic Epoxy

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
1	53.606	1.784	53.606	1.784	3.548
3	52.218	1.639	52.218	1.639	3.715
5	48.786	1.659	48.786	1.659	3.332
6	48.409	1.530	48.409	1.530	3.603
22	49.384	1.634	49.384	1.634	3.451
24	53.754	1.808	53.754	1.808	3.627
26	51.208	1.584	51.208	1.584	3.766
28	48.741	1.489	48.741	1.489	3.674
Average	50.76	1.64	50.76	1.64	3.59
Std Dev	2.23	0.11	2.23	0.11	0.14
Count	8	8	8	8	8

Figure 8.44: Tensile Results for 5 wt% Glass Microspheres in Cycloaliphatic Epoxy

Table 8.138: Tensile Results for 5 wt% Glass Microspheres in Cycloaliphatic Epoxy

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
1	39.408	1.381	39.408	1.381	2.937
2	35.823	1.282	35.823	1.282	2.835
8	38.650	1.397	38.650	1.397	2.834
13	43.466	1.570	43.466	1.570	2.895
17	37.901	1.386	37.901	1.386	2.752
22	39.023	1.355	39.023	1.355	2.967
27	40.732	1.545	40.732	1.545	2.722
28	35.031	1.229	35.031	1.229	2.886
29	45.470	1.655	45.470	1.655	2.863
Average	39.50	1.42	39.50	1.42	2.85
Std Dev	3.35	0.14	3.35	0.14	0.08
Count	9	9	9	9	9

Figure 8.45: Tensile Results for 10 wt% Glass Microspheres in Cycloaliphatic Epoxy

Table 8.139: Tensile Results for 10 wt% Glass Microspheres in Cycloaliphatic Epoxy

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
2	35.280	1.132	35.280	1.132	3.158
3	42.378	1.590	42.378	1.590	3.082
4	44.087	1.456	44.087	1.456	3.159
6	39.194	1.229	39.194	1.229	3.264
7	44.868	1.384	44.868	1.384	3.422
8	39.360	1.258	39.360	1.258	3.206
9	40.945	1.343	40.945	1.343	3.126
11	42.678	1.350	42.678	1.350	3.301
Average	41.10	1.34	41.10	1.34	3.21
Std Dev	3.11	0.14	3.11	0.14	0.11
Count	8	8	8	8	8

Figure 8.46: Tensile Results for 20 wt% Glass Microspheres in Cycloaliphatic Epoxy

Table 8.140: Tensile Results for 20 wt% Glass Microspheres in Cycloaliphatic Epoxy

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
1	37.266	1.035	37.266	1.035	3.721
2	37.924	1.004	37.924	1.004	3.877
6	41.090	1.084	41.090	1.084	3.971
7	43.225	1.129	43.225	1.129	4.009
8	39.874	1.058	39.874	1.058	3.868
12	41.836	1.201	41.836	1.201	3.589
Average	40.20	1.09	40.20	1.09	3.84
Std Dev	2.30	0.07	2.30	0.07	0.16
Count	6	6	6	6	6

Figure 8.47: Tensile Results for 30 wt% Glass Microspheres in Cycloaliphatic Epoxy

Table 8.141: Tensile Results for 30 wt% Glass Microspheres in Cycloaliphatic Epoxy

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
8	37.245	0.871	37.245	0.871	4.321
9	38.554	0.850	38.554	0.850	4.680
11	37.425	0.819	37.425	0.819	4.724
13	39.320	0.896	39.320	0.896	4.538
18	34.052	0.779	34.052	0.779	4.455
22	37.717	0.861	37.717	0.861	4.489
24	39.140	0.945	39.140	0.945	4.253
Average	37.64	0.86	37.64	0.86	4.49
Std Dev	1.78	0.05	1.78	0.05	0.17
Count	7	7	7	7	7

Figure 8.48: Tensile Results for 40 wt% Glass Microspheres in Cycloaliphatic Epoxy

Table 8.142: Tensile Results for 40 wt% Glass Microspheres in Cycloaliphatic Epoxy

Specimen	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
8	38.824	0.776	38.824	0.776	5.163
16	36.876	0.699	36.876	0.699	5.368
18	42.594	0.885	42.594	0.885	5.125
22	42.046	0.839	42.046	0.839	5.211
23	35.821	0.699	35.821	0.699	5.243
Average	39.23	0.78	39.23	0.78	5.22
Std Dev	3.02	0.08	3.02	0.08	0.09
Count	5	5	5	5	5

8.12 Aging TC307 Water Mass Gain Data

Table 8.143: Water Mass Gain Data for GNP/Epoxy Composites, ± 1 Standard Deviation

Aging Time (hr)	Neat Epoxy Mass gain (%)	2 wt% GNP Mass gain (%)	4 wt% GNP Mass gain (%)
0	0.0 n=4	0.0 n=4	0.0 n=4
2	0.420 $\pm$ 0.129 n=4	0.328 $\pm$ 0.033 n=4	0.320 $\pm$ 0.091 n=4
8	0.902 $\pm$ 0.073 n=4	0.599 $\pm$ 0.060 n=4	0.616 $\pm$ 0.040 n=4
33	1.409 $\pm$ 0.041 n=4	1.138 $\pm$ 0.038 n=4	1.161 $\pm$ 0.100 n=4
50	1.531 $\pm$ 0.077 n=4	1.363 $\pm$ 0.025 n=4	1.383 $\pm$ 0.060 n=4
75	1.685 $\pm$ 0.097 n=3	1.492 $\pm$ 0.073 n=3	1.594 $\pm$ 0.055 n=3
100	1.716 $\pm$ 0.066 n=3	1.629 $\pm$ 0.044 n=3	1.703 $\pm$ 0.055 n=3
122	1.762 $\pm$ 0.057 n=2	1.627 $\pm$ 0.023 n=2	1.743 $\pm$ 0.015 n=2
150	1.802 $\pm$ 0.046 n=2	1.786 $\pm$ 0.031 n=2	1.869 $\pm$ 0.107 n=2
200	1.866 $\pm$ 0.024 n=2	1.931 $\pm$ 0.020 n=2	1.947 $\pm$ 0.020 n=2
240	2.008 n=1	1.993 n=1	2.103 n=1
290	2.024 n=1	2.069 n=1	2.103 n=1
341	2.008 n=1	2.069 n=1	2.134 n=1
384	2.006 n=1	2.099 n=1	2.149 n=1
408	2.008 n=1	2.084 n=1	2.103 n=1

8.13 Aging TC307 Flexural Data

Figure 8.49: Flexural Results for Neat Cycloaliphatic Epoxy Unaged

Table 8.144: Flexural Results for Neat Cycloaliphatic Epoxy Unaged

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
7	75.195	5.057	1.911
9	80.498	5.650	1.938
11	78.802	6.448	1.923
16	77.368	5.295	1.971
Average	77.97	5.61	1.94
Std Dev	2.25	0.61	0.03
Count	4	4	4

Figure 8.50: Flexural Results for Neat Cycloaliphatic Epoxy aged 50 hours

Table 8.145: Flexural Results for Neat Cycloaliphatic Epoxy aged 50 hours

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
20	76.332	6.243	1.919
19	77.456	6.060	1.954
18	75.450	6.085	1.905
17	78.501	6.222	2.003
Average	76.93	6.15	1.95
Std Dev	1.33	0.09	0.04
Count	4	4	4

Figure 8.51: Flexural Results for Neat Cycloaliphatic Epoxy aged 100 hours

Table 8.146: Flexural Results for Neat Cycloaliphatic Epoxy aged 100 hours

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
15	75.984	5.082	1.971
13	77.700	5.572	1.945
12	75.888	5.486	1.877
11	76.195	5.308	1.924
Average	76.44	5.36	1.93
Std Dev	0.85	0.22	0.04
Count	4	4	4

Figure 8.52: Flexural Results for Neat Cycloaliphatic Epoxy aged 200 hours

Table 8.147: Flexural Results for Neat Cycloaliphatic Epoxy aged 200 hours

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
10	75.254	4.995	1.908
9	79.237	6.468	1.887
8	76.283	5.954	1.864
7	76.891	6.361	1.843
6	73.033	4.895	1.873
Average	76.14	5.73	1.87
Std Dev	2.27	0.75	0.02
Count	4	4	4

Figure 8.53: Flexural Results for Neat Cycloaliphatic Epoxy aged 400 hours

Table 8.148: Flexural Results for Neat Cycloaliphatic Epoxy aged 400 hours

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
5	77.399	5.376	1.897
4	77.268	5.659	1.861
3	76.670	5.222	1.879
2	78.030	5.895	1.857
1	77.571	5.472	1.869
Average	77.39	5.52	1.87
Std Dev	0.49	0.26	0.02
Count	4	4	4

Figure 8.54: Flexural Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Unaged

Table 8.149: Flexural Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Unaged

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
4	83.185	5.943	1.974
5	70.842	4.153	2.004
6	85.429	5.799	2.024
8	89.134	6.403	2.035
9	77.382	4.282	2.075
Average	81.19	5.32	2.02
Std Dev	7.19	1.03	0.04
Count	5	5	5

Figure 8.55: Flexural Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 50 hours

Table 8.150: Flexural Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 50 hours

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
20	81.455	6.009	2.018
19	77.672	5.163	1.954
17	68.194	3.691	2.082
16	84.409	5.798	2.075
Average	77.93	5.17	2.03
Std Dev	7.05	1.05	0.06
Count	4	4	4

Figure 8.56: Flexural Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 100 hours

Table 8.151: Flexural Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 100 hours

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
15	80.574	5.715	2.008
14	78.957	5.969	1.915
12	73.814	4.356	1.994
11	82.344	5.602	2.027
Average	78.92	5.41	1.99
Std Dev	3.68	0.72	0.05
Count	4	4	4

Figure 8.57: Flexural Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 200 hours

Table 8.152: Flexural Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 200 hours

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
10	79.342	5.349	1.962
9	78.359	5.217	1.927
6	81.378	5.691	1.938
Average	79.69	5.42	1.94
Std Dev	1.54	0.24	0.02
Count	3	3	3

Figure 8.58: Flexural Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 400 hours

Table 8.153: Flexural Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 400 hours

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
5	66.271	3.843	1.911
4	73.792	4.549	1.920
3	76.593	4.602	1.981
2	79.914	5.727	1.945
1	76.227	5.117	1.940
Average	74.56	4.77	1.94
Std Dev	5.12	0.70	0.03
Count	4	4	4

Figure 8.59: Flexural Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Unaged

Table 8.154: Flexural Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Unaged

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
5	89.176	5.332	2.137
6	81.787	5.663	1.913
11	79.407	5.859	1.831
12	89.349	4.375	2.459
14	83.246	5.154	2.079
Average	84.59	5.28	2.08
Std Dev	4.48	0.57	0.24
Count	5	5	5

Figure 8.60: Flexural Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 50 hours

Table 8.155: Flexural Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Age 50 hour

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
20	79.214	5.019	2.120
18	78.176	5.575	2.087
17	79.164	5.745	2.094
16	76.459	5.148	2.019
Average	78.25	5.37	2.08
Std Dev	1.29	0.34	0.04
Count	4	4	4

Figure 8.61: Flexural Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Age 100 hour

Table 8.156: Flexural Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Age 100 hour

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
15	84.261	4.941	2.257
14	79.104	4.956	2.103
11	78.639	5.163	2.052
Average	80.67	5.02	2.14
Std Dev	3.12	0.12	0.11
Count	3	3	3

Figure 8.62: Flexural Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Age 200 hour

Table 8.157: Flexural Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Age 200 hour

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
10	83.720	5.748	2.007
9	96.197	3.717	2.026
8	78.902	5.698	2.011
7	81.345	5.727	2.022
6	80.185	6.798	2.027
Average	84.07	5.54	2.02
Std Dev	7.01	1.12	0.01
Count	4	4	4

Figure 8.63: Flexural Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Age 400 hour

Table 8.158: Flexural Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Age 400 hour

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
5	75.422	4.513	2.072
4	78.901	5.152	2.057
3	78.218	4.726	2.093
2	80.113	5.536	2.016
1	80.177	5.009	2.109
Average	78.57	4.99	2.07
Std Dev	1.94	0.39	0.04
Count	4	4	4

8.14 Aging TC307 DMA Data

Table 8.159: DMA Results for Neat Cycloaliphatic Epoxy Unaged

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	193.26	217.70	231.50
2	194.15	217.40	231.35
3	197.55	217.54	231.34
Average	194.99	217.55	231.40
Std Dev	2.26	0.15	0.09

Table 8.160: DMA Results for Neat Cycloaliphatic Epoxy Aged 50 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	205.22	204.92	226.21
2	205.80	205.50	226.34
3	205.74	205.44	226.28
Average	205.59	205.29	226.28
Std Dev	0.32	0.32	0.07

Table 8.161: DMA Results for Neat Cycloaliphatic Epoxy Aged 100 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	202.08	201.78	226.97
2	200.80	201.25	227.19
3	201.65	202.85	227.14
Average	201.51	201.96	227.10
Std Dev	0.65	0.82	0.12

Table 8.162: DMA Results for Neat Cycloaliphatic Epoxy Aged 200 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	194.27	193.67	226.03
2	193.19	192.06	225.66
3	193.9	193.45	225.99
Average	193.79	193.06	225.89
Std Dev	0.55	0.87	0.20

Table 8.163: DMA Results for Neat Cycloaliphatic Epoxy Aged 400 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	191.51	190.99	226.38
2	190.98	190.68	227.12
3	190.92	190.55	226.54
Average	191.14	190.74	226.68
Std Dev	0.32	0.23	0.39

Figure 8.64: Storage Modulus for Neat Cycloaliphatic Epoxy

Figure 8.65: Loss Modulus for Neat Cycloaliphatic Epoxy

Figure 8.66: Tan Delta for Neat Cycloaliphatic Epoxy

Table 8.164 DMA Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Unaged

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	203.53	220.26	231.21
2	204.26	220.54	231.79
3	203.37	220.68	231.78
Average	203.72	220.49	231.59
Std Dev	0.47	0.21	0.33

Table 8.165 DMA Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 50 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	211.04	210.82	228.66
2	211.05	211.05	227.99
3	211.61	211.23	228.03
Average	211.23	211.03	228.23
Std Dev	0.33	0.21	0.38

Table 8.166 DMA Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 100 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	193.55	194.07	227.07
2	194.48	194.33	227.32
3	193.43	193.43	227.02
Average	193.82	193.94	227.14
Std Dev	0.57	0.46	0.16

Table 8.167 DMA Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 200 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	200.18	198.76	227.70
2	198.37	197.55	226.94
3	201.27	198.68	227.28
Average	199.94	198.33	227.31
Std Dev	1.46	0.68	0.38

Table 8.168 DMA Results for 2 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 400 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	186.97	188.25	227.25
2	186.63	187.68	227.72
3	185.91	186.14	227.69
Average	186.50	187.36	227.55
Std Dev	0.54	1.09	0.26

Figure 8.67: Storage Modulus for 2 wt% TC307 GNP in Cycloaliphatic Epoxy

Figure 8.68: Loss Modulus for 2 wt% TC307 GNP in Cycloaliphatic Epoxy

Figure 8.69: Tan Delta for 2 wt% TC307 GNP in Cycloaliphatic Epoxy

Table 8.169 DMA Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Unaged

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	209.12	221.56	231.01
2	209.16	221.48	230.76
3	208.8	221.33	230.61
Average	209.01	221.38	230.79
Std Dev	0.17	0.18	0.20

Table 8.170 DMA Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 50 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	200.78	200.48	227.62
2	201.62	201.25	228.54
3	201.51	201.13	228.28
Average	201.30	200.95	228.15
Std Dev	0.46	0.41	0.47

Table 8.171 DMA Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 100 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	198.40	198.18	227.87
2	198.30	198.00	227.84
3	197.00	196.85	227.30
Average	197.90	197.68	227.67
Std Dev	0.78	0.72	0.32

Table 8.172 DMA Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 200 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	194.75	194.60	227.60
2	196.77	195.05	227.74
3	194.42	194.36	226.88
Average	195.31	194.67	227.41
Std Dev	1.27	0.35	0.46

Table 8.173 DMA Results for 4 wt% TC307 GNP in Cycloaliphatic Epoxy Aged 400 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	187.50	187.43	227.93
2	186.68	186.76	227.56
3	203.46	203.15	228.65
Average	187.09	187.10	228.05
Std Dev	0.58	0.47	0.55

Figure 8.70: Storage Modulus for 4 wt% TC307 GNP in Cycloaliphatic Epoxy

Figure 8.71: Loss Modulus for 4 wt% TC307 GNP in Cycloaliphatic Epoxy

Figure 8.72: Tan Delta for 4 wt% TC307 GNP in Cycloaliphatic Epoxy

8.15 Aging Talc Water Mass Gain Data

Table 8.174: Water Mass Gain Data for Talc/Epoxy Composites, ± 1 Standard Deviation

Aging Time (hr)	Neat Epoxy Mass gain (%)	10 wt% Talc Mass gain (%)	20 wt% Talc Mass gain (%)
0	0.0 n=24	0.0 n=24	0.0 n=24
5	0.51 $\pm$ 0.20 n=19	0.32 $\pm$ 0.22 n=16	0.16 $\pm$ 0.07 n=7
11	0.77 $\pm$ 0.14 n=19	0.64 $\pm$ 0.17 n=14	0.23 $\pm$ 0.10 n=11
25	1.03 $\pm$ 0.18 n=11	0.77 $\pm$ 0.29 n=15	0.40 $\pm$ 0.26 n=7
34	1.11 $\pm$ 0.14 n=18	0.84 $\pm$ 0.15 n=9	0.42 $\pm$ 0.17 n=9
52	1.62 $\pm$ 0.31 n=17	1.24 $\pm$ 0.23 n=17	0.63 $\pm$ 0.16 n=12
72	2.02 $\pm$ 0.26 n=21	1.54 $\pm$ 0.26 n=19	1.03 $\pm$ 0.16 n=16
100	2.29 $\pm$ 0.16 n=10	1.81 $\pm$ 0.32 n=13	1.31 $\pm$ 0.23 n=16
127	2.41 $\pm$ 0.27 n=16	2.01 $\pm$ 0.30 n=20	1.51 $\pm$ 0.22 n=16
172	2.70 $\pm$ 0.27 n=22	2.25 $\pm$ 0.18 n=20	1.68 $\pm$ 0.21 n=15
193	2.85 $\pm$ 0.20 n=20	2.49 $\pm$ 0.18 n=20	1.85 $\pm$ 0.23 n=17
220	2.90 $\pm$ 0.22 n=17	2.55 $\pm$ 0.21 n=20	1.86 $\pm$ 0.21 n=16
240	2.97 $\pm$ 0.21 n=20	2.57 $\pm$ 0.18 n=19	1.95 $\pm$ 0.15 n=14
267	2.96 $\pm$ 0.24 n=17	2.56 $\pm$ 0.23 n=17	1.89 $\pm$ 0.23 n=15
314	3.01 $\pm$ 0.27 n=17	2.63 $\pm$ 0.23 n=15	1.89 $\pm$ 0.20 n=17
339	3.07 $\pm$ 0.23 n=20	2.65 $\pm$ 0.16 n=15	2.06 $\pm$ 0.21 n=17
361	3.09 $\pm$ 0.22 n=20	2.66 $\pm$ 0.14 n=19	2.04 $\pm$ 0.17 n=17
386	3.13 $\pm$ 0.22 n=18	2.66 $\pm$ 0.19 n=17	2.07 $\pm$ 0.22 n=16
400	3.20 $\pm$ 0.17 n=12	2.66 $\pm$ 0.14 n=8	2.10 $\pm$ 0.15 n=9

8.16 Aging Talc Flexural Data

Figure 8.73: Flexural Results for Neat Cycloaliphatic Epoxy Unaged

Table 8.175: Flexural Results for Neat Cycloaliphatic Epoxy Unaged

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
14a	117.554	3.852	3.273
15a	117.296	4.036	3.234
19a	120.746	4.365	3.296
21a	118.428	4.040	3.238
22a	105.658	3.650	3.126
23a	107.748	3.656	3.163
24a	115.630	3.964	3.203
Average	114.72	3.94	3.22
Std Dev	5.72	0.25	0.06
Count	7	7	7

Figure 8.74: Flexural Results for Neat Cycloaliphatic Epoxy aged 50 hours

Table 8.176: Flexural Results for Neat Cycloaliphatic Epoxy aged 50 hours

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
2	111.093	3.952	3.156
3	122.073	4.499	3.194
4	118.785	4.052	3.348
5	123.928	4.362	3.317
18	108.917	4.051	3.073
24	93.889	3.233	3.087
31	87.556	2.912	3.127
27	89.556	3.098	3.026
16	104.714	3.832	3.027
35	109.252	3.965	3.100
Average	106.98	3.80	3.15
Std Dev	13.07	0.54	0.11
Count	10	10	10

Figure 8.75: Flexural Results for Neat Cycloaliphatic Epoxy aged 100 hours

Table 8.177: Flexural Results for Neat Cycloaliphatic Epoxy aged 100 hours

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
6	91.009	2.810	3.338
7	89.280	2.863	3.198
8	86.732	2.822	3.181
19	82.032	2.698	3.131
32	93.391	2.996	3.277
70	116.311	4.110	3.255
15	110.498	3.934	3.151
25	124.716	5.746	3.117
34	94.239	3.232	3.088
20	94.709	3.108	3.208
23	96.582	3.347	3.096
Average	98.14	3.42	3.19
Std Dev	13.27	0.90	0.08
Count	11	11	11

Figure 8.76: Flexural Results for Neat Cycloaliphatic Epoxy aged 200 hours

Table 8.178: Flexural Results for Neat Cycloaliphatic Epoxy aged 200 hours

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
11	115.414	3.995	3.270
12	91.984	3.107	3.091
14	120.326	4.422	3.149
15	107.088	3.616	3.201
45	116.254	4.365	3.083
49	95.533	3.176	3.161
54	108.402	3.714	3.210
55	110.364	3.704	3.276
43	93.762	3.029	3.221
58	93.470	3.037	3.224
Average	105.26	3.62	3.19
Std Dev	10.71	0.53	0.07
Count	10	10	10

Figure 8.77: Flexural Results for Neat Cycloaliphatic Epoxy aged 400 hours

Table 8.179: Flexural Results for Neat Cycloaliphatic Epoxy aged 400 hours

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
16	76.277	2.392	3.230
18	121.332	4.520	3.174
19	108.003	3.707	3.190
20	107.914	3.797	3.145
Average	103.38	3.60	3.18
Std Dev	19.14	0.89	0.04
Count	4	4	4

Figure 8.78: Flexural Results for 10% Talc in Cycloaliphatic Epoxy Unaged

Table 8.180: Flexural Results for 10% Talc in Cycloaliphatic Epoxy Unaged

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
12	95.274	3.047	3.250
15	97.169	2.927	3.621
16	89.409	2.916	3.311
17	103.881	3.261	3.561
19	97.463	2.794	3.731
Average	96.64	2.99	3.49
Std Dev	5.19	0.18	0.21
Count	5	5	5

Figure 8.79: Flexural Results for 10% Talc in Cycloaliphatic Epoxy Aged 50 hours

Table 8.181: Flexural Results for 10% Talc in Cycloaliphatic Epoxy Aged 50 hours

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
2	99.606	3.261	3.598
3	96.881	3.072	3.194
4	86.970	2.679	3.608
5	86.308	2.761	3.482
Average	92.44	2.94	3.47
Std Dev	6.80	0.27	0.19
Count	4	4	4

Figure 8.80: Flexural Results for 10% Talc in Cycloaliphatic Epoxy Aged 100 hours

Table 8.182: Flexural Results for 10% Talc in Cycloaliphatic Epoxy Aged 100 hours

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
7	83.591	2.648	3.335
8	83.931	2.631	3.498
9	73.837	2.223	3.533
10	87.722	2.828	3.459
Average	82.27	2.58	3.46
Std Dev	5.93	0.26	0.09
Count	4	4	4

Figure 8.81: Flexural Results for 10% Talc in Cycloaliphatic Epoxy Aged 200 hours

Table 8.183: Flexural Results for 10% Talc in Cycloaliphatic Epoxy Aged 200 hours

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
11	83.834	2.649	3.494
12	79.586	2.483	3.351
13	72.710	2.219	3.492
14	82.768	2.652	3.451
15	78.832	2.440	3.519
Average	79.55	2.49	3.46
Std Dev	4.36	0.18	0.07
Count	5	5	5

Figure 8.82: Flexural Results for 10% Talc in Cycloaliphatic Epoxy Aged 400 hours

Table 8.184: Flexural Results for 10% Talc in Cycloaliphatic Epoxy Aged 400 hours

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
16	70.924	2.140	3.506
17	64.049	1.895	3.538
18	64.333	1.971	3.485
19	88.331	2.690	3.621
20	69.996	2.051	3.575
Average	71.53	2.15	3.55
Std Dev	9.91	0.32	0.05
Count	5	5	5

Figure 8.83: Flexural Results for 20% Talc in Cycloaliphatic Epoxy Unaged

Table 8.185: Flexural Results for 20% Talc in Cycloaliphatic Epoxy Unaged

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
1	89.116	2.332	4.272
3	80.928	2.314	3.812
4	87.328	2.327	4.174
5	74.857	2.092	3.867
9	84.060	2.319	3.949
Average	83.26	2.28	4.01
Std Dev	5.65	0.10	0.20
Count	5	5	5

Figure 8.84: Flexural Results for 20% Talc in Cycloaliphatic Epoxy Aged 50 hours

Table 8.186: Flexural Results for 20% Talc in Cycloaliphatic Epoxy Age 50 hour

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
1	81.731	2.230	4.150
2	76.660	2.049	4.145
3	80.568	2.215	4.047
4	77.102	2.139	4.001
5	82.809	2.270	4.092
Average	79.77	2.18	4.09
Std Dev	2.76	0.09	0.06
Count	5	5	5

Figure 8.85: Flexural Results for 20% Talc in Cycloaliphatic Epoxy Age 100 hour

Table 8.187: Flexural Results for 20% Talc in Cycloaliphatic Epoxy Age 100 hour

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
6	72.510	1.985	4.015
8	74.021	2.022	4.020
9	67.661	1.825	4.076
10	68.628	1.849	4.084
Average	70.71	1.92	4.05
Std Dev	3.05	0.10	0.04
Count	4	4	4

Figure 8.86: Flexural Results 20% Talc in Cycloaliphatic Epoxy Age 200 hour

Table 8.188: Flexural Results for 20% Talc in Cycloaliphatic Epoxy Age 200 hour

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
11	67.528	1.840	4.108
12	73.348	2.065	4.014
13	71.884	1.884	4.292
14	56.972	1.551	3.961
15	60.623	1.589	4.103
Average	66.07	1.79	4.10
Std Dev	7.09	0.21	0.13
Count	5	5	5

Figure 8.87: Flexural Results for 20% Talc in Cycloaliphatic Epoxy Age 400 hour

Table 8.189: Flexural Results for 20% Talc in Cycloaliphatic Epoxy Age 400 hour

Specimen	Peak Stress, σ_{fM} (MPa)	Strain at Peak Stress, ϵ_{fM} (%)	Modulus, (GPa)
16	69.898	1.880	4.117
17	64.648	1.768	3.983
18	77.189	2.152	4.060
19	65.042	1.762	4.078
20	67.794	1.776	4.250
Average	68.91	1.87	4.10
Std Dev	5.10	0.17	0.10
Count	5	5	5

8.17 Aging Talc DMA Data

Table 8.190: DMA Results for Neat Cycloaliphatic Epoxy Unaged

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	213.83	231.95	251.0
2	213.25	232.34	249.4
3	213.74	233.40	250.1
4	213.53	233.08	251.8
Average	213.59	232.69	250.58
Std Dev	0.26	0.66	1.05

Table 8.191: DMA Results for Neat Cycloaliphatic Epoxy Aged 50 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	199.81	217.24	248.7
2	200.74	217.96	248.6
3	200.14	216.95	246.9
4	201.15	217.85	248.5
Average	200.46	217.5	248.18
Std Dev	0.60	0.48	0.85

Table 8.192: DMA Results for Neat Cycloaliphatic Epoxy Aged 100 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	195.57	211.66	248.7
2	193.32	212.87	248.4
3	192.99	212.10	249.9
4	191.62	211.78	247.5
Average	193.38	212.1	248.63
Std Dev	1.64	0.54	0.99

Table 8.193: DMA Results for Neat Cycloaliphatic Epoxy Aged 200 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	188.67	208.97	247.1
2	185.89	206.61	246.8
3	186.9	209.09	247.2
4	186.72	208.85	245.0
Average	187.05	208.38	246.53
Std Dev	1.17	1.18	1.03

Table 8.194: DMA Results for Neat Cycloaliphatic Epoxy Aged 398 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	178.14	200.87	245.2
2	181.31	204.12	246.2
3	180.22	202.72	244.9
4	178.44	203.19	247.0
Average	179.53	202.73	245.83
Std Dev	1.50	1.37	0.96

Table 8.195: DMA Results for 10 wt% Talc in Cycloaliphatic Epoxy Unaged

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	211.85	230.34	256.4
2	212.97	231.65	256.0
3	210.71	227.28	252.5
4	210.66	228.41	253.1
Average	211.55	229.42	254.5
Std Dev	1.10	1.95	1.98

Table 8.196 DMA Results for 10 wt% Talc in Cycloaliphatic Epoxy Aged 50 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	203.9	218.76	252.9
2	203.12	218.75	253.5
3	199.84	216.41	250.8
4	198.59	216.54	250.8
Average	201.36	217.62	252.0
Std Dev	2.55	1.32	1.41

Table 8.197: DMA Results for 10 wt% Talc in Cycloaliphatic Epoxy Aged 100 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	196.27	213.47	246.2
3	197.86	215.95	249.4
4	194.76	214.35	247.0
Average	196.3	214.59	247.53
Std Dev	1.55	1.26	1.67

Table 8.198: DMA Results for 10 wt% Talc in Cycloaliphatic Epoxy Aged 200 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	189.29	206.23	247.8
2	187.61	208.67	248.8
4	194.6	208.36	251.6
Average	190.5	207.75	249.4
Std Dev	3.65	1.33	1.97

Table 8.199: DMA Results for 10 wt% Talc in Cycloaliphatic Epoxy Aged 398 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	183.91	204.63	249.6
2	181.85	204.14	249.2
3	185.45	204.35	245.3
4	184.76	204.68	247.3
Average	183.99	204.45	247.85
Std Dev	1.56	0.25	1.97

Table 8.200: DMA Results for 20 wt% Talc in Cycloaliphatic Epoxy Unaged

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	209.88	225.58	249.2
2	209.73	226.38	250.7
3	210.31	226.75	252.2
Average	209.97	226.24	250.7
Std Dev	0.30	0.60	1.50

Table 8.201: DMA Results for 20 wt% Talc in Cycloaliphatic Epoxy Aged 50 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
2	200.18	216.39	248.3
3	190.8	215.84	244.9
4	198.42	216.16	247.9
Average	196.47	216.13	247.03
Std Dev	4.99	0.28	1.86

Table 8.202: DMA Results for 20 wt% Talc in Cycloaliphatic Epoxy Aged 100 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	192.78	209.79	246.3
3	189.59	209.63	245.4
4	192.45	211.0	249.2
Average	191.61	210.14	246.97
Std Dev	1.75	0.75	1.99

Table 8.203: DMA Results for 20 wt% Talc in Cycloaliphatic Epoxy Aged 200 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	188.18	204.20	247.2
2	187.76	204.58	248.3
3	187.16	203.79	248.0
4	183.15	203.65	248.1
Average	186.56	204.06	247.9
Std Dev	2.31	0.42	0.48

Table 8.204: DMA Results for 20 wt% Talc in Cycloaliphatic Epoxy Aged 398 Hours

Test	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
1	184.41	201.95	247.5
2	181.94	201.56	247.1
3	181.76	200.95	247.3
4	183.4	202.34	247.0
Average	182.88	201.7	247.23
Std Dev	1.26	0.59	0.22

Figure 8.88: Tan Delta Peak for Aged Talc/CE composites

Figure 8.89: Storage Modulus Onset for Aged Talc/CE composites

Figure 8.90: Loss Modulus Peak for Aged Talc/CE composites

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