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PROBING THE PRECAMBRIAN GEODYNAMO: ANALYSIS OF THE GEOMAGNETIC FIELD BEHAVIOR AND CALIBRATION OF PSEUDO-THELLIER PALEOINTENSITY METHOD FOR MESOPROTEROZOIC ROCKS

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ANALYSIS OF THE GEOMAGNETIC FIELD BEHAVIOR AND CALIBRATION OF
PSEUDO-THELLIER PALEOINTENSITY METHOD FOR MESOPROTEROZOIC
ROCKS

By
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# Table of contents

Table of contents .................................................................................................................. iii

List of figures.................................................................................................................................... v

List of tables..................................................................................................................................... ix

Acknowledgments .......................................................................................................................... x

Abstract........................................................................................................................................ xi

## 1 Introduction .......................................................................................................................... 13

1.1 Overview .................................................................................................................................. 13

1.2 Earth’s magnetic field geometry, paleosecular variation, and paleointensity 14

1.2.1 Field geometry and paleosecular variation ............................................................ 15

1.2.2 Paleointensity ........................................................................................................... 17

1.3 Current challenges of paleomagnetic research ............................................................... 18

1.4 Goals and questions addressed in this work ................................................................. 30

## 2 New paleomagnetic and paleointensity investigation of the Late-Mesoproterozoic mafic dikes related to the North American Midcontinent Rift in Michigan and Wisconsin ......................................................................................................................... 32

2.1 Introduction ............................................................................................................................. 32

2.2 Geological setting .................................................................................................................. 35

2.3 Sampling ................................................................................................................................. 42

2.4 Measurement of paleomagnetic directions ........................................................................ 46

2.5 Magnetic mineralogy ............................................................................................................ 59

2.6 Paleointensity determinations .............................................................................................. 68

2.7 Discussion ............................................................................................................................... 75

2.7.1 Paleosecular variation .............................................................................................. 75

2.7.2 Intrusion and age of remanence ................................................................................ 77

2.7.3 Paleointensity and the geodynamo ............................................................................. 81

2.8 Conclusions ............................................................................................................................ 85

## 3 Paleomagnetism and rock magnetism of the Greenstone Flow: Implications for the flow emplacement process and geomagnetic secular variation ......................................................................................................................... 87

3.1 Introduction ............................................................................................................................ 87
3.2 Geological setting ........................................................................................................92
3.2.1 The Portage Lake Volcanics ..................................................................................92
3.2.2 The Greenstone Flow ............................................................................................93
3.3 Methods .....................................................................................................................98
3.4 Magnetic mineralogy ...............................................................................................100
3.5 Paleomagnetic directions ..........................................................................................106
3.6 Discussion ..................................................................................................................116
3.6.1 Stability of the remanence direction ...................................................................116
3.6.2 Paleosecular variation .........................................................................................116
3.6.3 Implications for the Greenstone Flow formation ..............................................118
3.7 Conclusions ..............................................................................................................121

4 Calibration of the pseudo-Thellier paleointensity determination for Precambrian Rocks .........................................................122
4.1 Introduction ..............................................................................................................122
4.2 Methods ...................................................................................................................127
4.3 Rock magnetic properties .........................................................................................131
4.4 Pseudo-Thellier results ............................................................................................135
4.5 Selection of data based on magnetic grain size and thermomagnetic behavior .................................................................139
4.6 Calibrated pseudo-Thellier results on Precambrian rocks .......................................142
4.7 Implications for the paleointensity of the Baraga and Marquette dikes ..................144
4.8 Conclusions ..............................................................................................................146

5 Conclusions ................................................................................................................147

6 References ..................................................................................................................152
List of figures

Figure 1.1. Changes in the magnetic field characteristics ...............................................19
Figure 1.2. Predicted field modeling and the current paleointensity database .............24
Figure 1.3. Variation of the angular dispersion for Precambrian rocks .........................29

Figure 2.1. The rock formations and dikes swarms associated with the Mid-Continent Rift ........................................................................................................................................34
Figure 2.2. Sampled locations in the Mellen-Gogebic and Central-Wisconsin areas (circles) ........................................................................................................................................37
Figure 2.3. Sampling sites in the vicinity of Marquette (circles) ........................................38
Figure 2.4. Sampling sites in the Baraga area (circles) ..................................................39
Figure 2.5. Rose diagrams of the dike orientations .........................................................43
Figure 2.6. Annotated photos of the Baraga and Marquette dikes ..................................49
Figure 2.7. Examples of paleomagnetic results ..............................................................50
Figure 2.8. Equal-area plot with the reversed site-mean paleomagnetic directions from the Baraga ........................................................................................................................................53
Figure 2.9. Equal-area plot with the reversed site-mean paleomagnetic directions from the Marquette cites ........................................................................................................................................54
Figure 2.10. Equal-area plot with the normal site-mean paleomagnetic directions .......55
Figure 2.11. Summary of the paleomagnetic results .........................................................57
Figure 2.12. Equal-area plots showing examples of relationships between paleomagnetic results from dike, host-rocks, and/or cross-cutting dikes investigated in this study ........................................................................................................................................58
Figure 2.13. Examples of the three main types of thermomagnetic curves from the dikes ........................................................................................................................................61
Figure 2.14. Weak-field magnetic susceptibility versus natural remanent magnetization (NRM) intensity plot ........................................................................................................................................61
Figure 2.15. Comparison of the stability and alteration of the analyzed dikes ..........62
Figure 2.16. Day plot (Day et al., 1977) of the studied dikes sorted by paleomagnetic results and locations .................................................................62

Figure 2.17. Examples of magnetic hysteresis loops and backfield remanence demagnetization curves from samples with different thermomagnetic behavior ..64

Figure 2.18. Examples of FORC distributions measured at room temperature from the Baraga-Marquette dikes ........................................................................................................65

Figure 2.19. Representative reflected light images of the Baraga-Marquette dikes with a primary reversed direction .................................................................66

Figure 2.20. Alteration features in dikes carrying a primary and/or secondary normal remanence .....................................................................................67

Figure 2.21. Example of a successful result from the LTD-DHT Shaw dataset for a natural specimen ........................................................................................................70

Figure 2.22. Example of an unsuccessful result from the LTD-DHT Shaw dataset for a natural specimen ........................................................................................................71

Figure 2.23. Angular dispersion of the VGP obtained in this study .......................76

Figure 2.24. Analysis of the Virtual Geomagnetic Poles (VGP) from this study on the Logan loop .................................................................................................78

Figure 2.25. Relative position of the VGP obtained in this study with respect to the Late Mesoproterozoic APWP. .....................................................................................80

Figure 2.26. Analysis of the mean paleointensity and mean Virtual Dipole Moment (VDM) ....................................................................................................................82

Figure 2.27. Comparison of the VDM values from our study (red squares and a diamond) with other Precambrian intensity determinations. .....................................................84

Figure 3.1. Geological map showing the Mid-Continent Rift formations in Lake Superior area ..................................................................................................................88

Figure 3.2. Schematic cross-section of the North-American Mid-continent Rift ........89

Figure 3.3. Extent of the Greenstone Flow and sampling locations on the Keweenaw Peninsula .................................................................................................90

Figure 3.4. Simplified geological map showing the extent of the Greenstone Flow on Isle Royale .................................................................................................91
Figure 3.5. Sketch showing the thickness variation of the Greenstone flow and the Allouez conglomerate ........................................................................................................93

Figure 3.6. Simplified composition of the Greenstone Flow at each studied location ..............................................................95

Figure 3.7. Simplified models for formation of the Greenstone Flow .........................................................................................97

Figure 3.8. Variation of magnetic susceptibility with temperature throughout the different units of the Greenstone Flow. ..............................................................................102

Figure 3.9. Examples of thermomagnetic curves measured from the different parts of the GSF ...........................................................................................................................................103

Figure 3.10. Rock magnetic characteristics of the Greenstone Flow ..........................................................................................104

Figure 3.11. Examples of magnetic hysteresis curves measured from the different parts of the GSF ........................................................................................................................................105

Figure 3.12. Examples of paleomagnetic results from the Greenstone Flow ..................................................................................108

Figure 3.13. Paleomagnetic results for the Greenstone Flow .......................................................................................................109

Figure 3.14. Variation of the paleomagnetic directions with depth into the Greenstone Flow .................................................................................................................................110

Figure 3.15. Comparison of paleomagnetic results from different locations within the GSF and from the other PLV flows ..............................................................................................111

Figure 3.16. Analysis of the Virtual Geomagnetic Pole (VGP) dispersion ..................................................................................117

Figure 4.1. Calibration of the pseudo-Arai slope from previous studies .........................................................................................126

Figure 4.2. Schematic representation of the pseudo-Thellier-Shaw protocol used in this study ........................................................................................................................................129

Figure 4.3. Examples of pseudo-Thellier plots for synthetic and natural specimens ..............................................................................130

Figure 4.4. Variation of the low-field magnetic susceptibility with temperature ................................................................................132

Figure 4.5. Comparison of the magnetic hysteresis parameters and IRM acquisition curves before and after paleointensity experiments ...........................................................................133

Figure 4.6. Variation of the half-saturating ARM fields ..................................................................................................................134

Figure 4.7. Average pseudo-Thellier slope results for our synthetic specimens according to the B_{TRM}/B_{ARM} ratio ..............................................................................136
Figure 4.8. Comparison between the ratio of calibrated pseudo-Thellier field estimates over the laboratory field, and the half-saturating ARM field ..........................140

Figure 4.9. Correlation between the generalized correlation factor and rock magnetic characteristics..............................................................................................................141

Figure 4.10. Boxplot diagram showing the statistical distribution of the average paleointensity estimates ........................................................................................................142

Figure 5.1. New group-mean paleointensity data ..........................................................................................................................149
List of tables

Table 2.1 Summary of the sites sampled for this study .....................................................44
Table 2.2. Paleomagnetic results ........................................................................................51
Table 2.3. The group-mean directions and paleomagnetic poles from the Mesoproterozoic
   dikes .......................................................................................................................56
Table 2.4. Successful LTD-DHT Shaw paleointensity determinations from the R-polarity
   BM dikes. ...............................................................................................................72
Table 2.5. Results of the LTD-DHT Shaw experiments .....................................................83
Table 3.1. Summary of the sampling sites .........................................................................99
Table 3.2. Summary of the paleomagnetic and corresponding virtual geomagnetic pole
   (VGP) data from this study ..................................................................................112
Table 3.3. Paleomagnetic results from previous studies on the Greenstone Flow ............113
Table 3.4. Summary of the mean discrimination test performed on the paleomagnetic site
   means from the GSF ............................................................................................114
Table 3.5. Summary of the group means calculated for the Greenstone Flow ...............115
Table 4.1. Summary of the selection criteria statistics for the accepted specimens ........137
Table 4.2. Pseudo-Thellier results for the synthetic specimens .................................138
Table 4.3. Efficiency of the calibration formulas to predict the acquisition field of the
   laboratory TRM induced in the natural synthetic specimens ...............................140
Table 4.4. Summary of the average pseudo-Thellier slopes for the natural specimens from
   the Baraga-Marquette dikes ..................................................................................143
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Abstract

Understanding the geomagnetic field direction and strength (e.g., paleomagnetism and paleointensity, respectively) recorded by Precambrian rocks is essential to obtain insight into the nature and evolution of the Earth’s early geodynamo and for constraining models of planetary evolution. Major milestones of our planet’s history, such as beginning of plate tectonics, development of the atmosphere and life, took place during the first four billion years. However, the available data on the Earth’s magnetic field in the Precambrian are very limited, especially the information about the field intensity which represents one of the most challenging aspect of paleomagnetic research. Many Precambrian rocks are not suitable for paleointensity determinations with the most commonly used Thellier double-heating method because of their geological and experimental alteration. Complementing the Precambrian paleomagnetic and paleointensity database and improving alternative paleointensity techniques is essential to better our understanding of the Earth.

This dissertation presents the results of investigations of the Precambrian geomagnetic field direction and strength, and the results of a methodological study of the pseudo-Thellier experimental protocol for absolute paleointensity determination. Detailed paleomagnetic and rock magnetic observations were performed on rocks emplaced ~1100 Ma during the formation of the North-American Mid-Continent Rift. The identification of three independent paleomagnetic directions in the Baraga and Marquette dikes and their comparison to the Mesoproterozoic Apparent Polar Wonder Path (the so-called Logan loop) permitted the estimation of the relative timescale for their intrusion (Chapter 2). The Shaw paleointensity results on the dikes from Baraga and Marquette areas yielded consistent intensity values of ~14.4 μT and ~20.4 μT corresponding to a VDM of ~2 x 10^{22} Am^2 and 3.3 x 10^{22} Am^2, respectively. The detailed paleomagnetic analysis of the ~1094 Ma Greenstone flow, one of the largest lava flows on the planet, was applied to evaluate the various scenarios of emplacement and cooling of the flow and to assess the paleosecular variation of the geomagnetic field (Chapter 3). The results of an experimental study using the combination of the pseudo-Thellier and Shaw experimental protocols on synthetic and natural magnetite-bearing samples suggest that the calibrated pseudo-Thellier method can
provide absolute paleointensity estimates for Precambrian rock equivalent to the results from conventional heating methods (Chapter 4). The paleointensity values (2-3 x 10^{22} \text{ Am}^2) obtained from the Baraga and Marquette dikes are low in comparison to the average field strength value (~8.0 x 10^{22} \text{ Am}^2) for the last 10 Myr but agree with some low paleointensity determinations from other Precambrian rocks. These results suggest that the Baraga-Marquette dikes intruded during a period where the geomagnetic field was weak. However, a possibility of underestimating the field strength due to the presence of thermochemical remanent magnetization cannot be ruled out.
1 Introduction

1.1 Overview

Many rocks preserve the record of the strength and direction of Earth’s magnetic field from the time of their formation. For example, during their initial cooling, igneous rocks may acquire a thermal remanent magnetization (TRM); the direction of this TRM is parallel to the paleofield and its intensity is directly proportional to the paleofield strength (paleointensity). This record (paleomagnetism, or fossil magnetism) represents one of the most important sources of our empirical knowledge about the Earth’s planetary evolution before the era of direct observation and historical records (e.g., Irving, 1964). The paleodirectional and paleointensity data have played an instrumental role in deciphering the history of our planet (e.g., Voo and Jelenska, 1993; Tarduno et al., 2006; Torsvik et al., 2008) including a decisive evidence for continental drift (e.g., Irving, 1964). Continental reconstructions based on the paleomagnetic record provide a useful framework for interpretation of geological and geochemical data (e.g., Halls, 2008). However, the importance of paleomagnetic data extends well beyond paleotectonic applications because the geomagnetic field is closely linked with other Earth system processes.

The Earth’s magnetic field (magnetosphere) shields the biosphere and atmosphere from solar and cosmic radiation. Variations in the field strength and geometry have been shown to affect the atmospheric chemistry, for example, by modulating the production rates of cosmogenic nuclides such as $^{14}$C, $^{36}$Cl, and $^{10}$Be (e.g., Fournier et al., 2015; Lifton, 2016). A defining role of the field strength and stability has also been suggested for the early life development (e.g., Doglioni et al., 2016), biological mutation rates (e.g., Simpson, 1966; Hays, 1971), and even human evolution (Kopper and Papamarinopoulos, 1978); however these hypotheses remain controversial (e.g., Glassmeier and Vogt, 2010). One of the most important factors that hampers the progress in our understanding of the link between magnetosphere, atmosphere, and biosphere is the deficiency of reliable paleointensity data for most of the geological history.
The characteristics and evolution of the geomagnetic field are also ultimately linked to the Earth’s deep interior processes. The process of field generation by convection of iron in the liquid outer core (geodynamo) is fueled by the temperature gradient due to secular cooling of the planet, and by compositional gradient and latent heat of the crystallizing inner core (Verhoogen, 1961; Stacey, 1972). In this conventional view, the compositionally-driven convection plays a crucial role because a purely thermally-driven geodynamo in the entirely liquid core is thought to be incapable of sustaining a strong long-term field (e.g., Gubbins et al., 2003; Buffett, 2003).

Another critically important factor that controls the geodynamo efficiency is the transport of heat through the mantle because the vigor of the outer core convection directly depends on the rate of core cooling (i.e., the heat flow through the core-mantle boundary, CMB) (e.g., Olson et al., 2015). In particular, the long-term variations of the geomagnetic field strength and stability (e.g., the strength of secular variation) may reflect the changes of the heat flux through the mantle (including the variation in mantle plume activity) (e.g., Loper, 1992). For example, the long period of non-reversing and less variable field between ~124 and 84 Ma (the Cretaceous Normal Polarity Superchron, CNS) may reflect a highly efficient geodynamo due to a high CMB heat flow caused by an increased mantle plume activity in the early Cretaceous (e.g., Larson and Olson, 1991). Therefore, investigations of the long-term history of Earth’s magnetic field, including geomagnetic polarity reversals, secular variation, and paleointensity variation, provide crucial information for understanding both geodynamo and thermal evolution of our planet.

1.2 Earth’s magnetic field geometry, paleosecular variation, and paleointensity

At any point of space, the Earth’s magnetic field is fully characterized by its strength (intensity) and direction. The field direction is described by two angles: declination (D) and inclination (I). Declination is the angle between the directions to the magnetic pole (i.e., the horizontal projection of the total field vector) and to the geographic north, whereas inclination is the angle between the total field vector and the horizontal plane (i.e., the dip
of freely suspended magnetic needle). Paleomagnetic directional and paleointensity research provides data on the past values of declination and inclination, and on the field intensity, respectively.

1.2.1 Field geometry and paleosecular variation

The cornerstone of paleomagnetic research is the Geocentric Axial Dipole (GAD) assumption which states that the Earth’s magnetic field averaged over sufficient time (10,000–100,000 years) is equivalent to the field of a magnetic dipole positioned at Earth’s center and aligned with the rotational axis. This allows us to apply the dipole equations to reconstruct past positions of continents using the direction (D and I) of ancient magnetic field recorded in rocks. The GAD geometry of time-averaged field is reliably established for at least 500-600 millions of years. Furthermore, analyses of paleoclimatological or paleogeographical indicators (e.g., Kent and Smethurst, 1998; Evans, 2006), paleosecular variation (Smirnov et al., 2011), and the zonal harmonics of the geomagnetic field (Veikkolainen et al., 2014; Salminen et al., 2017) indicate that the time-averaged field probably has had the GAD geometry since the Neoarchean. However, the amount and spatiotemporal distribution of the paleomagnetic directional data to date remain insufficient to make a final decisive conclusion for the Precambrian field geometry.

To be used for paleocontinental reconstructions and analyses of the geodynamo evolution, paleomagnetic directions (i.e., D and I) obtained from ancient rock samples are usually converted into the coordinates of virtual paleomagnetic poles (VGP) using the GAD assumption (e.g., Butler, 1998). The VGP coordinates describe the location of an ancient geomagnetic pole with respect to the sampling site. However, an individual VGP represents an instant snapshot of geomagnetic field. In order to obtain the pole position representing the time-averaged field, a set of VGPs measured from a rock sequence (e.g., a lava flow sequence) is used to calculate a mean pole using the Fisher statistics (Fisher, 1953). In addition, the scatter of the individual VGPs measured from independently cooled units is used to evaluate the strength of paleosecular variation (PSV) by calculating the angular dispersion (S) of VGPs:
\[ S = \sqrt{\frac{1}{N-1} \sum_{i=1}^{N} \Delta_i^2} \]  

(1)

where \( N \) is the total number of VGP's and \( \Delta_i \) is the angle between the \( i \)-th VGP and the group mean pole (e.g., McFadden et al., 1991). When possible (i.e. the sample-mean statistics are known), the dispersion values are corrected for within-site dispersion (\( S_w \)) related to intrinsic variation and experimental uncertainty using:

\[ S_b^2 = S^2 - S_w^2/n \]  

(2)

where \( S_b \) is the true (between-site) dispersion and \( n \) is the average number of samples per site (Doell, 1970). However, the difference between the values of \( S \) and \( S_b \) rarely exceeds one degree. The confidence interval for \( S_b \) are typically estimated using the N-1 jackknife method (Efron, 1982).

The data on PSV are usually interpreted using Model G (McFadden et al., 1991) in which the angular dispersion of VGPs (\( S \)) is represented as a superposition of contributions from two independent geodynamo families - the dipole (\( S_D \), or antisymmetric) family and the quadrupole (\( S_Q \), or symmetric) family, with the latter dominating at the equator:

\[ S(\lambda) = \sqrt{S_D^2 + S_Q^2} = \sqrt{(a\lambda)^2 + b^2} \]  

(3)

where \( \lambda \) is paleolatitude, and \( a \) and \( b \) are constants. Although complete independence of the two families is unlikely, Model G remains a useful framework to gauge past PSV. For example, the shape of \( S(\lambda) \) curve may reflect the latitudinal pattern of the CMB heat flow. More importantly, Model G allows us to make inferences on the field dipolarity (the relative contribution of dipole family to quadrupole family) in the past by comparing the values of equatorial intercept of the \( S(\lambda) \) curve for different time periods. Because the dipole geodynamo family does not contribute to the value of \( S \) at the equator, the relatively lower or higher \( S(0^\circ) \) values are interpreted as representing a more or less dipolar field, respectively (e.g., Smirnov and Tarduno, 2004).
1.2.2 Paleointensity

Data on the ancient field strength \( (H_{anc}) \) provide a unique insight into the energetics of geodynamo; however, paleointensity determination represents the most challenging aspect of paleomagnetic research. For example, while paleodirections can be reliably measured even if only a small part of the primary magnetization vector is retained in the rock, determination of \( H_{anc} \) requires a much more complete preservation of the vector.

The overwhelming majority of paleointensity data have been obtained using the Thellier double-heating method (Thellier and Thellier, 1959) in which the natural remanent magnetization (NRM) of a sample is gradually replaced with a thermal remanent magnetization (TRM) induced in a known magnetic field in a series of paired heating steps (e.g., Coe, 1967). First, the NRM is partly demagnetized by cycling to a given temperature in a field-free space. Next, the sample is cycled to the same temperature in a laboratory field \( H_{lab} \) to impart a partial TRM (pTRM). These double “zero-field” and “field-on” steps are repeated for higher temperature increments until the NRM is replaced with the laboratory TRM. If the pTRMs obey the Thellier laws of reciprocity, independence, and additivity (e.g., Thellier, 1941), the paleointensity \( (H_{anc}) \) can be calculated from the NRM lost \( (\Delta M_{NRM}) \), the pTRM gained \( (\Delta M_{pTRM}) \), and \( H_{lab} \) using:

\[
H_{anc} = \frac{\Delta M_{NRM}}{\Delta M_{TRM}} H_{lab}
\]  

(4)

Conventionally, the paleointensity is calculated from the slope of a linear segment on an Arai (NRM-remaining versus pTRM-gained) plot (Nagata et al., 1963).

In order to account for the latitudinal dependence of field strength, the measured paleointensity value is recalculated into virtual dipole moment (VDM) using the following relationship based on the GAD assumption:

\[
VDM = \frac{4\pi R^3}{\mu_0 \sqrt{(1 + 3 \sin^2 \lambda)}} H_{anc}
\]

(5)
where $R$ is the Earth radius, $\mu_0$ is the magnetic permeability of free space ($\mu_0 = 4\pi \times 10^{-7}$ H·m$^{-1}$), and $\lambda$ is the site paleolatitude.

One of the most critical requirements of the Thellier method is that the magnetic mineral makeup of the sample must not change during paleointensity experiments. However, multiple heating steps required by the Thellier method often result in magnetomineralogical alteration due to formation of fine-grained magnetic minerals from clays (Hirt and Gehring, 1991; Hirt et al., 1993), the transformation or neoformation of magnetic minerals (e.g., Kosterov and Prévot, 1998; Smirnov and Tarduno, 2003), and other processes. Such an alteration often begins innocuously, with only slight changes in magnetic properties. At higher temperature increments and longer total times of heating the alteration can be detected by the standard experimental protocol (the pTRM-checks; Coe et al., 1978), when a pTRM is imparted at a lower temperature to check for the growth of magnetic minerals. Application of the pTRM checks and other alteration-detection procedures (e.g., Riisager and Riisager, 2001) results in rejecting many paleointensity determinations. The pervasive laboratory alteration substantially limits the scope of rocks suitable for Thellier experiments, which emphasizes the importance of development of alternative paleointensity determination methods that are less affected by alteration.

1.3 Current challenges of paleomagnetic research

Despite many substantial achievements of paleomagnetic research over the course of several decades, some first-order questions about the long-term behavior of geomagnetic field during the geological history remain open. One such question is whether long-term variations of geomagnetic polarity reversals, secular variation, and field strength are intrinsically related or whether they have varied independently throughout geological history. Importantly, a large class of geodynamo models consistently predicts an inverse relationship between geomagnetic reversal frequency and paleointensity (e.g., Glatzmaier et al., 1999; Driscoll and Olson, 2009, 2011; Olson et al., 2010; Aubert et al., 2010; Amit and Olson, 2015; Olson and Amit, 2015). The models also predict a more stable (i.e. lower $S$ values) and more dipolar field for the periods of low reversal frequency (e.g.,
superchrons), and a more variable (higher $S$ values) and less dipolar field for the periods of high reversal frequency.

Figure 1.1. Changes in the magnetic field characteristics (a) Variation of dipole family ($S_D$; open circles) and quadrupole family ($S_Q$; closed circles) contributions versus time from McFadden et al. (1991). The blue line shows variation of geomagnetic reversal rate based on a 10 Myr sliding window. (b) Paleointensity data for the last 180 million years obtained with the Thellier method with pTRM checks (see Section 1.2). The small grey open circles are individual site-means from bulk rock samples for which the standard deviation to the site-mean ratio does not exceed 25 per cent. The larger green circles show the study-means based on at least 12 determinations from at least four cooling units. The red diamonds show the study-means obtained from single silicate crystals. The error bars are 1 $\sigma$. The dashed line shows the average field value for the last 10 Ma. The pink rectangle highlights the Cretaceous normal polarity superchron (CNS). The red box over the geomagnetic reversal timescale shows the period of a very high reversal rate in the late Jurassic. Note that the
CNS is characterized by the weakest $S_Q$ family contribution and the highest single crystal paleointensities, whereas the opposite is observed for the highest reversal frequency period in the late Jurassic.

However, while the PSV analyses confirm the predicted relationship (Figure 1.1.a), most of the existing paleointensity data - dominated by results from bulk volcanic rocks - fail to confirm this relationship (e.g., Ingham et al., 2014) (Figure 1.1.b). Although low paleointensity values (50-60% of the present-day intensity) are recorded during periods of high reversal rate and field variability (e.g., a portion of the late Jurassic) (Tarduno and Cottrell, 2005; Tauxe et al., 2013; Sprain et al., 2016), high values that markedly exceed the present-day intensity are extremely rare during periods with no or few reversals and low field variability (most notably, the CNS) (Zhu et al., 2008; Ingham et al., 2014) (Figure 1.1.b). However, at the same time, high-fidelity paleointensity data from single silicate crystals corroborate an inverse relationship between geomagnetic reversal rate and field strength (Tarduno et al., 2006). The single crystal data consistently indicate high field strengths during superchrons (Tarduno et al., 2001, 2002; Cottrell et al., 2008) and lower field strengths during periods of high reversal frequency (Tarduno and Cottrell, 2005) (Figure 1.1.b).

Recently, Smirnov et al. (2017) addressed this contradiction and showed that many paleointensity data obtained from bulk rock samples using the conventional Thellier method (Section 1.2) are affected by a low-field bias due to the presence of non-ideal magnetic carriers, which is likely to contribute to the dominance of low VDM values in the global paleointensity database. The authors suggested that this low-field bias together with several additional mechanisms, such as experimental alteration of basalts and basaltic glass, low-temperature oxidation, and hydrothermal alteration explains the apparent discordance between experimental data based on bulk rock analyses and numerical models. The findings by Smirnov et al. (2017) further emphasize the need for development of non-conventional methods of paleointensity determination that may be applied to the rocks not suitable for traditionally used methods such as the Thellier method.
Another fundamental but yet unresolved problem of Earth’s magnetism concerns the long-term evolution of geomagnetic field in the Precambrian and its link with the early Earth’s thermal evolution, especially with respect to the time of solid inner core nucleation (ICN). The age of ICN remains largely unknown today. While models based on geochemistry (e.g., Brandon et al., 2003) suggest an old inner core (>3.5 Ga), recent thermal models predict that the inner core formed much later, between 2.5 and 0.4 Ga (e.g., Aubert et al., 2010), with preference to a younger age in more recent models. Importantly, the time of ICN depends critically on core thermal conductivity, $\kappa_{\text{core}}$ (Olson, 2013). Conventional values of $\kappa_{\text{core}}$ (30–40 W m$^{-1}$ K$^{-1}$; e.g., Stacey and Loper, 2007) allowed inner core ages that extended well into Archean times (e.g., Gubbins et al., 2004). However, recent experimental and theoretical studies suggest much higher values of $\kappa_{\text{core}}$ ranging from 90 to 150 W m$^{-1}$ K$^{-1}$ (e.g., de Koker et al., 2012; Pozzo et al., 2012; Gomi et al., 2013; Gubbins et al., 2015). These values in turn suggest ages less than 1 Ga for the onset of inner core growth (e.g., Olson et al., 2015; Driscoll, 2016). Overall, the question about the value of $\kappa_{\text{core}}$ remains open, as some studies suggest lower values, consistent with classical core thermal conductivities (e.g., Zhang et al., 2015; Konôpková et al., 2016).

This uncertainty naturally raises the question of whether some paleomagnetic parameter might be able to detect independently inner core growth and, consequently, constrain core thermal conductivity values. One possible parameter is the paleointensity of the ancient geomagnetic field of the Earth ($H_{\text{anc}}$). Some models for planetary evolution proposed a sharp increase in the geomagnetic field intensity associated with the nucleation and growth of the inner core due to the start of a compositionally-driven dynamo (e.g., Stevenson, 2003). In these models, the ICN event is preceded by a very weak or no field (e.g., Gubbins et al., 2003; Buffett, 2003; Labrosse et al., 2001; Costin and Butler, 2006; Hori et al., 2010) (Figure 1.2.a, red line).

One of the recent attempts to detect such a stepwise increase of $H_{\text{anc}}$ was conducted by Biggin et al. (2015) based on a statistical analysis of the Precambrian paleointensity data selected using the criteria proposed by Biggin and Paterson (2014). The authors concluded that there was a sharp increase in the field strength in the Mesoproterozoic (at ~1.3 Ga) and
interpreted this increase as a signal from the ICN onset. They also concluded that such a relatively “old” inner core age suggested moderate values of $\kappa_{\text{core}}$.

However, Smirnov et al. (2016) demonstrated that the critical data used by Biggin et al. (2015) significantly overestimated the true paleofield strength. Smirnov et al. (2016) also showed that Biggin et al. (2015) artificially increased the statistical significance of their results by lowering the data acceptance threshold to allow more data to pass screening. The use of a stricter set of criteria leads to a significantly smaller selected dataset, precluding application of any statistical methods. Smirnov et al. (2016) concluded that the presently available paleointensity data for the Precambrian are insufficient to constrain the age of the solid inner core and, by implication, the core thermal conductivity values. Consequently, the question whether paleointensity data may be used to determine the age of the inner core remains open.

Importantly, statistically significant changes in the average level of the field strength $H_{\text{anc}}$ can in principle be associated with processes other than the inner core nucleation. For example, a numerical geodynamo model by Driscoll (2016) suggests that a transition from a multipolar to dipolar geodynamo regime in the entirely liquid core caused a four-fold increase in the Earth’s magnetic moment from $\sim2 \cdot 10^{22}$ Am$^2$ to $\sim8 \cdot 10^{22}$ Am$^2$ about 1.7 billion years ago (Figure 1.2.a; blue line). In this model, the generation of a strong field was replaced by a weak-field generation mode (with Earth’s magnetic moments $< 2 \cdot 10^{22}$ Am$^2$) about 1.0 billion years ago; this weak-field regime continued until the crystallization of the inner core about 0.65 billion years ago, which again led to an increase in the average magnetic moment to about $6 \cdot 10^{22}$ Am$^2$. Thus, the model by Driscoll (2016) “predicts” three statistically significant stepwise changes in the paleointensity average – two increases at $\sim1.7$ Ga and $\sim0.65$ Ga, and a decrease at $\sim1.0$ Ga (Figure 1.2.a).

Another scenario of the geodynamo evolution was proposed by Ziegler and Stegman (2013) who suggested that the Earth’s magnetic field was first generated by convection in the molten part of the lower mantle (the basal mantle ocean) while the entirely liquid core was in a stratified (sub-adiabatic) state, not generating a field. During that period, the field
strength was comparable with its average values in the Phanerozoic (i.e., \(\sim 7·8 \times 10^{22} \text{ Am}^2\)) (Figure 1.2.a; green line). In this model, the solidification of the basal mantle ocean occurred at about 2.5 Ga, which resulted in transition of the liquid core into a convecting (super-adiabatic) regime with generation of a weak field (with a possible period of total absence of geodynamo in the Paleoproterozoic). According to this model, the formation of the inner core occurred no earlier than 0.4 billion years ago and was marked by a transition from the weak-field regime to the modern compositionally-driven geodynamo. Thus, the model by Ziegler and Stegman (2013) “predicts” two statistically significant stepwise changes in the paleointensity average – a decrease at \(~2.5\) Ga, and an increase at \(~0.4\) Ga or later (Figure 1.2.a). A similar “two-step” behavior is also expected in the scenario of dissipation of a solid proto-core and the subsequent growth of the inner core proposed by Starchenko and Pushkarev (2013) although time constraints are not given in this model.
Figure 1.2. Predicted field modeling and the current paleointensity database. (a) Changes in the long-term averaged strength of Earth’s magnetic field predicted by the models discussed in the text. The red line shows a sharp increase in the field strength due to the inner core nucleation, ICN (the timing at ~1.3 Ga is tentatively selected and corresponds to the ICN age proposed by Biggin et al. 2015). The dashed red lines and pink box indicate the possible timing of ICN according recent models (see text). The green and blue lines show the “two-step” and “three-step” scenarios suggested by Ziegler and Stegman (2013).
and Driscoll (2016), respectively (see text). (b) The site-mean paleointensity data (dipole moment) from the global database for the 500-3500 Ma period selected by the criteria described in the text - the green circles show determinations from bulk rock samples, the red diamonds show determinations from single silicate crystals. The dashed line corresponds to the mean paleointensity value calculated from the data for the 0-10 Ma period selected using the same criteria. The open circles/diamond show the group-mean values calculated from three or more sites. The errors shown are 1σ. (c) The same data as in (b) complemented with the site-mean values for the Phanerozoic (0.05-500 Ma) (grey oblique crosses). The rectangles outlined with blue dashed lines schematically show the approximate duration and field strength corresponding to the three stages of geodynamo evolution in the model by Ziegler and Stegman (2013): 1) a relatively strong field generated in the basal mantle ocean; 2) the period of generation of a weak field in the absence of an inner core; 3) the period of generation of a strong field in the presence of the inner core (compositionally-driven geodynamo).

Analysis of the variation of paleointensity over the geological time is crucial for testing these different hypotheses (models). However, paleointensity investigations of Precambrian rocks are associated with additional difficulties. For example, many extrusive rocks that are most suitable for the Thellier method (for example, basaltic lava flow sequences) have been destroyed by erosion. Consequently, many of the determinations of $H_{anc}$ for the Precambrian have been obtained from intrusive rocks (for example, diabase dikes) which introduces additional uncertainty associated with the effects of slow cooling (e.g., Dodson and McClelland-Brown, 1980; Halgedahl et al., 1980; Brown, 1984). In addition, most Precambrian rock sequences have been affected by weathering, deformation, and/or low-grade metamorphism. In many cases, these processes led to the alteration of the magnetic minerals with attendant corruption of the paleomagnetic signal, and/or to the formation of secondary minerals (e.g., clays) that compromise the suitability of the rocks for paleointensity determinations due to experimental alteration (see Section 1.2).
Because of these problems, our knowledge of the Precambrian paleointensity remains very limited. The latest version of the global paleointensity database (http://earth.liv.ac.uk/pint/) contains 357 site-mean paleointensity determinations for the Precambrian published from 1968 to 2015, which constitutes about 8% of the entire database (although the Precambrian constitutes more than 85% of the geological history). However, even these results are characterized by a different degree of reliability. Consequently, to identify the data that most accurately reflect long-term changes in the strength of the geomagnetic field, certain selection criteria need to be applied. In this work, we used the following criteria:

1) The data are obtained by one of the variants of the Thellier method;
2) The data do not represent transitional (reversing) geomagnetic field;
3) The data are not explicitly based on secondary magnetizations;
4) The absence of magneto-mineralogical alteration is demonstrated by an alteration check (e.g., the pTRM checks). However, we also accepted the results obtained using microwave heating because the latter does not cause significant alteration of the magnetic carrier (e.g., Halls et al., 2004);
5) The site-mean paleointensity values are based on two or more samples and are characterized by a within-site scatter not exceeding 25% of the site-mean value of \( H_{\text{anc}} \).

Upon application on these criteria, 148 site-mean paleointensity determinations were selected (Figure 1.2.b). If the investigated rocks were represented by three or more sites, a group-mean value was calculated from the site-mean values (Figure 1.2.b).

The selected data do not show any sharp increase in the Mesoproterozoic that could be associated with ICN as suggested by Biggin et al. (2015). Moreover, our analysis indicates that the model by Driscoll (2016) is not supported by the existing data on the Precambrian paleointensity, which in particular do not show the predicted increase about \(~1.7\) billion years ago (Figure 1.2.b). Interestingly, our analysis of the Precambrian paleointensity data indicates the presence of relatively high paleointensity values in the Neoarchean and early Paleoproterozoic whereas the Proterozoic is characterized by low paleointensity values (also
see Shcherbakova et al., 2011) (Figure 1.2.b). We note that high values of $H_{anc}$ were also recently obtained from the Neoarchean-Paleoproterozoic dikes of Karelia (Shcherbakov et al., 2017). In addition, the paleointensity data for the entire geological history (selected with the same criteria as described above) are in good agreement with the scenario proposed by Ziegler and Stegman (2013) (Figure 1.2.c). Nevertheless, it would be premature to consider that these data, due to their insufficiency, confirm the predictions of the model. Additional high-fidelity paleointensity data are needed to further test this hypothesis.

The further advancement in understanding the Earth's magnetic field evolution during the Precambrian requires development and application of non-conventional methods that minimize or eliminate the effects of experimental alteration. Alternative approaches to reduce the effects of the experimental alteration include using microwave radiation for heating (Walton et al., 1992, 1993) and using single silicate crystals (e.g., plagioclase) instead of bulk rock samples (Cottrell and Tarduno, 2000; Tarduno et al., 2006). Although these approaches (which still use the multi-heating Thellier protocol) result in a higher success rate of paleointensity experiments, they require unique laboratory equipment and, therefore, have not been widely applied. Several alternative paleointensity methods that use a single heating or a reduced number of heatings have been proposed. These reduced-heating methods include the Shaw method (Shaw, 1974), the quasi-perpendicular method (Kono and Ueno, 1977), and the multi-specimen method (Hoffman et al., 1989; Dekkers and Böhnel, 2006). In addition, paleointensity determination methods that do not require any temperature treatment of samples have also been proposed. These methods include the anhysteretic remanent magnetization (ARM) method (Markert and Heller, 1972), the ratio of equivalent magnetization (REM) method (Cisowski et al., 1975; Gattacceca and Rochette, 2004; Acton et al., 2007), and pseudo-Thellier method (Tauxe et al., 1995). These non-heating methods as well as the reduced-heating Shaw method use an anhysteretic remanent magnetization (ARM) or a saturation isothermal remanent magnetization (SIRM) to model the natural TRM and apply alternating field (AF) demagnetization in place of thermal demagnetization.
However, application of reduced-/non-heating methods based on ARM or SIRM for absolute paleointensity determinations has been complicated by the difference in acquisition efficiencies of TRM, ARM, and SIRM (i.e., different magnetization strength acquired in the same field). The different efficiencies require that the data generated by any of such methods must be normalized by a calibration factor in order to produce an absolute paleointensity estimate. However, the calibration of the methods has been investigated only by a limited number of studies, and the currently used correction factors are characterized by large uncertainties (e.g., Yu et al., 2003; Gattacceca and Rochette, 2004; Yu, 2010; de Groot et al., 2013; Lappe et al., 2013; Lerner et al., 2017). In addition, the question of which of the non-heating methods produces more accurate and precise absolute paleointensity estimates remains controversial (e.g., Yu, 2010; Lappe et al., 2013; Lerner et al., 2017).

Because of these complications, the reduced-heating and non-heating methods have not yet been widely utilized to determine absolute paleointensity from terrestrial rocks. However, these methods represent a valuable alternative for investigating the Precambrian rocks that are frequently affected by laboratory heating-used alteration. In addition, a reduced/non-heating approach represents the only feasible choice for extraterrestrial rocks that are almost universally prone to heating-induced alteration and are limited in terms of the amount of material available for investigation (e.g., Yu, 2010; Lappe et al., 2013). Taking into account these important fields of application, additional efforts are needed to better understand the physical foundations and efficiency of reduced/non-heating methods in recovering the true paleofield strength and to better constrain their calibration factors. In order to contribute to these efforts, in this project, we conducted paleointensity experiments using the Shaw and pseudo-Thellier methods on a set of synthetic magnetite-bearing samples (Chapter 4).

In addition to paleointensity, important insights into the long-term evolution of the Precambrian geomagnetic field can also be derived from the analyses of paleosecular variation (e.g., Smirnov and Tarduno, 2004; Biggin et al., 2008). Recently, Smirnov et al. (2011) identified two Precambrian time windows (1.0-2.2 Ga and 2.2-3.0 Ga) where global
igneous units (N = 23) allowed a reliable assessment of Precambrian PSV. The data in each group were analyzed using Model G (eqn. 2). Although there may be trends on tens to 100 million year timescales (McFadden et al., 1991), if data sets represent many latitudinal belts from multiple ancient cratons, a longer-term PSV signal can be derived. A non-parametric Sign Test suggested the pre- and post-2.2 Ga data (a = 0.21 ± 0.09, b = 11.10 ± 1.46 and a = 0.22 ± 0.02, b = 7.56 ± 0.84, respectively) are different at the 78% confidence level (Figure 1.3.a). The parameters of model G fits based on the intrusive data sets only (a = 0.21 ± 0.07, b = 11.56 ± 1.43 and a = 0.20 ± 0.03, b = 7.66 ± 1.13) were statistically indistinguishable from those obtained from fitting the total data set, supporting the difference in PSV (Figure 1.3.b).

Figure 1.3. Variation of the angular dispersion for Precambrian rocks (a) Latitudinal dependence of angular dispersion S of virtual geomagnetic poles for the Precambrian intrusive and extrusive units (solid symbols) and extrusives of the last five million years (open inverted triangles). Grey and black symbols: younger and older than 2.2 Ga, respectively. Dashed, grey, and solid lines: Model G fits for the 0-5 Ma (Time-Averaged
Field Initiative, TAFI), 1.0-2.2 Ga and 2.2-3.0 Ga data, respectively. Arrows show the paleo-latitudes of the proposed study targets (notations the same as in Figure 1.1). (b) Latitudinal dependence of $S$ only for the Precambrian (solid symbols) intrusive units (i.e. without the extrusive units).

When compared with data for the last 5 million years (Johnson et al., 2008; Lawrence et al., 2009; $a = 0.22 \pm 0.03$, $b = 14.64 \pm 0.83$), the data from both time windows suggest a more dipolar field (lower equatorial intercepts). The data from the pre-2.2 Ga window is even more dipolar than that in the post-2.2 Ga interval implying that the process causing the change of PSV was operating by at least $\sim$2.2 billion years ago. If Earth without a solid inner core had a more dipolar field, and inner core growth commenced at ages younger than 1 Ga, we would not expect to see a PSV difference between the data windows we have examined. Smirnov et al. (2011) suggested that the changes in angular dispersion could be associated with the onset of crystallization of the inner core about $\sim$2.2 billion years ago. However, these changes may also reflect changes in core stratification (before the crystallization of the inner core) due to changes in the structure of the heat flux at the core-mantle boundary as a result of subduction.

However, the paleomagnetic data available for the Precambrian were generally collected for paleolatitude (tectonic) studies. The change identified by Smirnov et al. (2011) is testable through renewed paleomagnetic studies of igneous units, with an eye toward dense sampling needed to reduce uncertainties in PSV analyses. Additional efforts should also be made to obtain robust PSV estimates for the time periods for which such estimates are currently rare or absent (for example, for the early/mid-Mesoproterozoic). This study contributes to these efforts by investigating PSV from the Mesoproterozoic rocks associated with the Midcontinent Rift (Chapters 2 and 3).

1.4 Goals and questions addressed in this work

This PhD study has focused on two main objectives. The first objective is the development of an improved reduced-heating methodology for paleointensity determination that would allow expanding the range of rocks suitable for paleointensity determinations, especially
for the Precambrian. To achieve this goal, a new experimental protocol combining the Shaw method with the pseudo-Thellier non-heating method to retrieve absolute paleointensity estimations was developed (Chapter 4).

The other objective is to contribute to understanding the behavior of geomagnetic field in the Precambrian. To achieve this objective, detailed paleointensity and paleomagnetic analyses of rocks of the North-American Mid-Continent rift - the ~1100 Ma Baraga-Marquette dike swarm and the ~1094 Ma Greenstone Flow – have been conducted (Chapters 2 and 3).

Chapter 2 presents the results of a paleomagnetic and paleointensity investigation performed on the ~1.1 Ga Baraga-Marquette mafic dike swarm using both conventional paleomagnetic methods to estimate the field stability (paleosecular variation) and the new modified Shaw-pseudo-Thellier protocol to estimate the paleointensity. The results are discussed in the context of the Precambrian geomagnetic field evolution and the Earth’s thermal history.

Chapter 3 presents the detailed investigation of paleomagnetism of the Greenstone Flow, the most voluminous exposed lava flows erupted during the activity of the North-American Mid-Continent rift. Implications of these results for the geomagnetic field stability as well as for the emplacement history of the Greenstone Flow are discussed.

Chapter 4 presents the results of a detailed investigation of the calibration factor of the modified pseudo-Thellier method. The investigation was performed on synthetic magnetite-bearing and natural specimens using a modification of the Shaw method protocol designed to incorporate the pseudo-Thellier experimental protocol. Different calibration strategies are discussed and compared in terms of their efficiency to retrieve the true paleofield strength. In addition, the effect of application of multiple ARM acquisition fields during the experiment is discussed.
2 New paleomagnetic and paleointensity investigation of the Late-Mesoproterozoic mafic dikes related to the North American Midcontinent Rift in Michigan and Wisconsin

2.1 Introduction

Data on the behavior of geomagnetic field during the Precambrian are of great importance to constrain models of the Earth’s magnetic dynamo and to better understand planetary evolution. The majority of paleointensity and paleomagnetic data for the Phanerozoic have been derived from extrusive rocks (e.g., basaltic lava flows). However, most of Precambrian extrusive sequences have been affected by weathering, deformation, and/or metamorphism, hindering the preservation and measurement of paleointensity and paleomagnetic signals (see Section 1.3). Due to the rarity of well-preserved extrusive Precambrian rocks, quickly cooled shallow intrusions such as mafic dikes and sills represent an attractive alternative target for paleointensity studies. Prominent globally distributed mafic dike swarms represent the feeder systems of large igneous provinces (LIPs) (e.g., Ernst and Buchan, 1997) and often preserve pristine paleomagnetic and geochemical signatures (e.g., Halls et al., 2008). These massive magmatic provinces are characterized by large eruptive volumes > 0.1 Mkm³ emplaced within 1-50 Ma, and are often associated with critical changes and/or events throughout the Earth’s history such as continent/supercontinent breakup, mass extinctions, and climate shifts (e.g., Bryan and Ernst, 2008; Ernst and Youbi, 2017).

The ~1109-1085 Ma North American Mid-Continent Rift (MCR) is the best documented amongst the Precambrian LIPs formed within the Laurentia continent (e.g., Nicholson et al., 1997). The MCR consists of igneous rocks intercalated with sediments, forming an over 3000 km long major arc-shaped anomaly on the gravity and magnetic maps of North America, extending from Oklahoma to the Lake Superior region, and into Lower Michigan (Figure 2.1). A recent study by Stein et al. (2018) suggested that the eastern arm of the MRC extended down to Alabama. The unique curvature of the rift is inherent to its formation and was not caused by later tectonic events (Hnat et al., 2006; Kulakov et al.,
The formation of MCR has been attributed to various mechanisms from a normal extension tectonic event associated with isolated midplate volcanism, to an active rifting that took place during the Precambrian between Laurentia and Amazonia (Stein et al., 2014), to a mantle plume activity (e.g., Hutchinson et al., 1990). The rift was most likely aborted during the convergence of the proto-North American plate and the Grenville Province (Ojakangas and Dickas, 2002).

The MCR magmatism resulted in eruption of large volumes (>300,000 km³) of primarily mafic, mantle-derived magma and emplacement of possibly an equal amount of intrusive rocks (Green et al., 1987; Davis and Green, 1997; Hutchinson et al., 1990). This magmatism has been divided into several stages; a precursor stage (ca. ~1145–1140 Ma), an early (ca. 1109–1105 Ma), latent (ca. 1105–1100 Ma), main (ca. 1100–1094 Ma), and late (younger than ca. 1094 Ma) stages (e.g., Davis and Green, 1997; Heaman et al., 2007; Miller and Vervoort, 1996; Vervoort et al., 2007; Miller et al., 2013). However, at present, the MCR-related rocks are exposed only in the Lake Superior region (e.g., Nicholson and Shirey, 1990; Hollings et al., 2010). The outcropping MCR rocks are generally well preserved and yield stable remanent magnetization directions of both normal and reversed polarities (e.g., Pesonen and Halls, 1979, 1982; Swanson-Hysell et al., 2009; Kulakov et al., 2014). The paleomagnetic poles retrieved from the MCR sequences form a descending branch of a prominent loop in the North American apparent polar wander path (APWP) during the late Mesoproterozoic, the so-called Logan loop (Robertson and Fahrig, 1971). However, while the main and final stages of MCR have been represented by several high-quality paleomagnetic datasets (Swanson-Hysell et al., 2009, 2014; Tauxe and Kodama, 2009; Kulakov et al., 2013c, 2014; Fairchild et al., 2017), the paleomagnetic data for the precursor stage remain limited.
In contrast to the numerous paleomagnetic directional data, very few paleointensity determinations have been retrieved from the MCR rocks to date (Pesonen and Halls, 1983; McArdle et al., 2004; Kulakov et al., 2013a; Sprain et al., 2018). However, even some of these data have been recently shown to be biased due to the presence of non-ideal (i.e. multidomain, MD) magnetic carriers, the presence of a viscous remanent magnetization (VRM), and/or a very small number of samples (e.g., Smirnov et al., 2016).

Multiple intrusions of diabase (also known as dolerite) dike swarms are exposed in the vicinity of Lake Superior (Figure 2.1). Generally trending parallel to the axes of the rift, these swarms most likely fed the lava flows erupted during the precursor and early stages
of MCR activity (e.g., Green et al., 1987). In this study, we concentrate our investigative effort on collecting new paleomagnetic and paleointensity data from the dike swarms exposed in the Upper Peninsula of Michigan and in Central Wisconsin (Figure 2.2). Both normal and reversed directions of magnetic remanence have been identified in these dikes but despite many attempts to precisely date these intrusions, their ages remain poorly constrained. In this chapter, we will present the new results of paleomagnetic, rock magnetic and paleointensity investigations of the dikes and discuss their implications for the age of dike emplacement as well as for the behavior of the geomagnetic field during the Late Mesoproterozoic.

2.2 Geological setting

Linear aeromagnetic anomalies, some of which are several tens of kilometers long (e.g., Case and Gair, 1965; Pesonen and Halls, 1979; Anderson and Natland, 2014), indicate the presence of numerous, approximately east-west trending dikes in Baraga and Marquette counties of Michigan and extending south to the Iron River area (e.g., Gair and Thaden, 1968) (Figure 2.2). In this area, the so-called Baraga-Marquette dike swarm cuts through three metasedimentary and metavolcanic basins of Animikian age, which belong to the Marquette Range Supergroup and lie unconformably on the dominantly granitic basement of Archean age (Schneider et al., 2002; Van Schmus and Hinze, 1985). The general strike of the dikes is parallel to sub-parallel to the rift axis from N50° to N110° with sub-vertical to vertical dips (e.g., Pesonen, 1978; Pesonen and Halls, 1979). The dikes are thought to be feeders for the now eroded lava flows during the formation of the MCR (Green et al., 1987).

The dike widths vary between several centimeters and several tens of meters. They are generally poorly exposed in the field, with the exception of the Lake Superior shoreline north of Marquette and at the Sugarloaf Mountain (the Marquette area) (Figure 2.3). Relatively good outcrops are also found south of Baraga (the Baraga area) (Figure 2.4). The dikes are mostly composed of dark grey diabase (porphyritic in places) containing plagioclase and pyroxene as the major minerals with subsidiary biotite, quartz, and olivine.
Primary subhedral titanomagnetite is the main opaque mineral, with occasionally occurring ilmenite, pyrite and probably hematite (e.g., Pesonen and Halls, 1979). Secondary alteration is generally minor, but when present the common secondary minerals are chlorite, sericite, and epidote (Puffett, 1974). Aeromagnetic data and prior paleomagnetic investigations of the Baraga-Marquette swarm (e.g., Pesonen and Halls, 1979) identified normal and reverse polarities in the dikes.

Despite many geochronological investigations of the Keweenawan rocks (e.g., Davis and Paces, 1990; Davis and Green, 1997; Zartman et al., 1997), the absolute and relative ages of the Baraga-Marquette swarm remain mainly estimated from geological relationships. The dikes intrude both the Archean and Paleoproterozoic rocks exhibiting distinct chilled margins and have not been affected by the metamorphism associated with the Penokean orogeny at ~1.8 Ga (e.g., Gair and Thaden, 1968; Cannon and Klasner, 1972; Schulz and Cannon, 2007). On the other hand, the western end of the swarm does not penetrate and is overlain by the Jacobsville sandstone of a post-Keweenawan (likely, Neoproterozoic) age (Roy and Robertson, 1978; Behrend et al., 1988).
Previous paleomagnetic studies showed that the directions of remanent magnetization are consistent with a Lower Keweenawan age of the Baraga-Marquette dikes (e.g., Pesonen and Halls, 1979; Green et al., 1987). In particular, Pesonen and Halls (1979) reported reversed directions for 14 and 4 dikes from Marquette and Baraga Counties, respectively, based on both alternating field (AF) and thermal demagnetization. Hence, these dikes are older than the reversed to normal polarity transition recorded in the western Lake Superior region (Halls and Pesonen, 1982) which is now constrained to be between 1105 ± 2 Ma and 1102 ± 2 Ma (Davis and Green, 1997). In addition, the similarity of the paleopole determined from the Marquette County dikes to that obtained from the well-dated Logan
sills (DuBois, 1962; Palmer, 1970; Pesonen, 1978; Davis and Sutcliffe, 1985) was interpreted as suggesting a ~1109 Ma age.

Figure 2.3. Sampling sites in the vicinity of Marquette (circles). Red/blue/yellow fill shows reversed/normal/undetermined polarity of paleomagnetic directions. Light blue fill shows the sites that yielded sample-mean directions of mainly normal polarity but for which no site-mean direction could be calculated. The triangles/purple font indicate the approximate locations of Baraga-Marquette dikes studied by Pesonen and Halls (1979).

However, the age structure of the Baraga-Marquette swarm may be complex. Cross-cutting relationships and petrological analyses suggest that the Baraga-Marquette dikes may represent at least two pulses of dike emplacement activity (e.g., Gair and Thaden, 1968; Kantor, 1969). The dikes in Baraga County are slightly more altered than those in Marquette County and thus may belong to an older diabase type (Pesonen and Halls, 1979;
Green et al., 1987). Furthermore, some preliminary unpublished data also indicated the presence of normally magnetized dikes in the Baraga-Marquette area. These dikes are characterized by dark grey diabase, while the older reversed polarity dikes exhibit a more greenish diabase with a salmon-colored groundmass. The greater alteration in the reversely polarized dikes suggests that they are older than the normally polarized dikes.

Figure 2.4. Sampling sites in the Baraga area (circles). Red/blue/yellow fill shows reversed/normal/undetermined polarity of paleomagnetic directions. Light blue/red fill shows the sites that yielded sample-mean directions of mainly normal/reversed polarity but for which no site-mean direction could be calculated. Triangles/purple font indicate the approximate locations of Baraga-Marquette dikes studied by Pesonen and Halls (1979). The Fall River Thrust fault is shown as dashed where it is inferred. NBB, Northern Baraga Basin; BM, Baraga-Marquette; RL, Roland intrusion; BD, Boulderdash; YD, Yellow Dog Peridotite, also known as the Eagle intrusion.
In addition, the geochemical analyses show that the composition of the reversely magnetized dikes is more evolved while the younger normal dikes are more primitive (Green et al., 1987). This geochemical difference could suggest (1) a dynamic change in the rift activity during the formation of the younger dike involving a more active rifting with an increase of magmatism, or (2) a different magmatic reservoir with a less-enriched mantle source and less interaction with the continental crust (Green et al., 1987).

In this paper, we refer to the northern portion of Baraga and Marquette counties north of the Fall River Thrust (an Archean thrust fault) as northern Baraga Basin (NBB; Figure 2.1 and Figure 2.4).

The northern Baraga Basin hosts the only two precisely dated intrusions. The first intrusion is the Yellow Dog Peridotite (Figure 2.4), now known as the Eagle deposit, dated at 1107.0 ± 5.7 Ma (U-Pb baddeleyite age; Morris, 1977; Klasner et al., 1977; Ding et al., 2010). Petrochemical analysis of the Yellow Dog Peridotite and its neighbor, the East Eagle intrusion, suggests that these formations are intrusive equivalent to lava flows erupted during the early stages of continental magmatism associated with the development of the MCR (Ding et al., 2010). Ding et al. (2010) correlated these magmatic Ni-Cu-PGE deposits with other volcanic formations from the early stage of magmatism of the MCR such as the Siemens Creek volcanic suite and the Group 1 volcanic suite at Mamainse Point. Both the age and the east-west trend of long axes of the Yellow Dog and East Eagle intrusions are consistent with the inferred age and trend of the Baraga-Marquette dike swarm; however, no direct relation has been established.

The second formation (our site BAR03; Figure 2.4) precisely dated at 1120 ± 4 Ma (U-Pb baddeleyite age; Dunlop III, 2013) belongs to the so-called northern Cu-depleted dikes. At least two Cu-depleted dikes characterized by normal aeromagnetic anomalies have been identified in the northern Baraga Basin (Figure 2.4). These dikes trend WNW-ESE and are spatially related to several magmatic sulfide deposits (e.g., Yellow Dog Peridotite, East Eagle, Boulderdash, and Roland Lake). The Cu-depleted dikes display moderately to intensely altered minerals in the northern dikes and heavily to completely altered primary
minerals in the southern dikes. The altered minerals are mostly chlorite, sericite, biotite, carbonate, and serpentine, and the northern dike contain minor amounts of full serpentinized olivine phenocrysts (Dunlop II, 2013). The Cu-depleted dikes were previously associated with the Yellow Dog complex but the precise age determinations suggests that these intrusions may be unrelated (Dunlop II, 2013). However, only the upper, most likely younger, portion of the Yellow Dog intrusion was analyzed and further investigation is needed to date the middle and lower (i.e. older) parts of this dynamic system (Ding et al., 2010; Dunlop II, 2013).

The two other dike swarms we investigated, the Mellen-Gogebic and Central Wisconsin, are not well studied (Figure 2.2). Both swarms are poorly exposed in the field but are well expressed as positive and negative anomalies on the aeromagnetic maps (Green et al., 1987).

The Central Wisconsin swarm (Figure 2.2) is composed of over a hundred dikes cutting Archean and Lower Proterozoic rocks and mainly occupy pre-Keweenawan faults (Green et al., 1987; Cannon and Nicholson, 1996). Crosscutting relationships and magnetic polarity are the only means to determine the relationships between these dikes due to the lack of precise age determinations (Davis and Sutcliffe, 1985; Cannon and Nicholson, 1996). Despite the lack of direct relationship observed between the two sets of Central Wisconsin dikes, the normal polarity dikes are found cutting through older Keweenaw formation which are younger than any rocks intruded by the reversed dikes therefore the reversed polarity dikes are considered older that the normally magnetized dikes (Weiblen, 1982; Green et al., 1987). Preliminary geochemical survey also suggested that the reversed polarity dikes are characterized by a higher Fe-Ti content than the normal polarity dikes (Myers et al., 1980). Finally, Myers et al. (1980) previously identified a paleomagnetic direction similar to the Keweenaw normal direction in a dike outcropping on the shore of Lake Wissota (our site B05; Figure 2.2).

The Mellen-Gogebic swarm is comprised of roughly thirty dikes intruding Archean and Lower Proterozoic rocks, similar to the Central Wisconsin dikes (Green et al., 1987;
The Mellen-Gogebic dikes crosscut the Keweenaw formation units of both normal and reversed magnetic polarity including the Mellen granite dated at 1101 ± 1.4 Ma (U-Pb baddeleyite age; Zartman et al., 1997). Although the magnetic polarity of many dikes is not determined, the normal polarity dikes generally trend NNE or NE while the reversed dikes trend generally E to NE (Green et al., 1987; Cannon and Nicholson, 1996). The normal polarity dikes may be related to the magmatism associated with the eruption of the Portage Lake Volcanics (1095 ± 1.6 Ma, U-Pb baddeleyite age; Davis and Paces, 1990) during the main stage of evolution of the MCR (Green et al., 1987; Cannon and Nicholson, 1996).

In this chapter, we present the analysis of the paleomagnetic directions recorded by the dikes in order to isolate different directional groups and exclude the non-interpretable dikes from further analysis. Then the results of the rock magnetic experiments are presented to characterize the magnetic minerals and their capacity to preserve the information and withstand paleointensity determination experiments. Finally, the results of our paleointensity experiments using the LTD-DHT Shaw protocol are presented (c.f. 2.6 Paleointensity determinations).

### 2.3 Sampling

Samples from sixty-two sites were collected in the Baraga, Marquette, Baraga Basin, Mellen-Gogebic, and Central Wisconsin areas during several field trips from 2012 to 2014 (Table 2.1). Rose diagrams showing the dike orientations for each area are shown in Figure 2.5. Five to thirty oriented cores and/or block samples were collected from each site resulting in a total of 637 samples for paleomagnetic, rock magnetic, and paleointensity investigations (Figure 2.2; Figure 2.3; Figure 2.4; Figure 2.5; Figure 2.6; Table 2.1). Orientation was done using both magnetic and sun compasses. All experiments were performed at the Earth Magnetism Laboratory at Michigan Technological University.
Figure 2.5. Rose diagrams of the dike orientations in the study area, Baraga (a), Northern Baraga Basin (b), Marquette (c), and Central Wisconsin combined with Mellen-Gogebic (d). The light and medium grey circles are used here as reference to show the circles for two and four dikes respectively.
Table 2.1 Summary of the sites sampled for this study. Area: MG, Mellen-Gogebic; CW, Central Wisconsin; B, Baraga; BB, Northern Baraga Basin; M, Marquette. W: Dike width. An asterisk marks the sites collected (or where some of the samples were collected) to analyze the paleomagnetic direction of the host rock and/or neighbor dike.

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2.4 Measurement of paleomagnetic directions

Magnetic remanence was measured using a three-axis DC-SQUID 2G Enterprises 760-R Superconducting Rock Magnetometer housed in magnetic shielded environment with an in-line alternating-field (AF) demagnetizer. Thermal demagnetization was performed with an ASC TD-48SC thermal specimen demagnetizer with controlled nitrogen atmosphere chamber. Both AF and thermal demagnetization were carried out progressively until less than 10% of the natural remanence magnetization (NRM) remained in the specimen or until the remanence direction in the specimen became erratic. After measuring their NRM and prior to thermal or AF demagnetization, specimens were subjected to low-temperature demagnetization (LTD) by immersing them in liquid nitrogen for at least 15 minutes and warming back to room temperature in a magnetically shielded space. During LTD, larger magnetite grains are cycled through the Verwey transition (Verwey, 1939) which randomizes the viscous magnetization component they carry (Schmidt, 1993).

The magnetic remanence of a total of 614 specimens was measured. Examples of the vector component diagrams and equal area plots are shown in Figure 2.7. Principal component analysis (Kirschvink, 1980) was used to evaluate the magnetizations vectors using the best-fit line involving 5 to 18 demagnetization steps. Most of the best-fit lines included the vector plot origin but were not anchored to it. ChRM directions of both normal (N) and reversed (R) polarity were identified by straight line pointing toward the origin. The R polarity vectors used to compute a site-mean direction have an average maximum angular deviation (MAD) value of 2.8°, with values ranging from 0.6° to 7.9°, and the average MAD of the N polarity vectors is 4.3°, with values ranging from 0.3° to 14.0° (Fisher, 1953). Thirty-four out of sixty-three measured sites yielded interpretable site-mean directions (Figure 2.8 and Table 2.2). All site-mean directions were obtained using at least five samples (n ≥ 5) and have confidence circles (α95) less than 15°, with the exception of site B05 (α95 = 19.5°). The data from the remaining twenty-six sites were rejected (not included in Table 2.2) based on a large scatter of their sample-mean directions so that no common site-mean could be calculated, or because of erratic demagnetization behavior.
The accepted site-mean directions were combined with the data from Pesonen and Halls (1979) to calculate the group-mean directions using Fisher statistics (Fisher, 1953). Two sites (A5 and H4) showed site-mean directions consistent with the Marquette R-direction (Table 2.2; Figure 2.12). However, their remanence was the result of a thermal overprint from the intrusion of the BM dike swarm and therefore they were not included in the group-mean direction.

The steep reversed directions from the Baraga, Mellen-Gogebic, and Central Wisconsin sites yield a well-defined group mean of $D = 100.5^\circ$, $I = -77.7^\circ$ ($n = 12$, $\alpha_{95} = 4.6^\circ$, and $k = 90$; Figure 2.8 and Table 2.3). The group mean calculated for the R-polarity Marquette dikes was $D = 116.2^\circ$, $I = -66.8^\circ$ ($n = 25$, $\alpha_{95} = 2.9^\circ$, and $k = 104$; Table 2.3 and Figure 2.9). These group-mean directions of R-polarity do not share a common mean at the 99.98% confidence level (McFadden and McElhinny, 1990).

The site-mean N-polarity directions obtained from the Marquette dikes have consistently steep inclinations but are characterized by a larger scatter than the R-polarity directions (Table 2.3). The corresponding group-mean for the N-polarized Marquette dikes ($D = 282.6^\circ$, $I = 86.3^\circ$, $n = 7$, $\alpha_{95} = 9.3^\circ$, and $k = 43$; Table 2.3 and Figure 2.10) is statistically different from the inverted group-mean of the R-polarized dikes from the same area. Finally, the normal polarity site-means from the Baraga, Baraga Basin, Mellen-Gogebic, and Central Wisconsin areas show intermediate to shallow inclinations with NW to W declinations (Figure 2.11; Table 2.3). However, the lack of well-defined grouping and the insufficient number of samples prevent us from identifying any additional group-means.

Analysis of the variation of the magnetic direction between cross-cutting dike, the intruded host rock and/or neighboring dikes can help assess whether the ChRM carried by the dikes is of primary origin. Examples of these relationships are shown in Figure 2.12. Two (A2 and B25) out of the eleven BCT did not yield interpretable data. As mentioned before, the site A5 and H4 possess remanence direction consistent with a remagnetization by the intrusion of the R-polarity Marquette dikes (A6, and H3, respectively; e.g., Figure 2.12.a). The host-rock and/or cross-cutting dikes of four dikes such as B16 (Figure 2.12.b) suggest
that the steep R-polarity in Marquette have a primary remanence directions. Two sites (LM-N, Figure 2.12.c, and B05, Figure 2.12.d) presents evidence for the moderate N-polarity and three sites (B18, A11, L2) for the steep N-polarity to be primary magnetization. The dike K3 characterized by a shallow N-polarity appears to hold a primary magnetization while the host rock displays specimen-means consistent with the steep N-polarity site-mean isolated for the site J3.
Figure 2.6. Annotated photos of the Baraga and Marquette dikes. a) and b) N-polarity dikes A11 and A7 on the shore of Lake Superior in Marquette, c) A positive baked contact test site, where a ~20 m wide R-polarity dike B16-A with chilled margin has baked the older B16-B dike, d) A10 intruded in the Archean granite (A) in the Sugar Loaf area, Marquette. e). Contact (red hatched line) between the Archean granite (A) and the reversely magnetized E-W trending dike (D) L1 in the Sugar Loaf area, Marquette.
Figure 2.7. Examples of paleomagnetic results. a. to f. Vector component diagrams and equal area plots showing representative examples of characteristic remanent magnetization (ChRM) results from the Baraga-Marquette dikes. D: Declination, I: Inclination, MAD: Maximum angular deviation, Range: the coercivity window selected for the best-fit.
Table 2.2. Paleomagnetic results. n/N, the number of samples used for the site mean/ the total number of specimen demagnetized; Dec/Inc, site mean declination and inclination; α₀⁵, 95% confidence interval of the estimated site mean direction, assuming a Fisherian distribution; k/K, best estimate of (Fisher) precision parameter for the site mean/virtual geomagnetic pole (VGP); dp, semi-axis of the confidence ellipse (degrees) along a site to pole great circle; dm, Semi-axis of confidence ellipse (degrees) perpendicular to the great circle. #, not included in the group-mean calculation. †, Included in the n = 4 group-mean calculation for the normal dikes. A, area: M, Marquette, B, Baraga, MG, Mellen-Gogebic, CW, Central Wisconsin.

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Potentially remagnetized sites excluded from the group-mean calculation

| A5#  | 5/8  | 133.5 | -54.8 | 12.8 | 29      | 54       | 181 | 13 | 18 | 19 | M |
| H4#  | 10/10 | 126.5 | -64.6 | 4.7  | 95      | 54       | 203 | 6  | 8  | 45 | M |

52
Figure 2.8. Equal-area plot with the reversed site-mean paleomagnetic directions from the Baraga (red circles), Mellen-Gogebic (red diamonds), and Central Wisconsin (red squares), and from Pesonen and Halls (1979) (red triangles). The star shows the group-mean with the 95% confidence limit. $D_m$, $I_m$: mean declination and inclination, Plat/Plong: latitude and longitude of the virtual geomagnetic pole (VGP), $\alpha_{95}$/$A_{95}$: radius of confidence for paleomagnetic direction / VGP, $k$: precision parameter, $n$: number of dikes included in the mean calculation and $S$: angular dispersion of VGPs.
Figure 2.9. Equal-area plot with the reversed site-mean paleomagnetic directions from the Marquette cites (red circles), from Pesonen and Halls (1979) (red triangles), and from J. Diehl (personal communication; red hourglasses). The star shows the group-mean with the 95% confidence limit. $D_m$, $I_m$: mean declination and inclination, $Plat/Plong$: latitude and longitude of the virtual geomagnetic pole (VGP), $a_{95}/A_{95}$: radius of confidence for paleomagnetic direction / VGP, $k$: precision parameter, $n$: number of dikes included in the mean calculation and $S$: angular dispersion of VGPs.
Figure 2.10. Equal-area plot with the normal site-mean paleomagnetic directions from the Northern Baraga Basin (pentagons), Baraga (inverted triangles), Marquette (circles), Central-Wisconsin (squares), and Mellen-Gogebic (diamonds) dikes from this study. The asterisks/number signs show site-means used to calculate the Mn7/Mn4 group-mean direction. The black star and square show the group-mean with the 95% confidence limit. Dm, Im: mean declination and inclination, Plat/Plong: latitude and longitude of the virtual geomagnetic pole (VGP), α95/A95: radius of confidence for paleomagnetic direction / VGP, k: precision parameter, n: number of dikes included in the mean calculation and S: angular dispersion of VGPs.
Table 2.3. The group-mean directions and paleomagnetic poles from the Mesoproterozoic dikes. The group-mean compiled with data from this study (a) and Pesonen and Halls, 1979 (b).

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**Baraga + MG + CW Reversed (a & b)**

12/13  100.5  -77.7  4.6  90  8.6  46  238  8.2  29  15.1  6.6

**Marquette Reversed (a & b)**

25/25  116.2  -66.8  2.9  104  7.9  49  212  4.2  48  11.7  5.1

**Marquette + BB Normal (a)**

4/12  311.0  79.7  6.7  189  5.9  60  245  57  59  10.6  9.8

7/12  282.6  86.3  9.3  43  12.4  57  262  29  12  23.3  5.2
Figure 2.11. Summary of the paleomagnetic results. Red/blue/black/white symbols correspond to reversed (R)/normal (N) polarity/mean directions and the surrounding circles represent the 95% confidence associated with the direction. The group-mean and site-means for the normal lamprophyre dikes from Piispa et al. (2018) are also shown.
Figure 2.12. Equal-area plots showing examples of relationships between paleomagnetic results from dike, host-rocks, and/or cross-cutting dikes investigated in this study. Red/blue represent up/down directions. Stars and associated circles indicate the means associated with a site (Table 2.2). In general, square symbols show the sample means of the dike or crosscutting dike, circles/diamonds indicate sample-means from a crosscut dike/host rock.
2.5 Magnetic mineralogy

Rock magnetic experiments were performed on the samples from each site collected. Anisotropy of magnetic susceptibility and thermomagnetic experiments were conducted using an AGICO MFK1-FA Kappabridge. Magnetic hysteresis parameters and first-order reversal curves (FORC) were estimated using a Model 2900 Princeton Measurement Corporation Alternating Gradient Field Magnetometer.

A total of 106 thermomagnetic analyses (low-field magnetic susceptibility versus temperature) were conducted from -195°C to 700°C in argon atmosphere (from 1 to 6 specimens per site). The measurements revealed three types of variations of low-field susceptibility with heating (Figure 2.13). The type 1 features a low-Ti titanomagnetite and/or magnetite as the main magnetic carrier (as indicated by the Verwey transition at ~153°C and Curie temperature ~580°C), with the presence in some dikes of titanomagnetite with a higher Ti content (a minor bump at ~400°C on the heating leg). The type 2 is very similar except for the presence of a second magnetic mineral with the Curie temperature above 600°C. This susceptibility feature is reversible and less pronounced suggesting the presence of titanomaghemite most likely formed due to low-temperature oxidation of titanomagnetite from weathering at ambient temperature, also known as maghemitization (Dunlop and Özdemir, 2001). Finally, the type 3 is characterized by the presence of low-Ti titanomagnetite and magnetite together with a well-defined hematite signature (reversible bump ~700°C). The type 3 is primarily distinguishable by the irreversible thermomagnetic curves; the cooling curve and second low-temperature curve indicate formation of Ti-content titanomagnetite and a subdued to absent Verwey transition indicating the oxidation or conversion of magnetite. This type is primarily found in the dikes for which the paleomagnetic directions were not interpretable. The interpretable dikes are predominantly type 1 and 2, only one site (C2) is of type 3.

The comparison of the weak-field susceptibility and natural remanent magnetization (NRM) intensity shows different behavior according to the dike polarity (Figure 2.14). While the normal polarity dikes show a proportional increase of susceptibility with increase
of NRM intensity, many reversed polarity dikes are grouped above susceptibility values of $6 \times 10^{-3}$ SI consistent with the presence of low-Ti titanomagnetite/magnetite. The reversed polarity specimen plotting below that threshold originate either from a dike crosscutting the BM dikes (A6), specimens with pyrite/specular hematite (half of B14), or baked sedimentary rocks (H4). On average, the Koenigsberger ratio (Q-value) from the interpretable dikes are constrained between 10 and 100. No noticeable difference between the dikes from Baraga and Marquette areas have been observed. Interestingly, the comparison of the mean alteration indice on the thermomagnetic curves (Hrouda, 2003) and the Koenigsberger Q-factor shows the BM reversed polarity dikes generally well clustered (type 1 and type 2) in comparison to the normal and not interpretable dikes (Figure 2.15). The analysis of reflected light images shows that most of the reversed polarity dikes display relatively unaltered minerals such as homogeneous titanomagnetite grains, with skeletal shapes, with ilmenite exsolution lamellae, and some pyrite grains (Figure 2.19). In contrast, the dikes carrying normal and/or secondary remanence contain altered titanomagnetite and hematite minerals (Figure 2.20).
Figure 2.13. Examples of the three main types of thermomagnetic curves from the dikes.

Figure 2.14. Weak-field magnetic susceptibility versus natural remanent magnetization (NRM) intensity plot for the normal, reserved (a) and not interpretable (b) dikes. Each plot is sorted by paleomagnetic results and locations.
Figure 2.15. Comparison of the stability and alteration of the analyzed dikes. The mean alteration indice corresponds to the average difference in susceptibility between heating and cooling susceptibilities on the thermomagnetic curves (Hrouda, 2003). R, N, N/A, reversed/normal/undetermined polarity of paleomagnetic directions.

Figure 2.16. Day plot (Day et al., 1977) of the studied dikes sorted by paleomagnetic results and locations. Grey dashed lines show SD-MD mixing models from Dunlop (2002).
Saturation remanent magnetization ($M_{RS}$), saturation magnetization ($M_s$), coercive force and coercivity of remanence ($H_C$ and $H_{CR}$, respectively).

The analysis of the hysteresis parameters reveals that most reversed dikes are composed of SD to PSD grains and plot on the Day plot between 0 % to 60 % on the SD-MD mixing curves (Figure 2.16, Dunlop, 2002). Few reversed dikes and the normal polarity dikes fall within few PSD-MD domains on the Day plot (Figure 2.13, Day, 1977). The shape of the magnetic hysteresis suggest a mixture of magnetic grain (e.g. “goose-neck” curve) and the identification of hematite on the thermomagnetic curves suggest that the deviation from the SD field could come from the presence of a hematite component (Figure 2.13, Figure 2.16, and Figure 2.17). The first-order reversal curves (FORC) are consistent with the magnetic hysteresis results; the magnetic mineralogy of the dikes is dominated by single to pseudo-single domain grains (Figure 2.18). The analysis of the FORC parameters suggest these specimens are suitable for paleointensity experiments (Carvallo et al., 2006).

In summary, the magnetic properties of the dikes are dominated by SD-PSD Ti-poor titanomagnetite with minor amounts of larger PSD-MD grains. Overall, the reversed polarity dikes show similar rock magnetic properties throughout the swarm, with better preserved magnetic grains with minor amount of low-temperature oxidation. In contrast, both the normal polarity and not interpretable dikes contain hematite and exhibit PSD-MD behaviors.
Figure 2.17. Examples of magnetic hysteresis loops and backfield remanence demagnetization curves from samples with different thermomagnetic behavior: Type 1 - B16A (a), type 2 - H3A (c), type 3 - C2A-1 (reversed polarity, b) and A8D (normal polarity, d).
Figure 2.18. Examples of FORC distributions measured at room temperature from the Baraga-Marquette dikes. Insets show the smoothing factor (SF), the full width at half maximum (FWHM), spread of the FORC distribution along the Hc axis (Width), and the bulk coercitive force (Hc).

**a.**

- FWHM = 0.03 mT
- Width = 0.12 mT
- Hc = 43.13 mT

**b.**

- FWHM = 0.05 mT
- Width = 0.14 mT
- Hc = 33.60 mT

**c.**

- FWHM = 0.05 mT
- Width = 0.11 mT
- Hc = 21.49 mT

**d.**

- FWHM = 0.04 mT
- Width = 0.07 mT
- Hc = 18.87 mT
Figure 2.19. Representative reflected light images of the Baraga-Marquette dikes with a primary reversed direction. (a, b, c, d) Titanomagnetite grains with ilmenite exsolution lamellae; (e) Complex type skeletal titanomagnetite crystals with non-orthogonal cross-arms; (f) A homogeneous titanomagnetite grain with pyrrhotite grains.
Figure 2.20. Alteration features in dikes carrying a primary and/or secondary normal remanence. a) Hematite radiance, b), c) and d) examples of altered titanomagnetites.
2.6 Paleointensity determinations

A total of thirteen R-polarity dikes (four sites from Baraga and nine from Marquette) were selected for paleointensity and subjected to the Shaw protocol (Shaw, 1974). This method allows for the retrieval of absolute paleointensity determinations by comparison of the AF demagnetization of the NRM with AF demagnetization of a laboratory induced thermoremanent magnetization (TRM). In its most recent version, the LTD-DHT Shaw method involves imparting two full laboratory TRMs (i.e., DHT, double heating technique) and the use of low-temperature demagnetization (i.e. LTD) to limit the contribution from non-ideal minerals (Shaw, 1974; Tsunakawa and Shaw, 1994; Tsunakawa et al., 1997; Yamamoto et al., 2003; Yamamoto and Tsunakawa, 2005). In addition, after each AF demagnetization of the NRM and TRMs, an anhysteretic remanent magnetization (ARM) is imparted and subsequently AF demagnetized. The three resulting ARMs (i.e. ARM₀, ARM₁, ARM₂) are used to estimate and correct the TRM value for possible changes in the magnetic mineralogy after the heating (Equation 2.1; Rolph and Shaw, 1985). Another monitoring of potential alteration upon heating is performed by comparing the first TRM (TRM₁) to the second TRM (TRM₂). The paleointensity determination is obtained using the slopes of the linear relations between NRM-TRM₁* (a) and between ARM₀-ARM₁ (b), and the laboratory field (F_lab; Equation 2.2).

Equation 2.1  \[ TRM_X^* = \frac{ARM_{X-1}}{ARM_X} TRM_X \]

Equation 2.2  \[ F_{ANC} = \frac{a}{b} F_{lab} \]

The LTD-DHT Shaw method was applied to 73 specimens using different laboratory fields (from 30 µT to 50 µT), and different ARM DC bias field (40, 50, 60, 80, and 100 µT). We note that the Shaw method is usually performed using only one F_lab field and one ARM field. We applied multiple fields to be able to use the data for the investigation detailed in Chapter 4. This approach, however, does not affect the outcome of the Shaw determination.

The laboratory TRMs were imparted along the specimen’s z-axis by heating the samples to 610°C with a hold at maximum temperatures of 10 min and 20 min for TRM₁ and TRM₂,
respectively. The samples underwent LTD (c.f. the section Measurements of paleomagnetic direction) after each measurement of the NRM, TRMs, and ARMs, and prior to AF demagnetization. Progressive AF demagnetization was conducted in 5, 10, or 20 mT steps up to 80 mT or 100 mT. The same AF demagnetization sequence was used throughout the experiment for the same specimen, but varied from one specimen to another. The ARMs were imparted at the peak AF demagnetization field (e.g., 80 or 100 mT) along the z-axis using the in-line ARM unit. The data from TRM$_1$ and TRM$_2$ were reduced by first correcting the data as outlined by Rolph and Shaw (1985; Equation 2.1). The data was then evaluated to satisfy the following criteria used in previous studies (e.g., Yamamoto et al., 2003; Mochizuki et al., 2004; Oishi et al., 2005; Yamamoto and Tsunakawa, 2005; Mochizuki et al., 2006; Yamamoto et al., 2007):

- the AF demagnetization of the NRM must reveal a stable primary component of the remanence;
- a linear portion should exist in the NRM-TRM$_1$* diagram which is not less than 15% of the original NRM intensity ($f_N \geq 0.15$), and the correlation coefficient should be larger than 0.995 ($r_N \geq 0.995$);
- a linear portion ($f_T \geq 0.15$ and $r_T \geq 0.995$) should also exist in the TRM$_1$-TRM$_2$* diagram. The slope must be unity within experimental errors ($1.05 \geq \text{slope}_T \geq 0.95$) as proof of the validity of the ARM correction.

These criteria were satisfied for 64 of the 73 specimens. Examples of accepted and rejected experiments are shown in Figure 2.21 and Figure 2.22, respectively, and a summary of the successful experiments can be found in Table 2.4. The paleointensity determinations from the successful specimens range from $5.1 \pm 0.72$ µT to $57.0 \pm 10.1$ µT. Unsuccessful specimens were rejected based on insufficient linearity of their ARM$_n$-ARM$_{n+1}$ plots (i.e. slope$_{A1}/$slope$_{A2} < 0.7$) and/or a negative double-heating check (slope$_T$ value < 0.95 or > 1.05). The specimens loose between 18 and 70% (32% in average) of the ARM intensity upon LTD, which suggests a medium to high contribution of MD grains in our specimens. This observation is consistent with the results of magnetic hysteresis measurements (Figure 2.16).
Figure 2.21. Example of a successful result from the LTD-DHT Shaw dataset for a natural specimen. The top three diagrams illustrate the results from the first laboratory heating (TRM1) while the bottom diagrams are for the second heating. Linear portions consist of closed symbols. Orthogonal vector plots of AF demagnetization of NRM are also shown as insets, where closed and open symbols indicate projections onto horizontal and vertical planes, respectively (square insets in NRM-TRM1* plots are NRM before LTD). Units are in $10^{-5}$ Am$^2$/kg.
Figure 2.22. Example of an unsuccessful result from the LTD-DHT Shaw dataset for a natural specimen. The top three diagrams illustrate results from the first laboratory heating while the bottom one is from the second heating. Linear portions consist of closed symbols. Orthogonal vector plots of AF demagnetization of NRM are also shown as insets, where closed and open symbols indicate projections onto horizontal and vertical planes, respectively. Units are in $10^{-7}$ Am$^2$/kg.
Table 2.4. Successful LTD-DHT Shaw paleointensity determinations from the R-polarity BM dikes. $F_A/F_T$, ARM bias/TRM field imparted on the specimen (in $\mu$T); NRM$_0$, ARM$_0$, intensities after LTD ($\times10^{-5}$ Am$^2$/kg); LTD, LTD fraction of ARM (in %); $H_L$, the lowest coercivity force taken for the linear segments (mT); A1 and A2, slopes of the ARM spectra in the ARM$_0$-ARM$_1$ and ARM$_1$-ARM$_2$ diagrams; N and T, slopes of the linear segments in the NRM-TRM$_1$* and TRM$_1$-TRM$_2$* diagrams; $f_N$, $f_T$, NRM and TRM$_1$ fractions of the linear NRM-TRM$_1$* and TRM$_1$-TRM$_2$* segments; $r_N$, $r_T$, correlation coefficients of the linear NRM-TRM$_1$* and TRM$_1$-TRM$_2$* segments, $F \pm \sigma_F$, calculated paleointensity and standard deviation (in $\mu$T).

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<th>ARM$_0$</th>
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<th>$H_L$</th>
<th>A1</th>
<th>n</th>
<th>$f_N$</th>
<th>r$_N$</th>
<th>A2</th>
<th>t</th>
<th>$f_T$</th>
<th>r$_T$</th>
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<th>second heating</th>
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<td>0.97 1.03 0.98 0.96 21.1 ± 1.6</td>
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<td>50 0.94 0.68 0.71 0.93</td>
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2.7 Discussion

2.7.1 Paleosecular variation

The analysis of the interpretable paleomagnetic data from thirty-two dikes showed the presence of three independent groups of paleomagnetic directions; two reversed and one normal polarities. The two better defined groups correspond to two R-polarities found for the dikes of Marquette and Baraga (including two dikes from the Mellen-Gogebic and Central Wisconsin). The analysis of the host rock performed on the Marquette and Baraga dikes supports the primary nature of their R-polarity ChRM (Figure 2.12.a to c). Hematite overprinting was shown to be responsible for the acquisition of secondary and/or viscous remanence in some MCR rocks (e.g., Ernst and Buchan, 1993; Costanzo-Alvarez et al., 1993; Borradaile and Middleton, 2006). Based on the lack of hematite and the good preservation of magnetic minerals in the R-polarity dikes, we consider their remanence to be primary. Both R-polarity group-mean directions do not share a common mean at the 99.98% confidence level (McFadden and McElhinny, 1990). Consequently, the paleomagnetic results support the previous field and petrological analyses suggesting that the dikes from Baraga and Marquette areas were intruded in two different pulses (e.g., Gair and Thaden, 1968; Kantor, 1969). Alternatively, tectonic tilting or remagnetization could explain the variation in the paleomagnetic direction, however, no geological evidence to support these events has been identified. The corresponding group-mean virtual geomagnetic poles (VGP) for the R-polarity data from Baraga and Marquette county are located at $P_{lat} = 45.6^\circ N$, $P_{long} = 237.8^\circ E$ ($A_{95} = 8.2^\circ$, $S = 15.1^\circ$) and $P_{lat} = 48.6^\circ N$, $P_{long} = 211.6^\circ E$ (with $A_{95} = 4.2^\circ$, $S = 11.7$), respectively (Figure 2.8, Figure 2.9, Table 2.3).

The seven steep normal polarity group-mean identified is composed of site-mean directions from Marquette. The resulting VGP is at $P_{lat} = 48.0^\circ N$, $P_{long} = 262.3^\circ E$ ($A_{95} = 18^\circ$, $S = 22.1^\circ$) (Figure 2.10; Table 2.3). The group-means computed for the Marquette N-polarity dikes is not very well-defined but can be improved if only four of the normal site-means are selected (i.e. $Mn4$ with $\alpha_{95}$ from 12.4° to 5.9° and $k$ from 43 and 189), and their magnetic minerals are more altered and generally composed of less ideal magnetic grains.
(i.e. PSD-MD). Nevertheless several baked-contact test suggests that these dikes hold a primary magnetization (Figure 2.12).

The analysis of the scatter of the VGP (S value) allows to estimate if the paleosecular variation of the magnetic field was properly averaged in the group-means (see Section 1.3). The calculated S values for each group were compared with the expected scatter for the 1.0 Ga to 2.2 Ga period estimated by Smirnov et al. (2011) (Figure 2.23). The R-polarity group-means from Baraga and Marquette and the steep N-polarity direction are close to the expected VGP angular dispersion for their respective paleolatitude (Figure 2.23). We note however that the S value for the normally polarized dikes is based on a limited number of data points.

Figure 2.23. Angular dispersion of the VGP obtained in this study compared to the paleosecular variation (PSV) model fitted for 1.0 to 2.2 Ga and corresponding standard deviation (dashed and dotted lines, Smirnov et al., 2011). The black dots shows the angular dispersion and associated standard deviation used by Smirnov et al., (2011) to compute the
PSV model. Mn4/Mn7 correspond to the Marquette normal VGPs with n = 4 and n = 7 (Table 2.3).

2.7.2 Intrusion and age of remanence

The VGPs retrieved from the three directional groups plot within the Logan loop along the paleomagnetic poles calculated for other MCR formations (Figure 2.24 and Figure 2.25). Their positions on the apparent polar wander path (APWP) along with the field observations and the available literature provides us with the means to estimate the relative order of dike intrusion and to evaluate the age at which they have acquired their remanence, i.e. a "paleomagnetic age".

The Baraga/Mellen-Gogebic/Central Wisconsin dikes and Marquette dikes represent two different emplacement episodes. The "paleomagnetic" age of the Marquette dikes (i.e. based on their position of the pole on the APWP) is likely to be around 1108-1105 Ma. This observation is supported by the two paleomagnetic group-means extracted from the Yellow Dog Peridotite (Figure 2.4). The two reversely polarized group-means (J. Diehl, personal communication) are consistent with the reversed polarity group-means extracted from the Marquette dikes (Figure 2.9). As previously mentioned, the Yellow Dog Peridotite was dated 1107 ± 5.7 Ma (U-Pb baddeleyite; Ding et al., 2010) which is consistent with the paleomagnetic age inferred for the Marquette dikes.

The paleomagnetic pole calculated from the reversed dikes of the Baraga area plots closer to the apex of the Logan loop and is indistinguishable from the ~1160 Ma pole from the Eriksfjord Group red beds (Piper et al., 1999) (Figure 2.25). The pole plots southeast from the ~1142 Ma paleomagnetic pole obtained from the lamprophyre and Abitibi dikes (Piispa et al., 2018) indicating the paleomagnetic age between 1142 and 1160 Ma. This age estimate is consistent with the more altered mineralogy of the dikes in the Baraga area.
Figure 2.24. Analysis of the Virtual Geomagnetic Poles (VGP) from this study on the Logan loop. The group-mean VGP poles of our dikes (open stars) with selected poles from the MCR sequences. Open/red and closed/blue symbols represent reversed and normal polarities, respectively. The mean VGP poles calculated for the Ontario lamprophyre dikes (OL; Püispa et al., 2018), for the Eriksfjord Group red beds (ER; Piper et al., 1999), the lower reversed section (MPlr1 and MPlr2), the lower normal section (MPLn), the upper reversed section (MPUr), and the upper normal (MPUn) section of the Mamainse Point lava flow sequence (Swanson-Hysell et al., 2009) are shown. The other poles are from the Powder Mill basalts (PM; Palmer and Halls, 1986), the Lower and Upper Osler Volcanics (OSr and OSn; Halls, 1974); the Lower North Shore Volcanics (NS; Halls and Pesonen, 1982), the Portage Lake Volcanics (PLV; Halls and Pesonen, 1982); the Lake Shore Traps (LST; Kulakov et al., 2013) and the Coldwell Complex (CCr and CCn; Kulakov, 2014).
The steep N-polarity group-mean pole from the Marquette dikes plots east of the reversed Baraga pole, which taken at face value suggests they are older (Figure 2.24; Figure 2.25). However, because of the small number of site-means, the pole may not represent the time-averaged field and therefore the N-polarized Marquette dikes can in fact be similar in age to the R-polarized Baraga dikes.

Unfortunately, no additional group-means could be isolated from the remaining seven normal polarity directions. However, we can make additional remarks on the sites K3 and J3 were previously identified as a dual-polarity dike informally called Mike’s dike (Diehl, personal communication). Resampling at these location revealed that instead of normal and reversed polarities, two normal polarities were identified, one shallow and one steep. The shallow mean extracted from K3 appears to be similar to the one extracted from the site (BAR03) where the Northern Cu-depleted dikes was recently dated 1120 ± 4 Ma (U-Pb baddeleyite; Dunlop Iii, 2013). The steep mean from J3 was included into our Marquette steep normal group-mean that were associated with the ~1143 Ma Northern Ontario lamprophyre dikes. The specimens taken for the baked-contact test from the host rock of K3 yielded directions consistent with the J3 group-mean. The ‘paleomagnetic’ age and baked-contact test suggests that this location of the Northern Baraga Basin has experienced at least two intrusions stages; first J3 during the early stages of the MCR magmatism then K3 during the formation of the Cu-depleted dikes.
Figure 2.25. Relative position of the VGP obtained in this study with respect to the Late Mesoproterozoic APWP. Grey and colored hexagons correspond to the mean VGP forming the “Logan” loop compiled by Piispa, (2015) and Piispa et al. (2018). The color scheme represents the estimated ages for the VGP. Note that the VGP Mn7 is not visible on this projection.
2.7.3 Paleointensity and the geodynamo

The summary of the statistical results obtained from our successful LTD-DHT Shaw determinations is shown in Table 2.5 and Figure 2.26. The paleointensity estimates from the Marquette and Baraga dikes show a dominance of low intensity values resulting in an average VDM of similar value to those from the Cordova gabbro A and Nova Floresta Gabbro, respectively (Figure 2.27; Yu and Dunlop, 2002 Celino et al., 2007). The analysis of the standard deviation relative to the mean can provide us with the precision of a group-mean. It was shown on historic lava flows that a variability above 10% of the mean might indicate incorrect data (Yamamoto et al., 2003; Oishi et al., 2005; Mochizuki et al., 2004). The standard deviation of the Baraga and Marquette dikes are 34% and 41% of their respective means which cannot be accepted. Four sites (A1, A10, H1 and H3) from the Marquette area show intensities that are statistically distinguishable from the other five dikes. The two means resulting from the separation of the Marquette dike results yield values of 13.6 ± 1.5 µT (group 1) and 28.8 ± 4.8 µT (group 2) which are not only statistically distinguishable but also possess acceptable variability of 11% and 17%. The corresponding VDM for the higher intensity dikes (group 2; Figure 2.27) is equivalent to the VDM retrieved from the Lake Shore Traps (Kulakov et al., 2013) or the Tudor Gabbro (Yu and Dunlop, 2001; Dunlop and Yu, 2004). The lower VDM (group 1; Figure 2.27) is comparable to the VDMs from the Nova Floresta Gabbro (Celino et al., 2007), and the Abitibi dikes (Macouin et al., 2003). These results may indicate that groups 1 and 2 of the R-polarity dike from Marquette record different periods of the geomagnetic field variation. These results also reveal that, at the time of formation of the group 2 dikes in the Precambrian, the Earth’s magnetic field strength was comparable to its recent values. Consequently, our paleointensity results could support the presence of a stable compositionally-driven geodynamo operating during the Late Mesoproterozoic.

However, the dikes from group 1 and 2 are very similar in their magnetic hysteresis properties and the paleomagnetic group-mean calculated for them are statistically indistinguishable. There is no correlation between the paleointensity and dike widths (varying from 0.2 m to 80.0 m for the group 1 and from 0.2 m-70.0 m for the group 2) although a slight difference in the dike trends can be observed (72°-90° for the group 1
with 90°-123° for the group 2). The most noticeable difference is found in the thermomagnetic behavior; the higher intensity dikes (group 2/G2) belong to the thermomagnetic type 2 and the lower intensities (group 1/G1) to the type 1. Consequently, the higher intensities correspond to the dikes affected by low-temperature oxidation which may result in an overestimation of their paleointensity estimates. Maghemitization of SD titanomagnetite may be expressed by a decrease of value of the Koenigsberger ratio, however this effect is not always noticeable for small PSD grains (Prévot et al., 1981). Consequently, even though our results show no difference between the Q values for type 2 and type 1 dikes (Figure 2.14), we cannot fully disregard the possibility that the higher intensity retrieved from the group 2 dikes may have been overestimated.

Figure 2.26. Analysis of the mean paleointensity and mean Virtual Dipole Moment (VDM).

a. Summary of the mean paleointensity determinations for the Baraga and Marquette
reversed polarity dikes. b. Comparison of the mean VDMs calculated for the Baraga and Marquette dikes. The mean (full line) and corresponding standard deviation (dashed lines) are shown for each grouping shown in Table 2.5.

Table 2.5. Results of the LTD-DHT Shaw experiments. \( n/n_F \), the number of specimen subjected to the LTD-DHT Shaw protocol/passing the selection criteria; NRM\(_0\) and ARM\(_0\), NRM and ARM intensities after LTD during the first cycle of the experiment (x 10\(^{-5}\) Am\(^2\)/kg); LTD, fraction of the ARM\(_0\) removed by the LTD (%); F and VADM, the paleointensity estimate (\(\mu\)T) and virtual axial dipole moment (x10\(^{22}\) Am\(^2\)) accompanied with their standard deviation.

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**Mean Baraga** 4 / 4 14.4 ± 4.9 2.0 ± 0.6

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<td>2.0 ± 0.9</td>
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**Mean Marquette** 9 / 9 20.4 ± 8.3 3.3 ± 1.5

Mean Group 1 5 / 5 13.6 ± 1.5 2.2 ± 0.2

Mean Group 2 4 / 4 28.8 ± 4.8 4.7 ± 1.1

83
Figure 2.27. Comparison of the VDM values from our study (red squares and a diamond) with other Precambrian intensity determinations. B and M, represent the overall average for the R-polarity dikes from Baraga and Marquette, respectively, G1/G2/M, group mean (M) and refined means (G1, group 1, and G2, group 2) for the Marquette dikes as explained in the text and shown in Table 2.5, Figure 2.26. ON: the microwave paleointensity from Keweenawan intrusions in Ontario (McArdle et al., 2004). CGA and CGB: Cordova Gabbro, components A and B (Yu and Dunlop, 2002); LST: Lake Shore Traps (Kulakov et al., 2013b); AD: Abitibi dikes (Macouin et al., 2003); NF: Nova Floresta Gabbro (Celino et al., 2007); TD: Tudor Gabbro (Yu and Dunlop, 2001; Dunlop and Yu, 2004); MD: Mackenzie dikes (Macouin et al., 2006); MP/SI: Mamainse Point and Simpson Island lava flows (Sprain et al., 2018). Figure modified from Kulakov et al., 2013b.
2.8 Conclusions

We have analyzed sixty-two mafic MCR-related dikes from the Upper Peninsula of Michigan and Central Wisconsin. Thirty-six of these dikes yielded interpretable paleomagnetic directions of both normal and reversed polarity. The magnetic mineralogy of the reversed polarity dikes is mainly represented by well-preserved SD-PSD low-Ti titanomagnetite grains, with some dikes affected by low-temperature oxidation. The normal polarity dikes are susceptible to heating-induced alteration, and contain more altered magnetic minerals, typically PSD, including low-Ti titanomagnetite, hematite, and pyrite. Three independent paleomagnetic group-means were identified; two reversed polarity groups from the Baraga (combined with Mellen-Gogebic and Central Wisconsin dikes) and Marquette counties, and one steep normal polarity group mostly from the Marquette shoreline dikes. All the group-means appear to properly average for paleosecular variation of the geomagnetic field expected for 1.0 to 2.2 Ga.

Based on the position of each individual virtual geomagnetic pole, cross-cutting relationship, petrographic analysis, we estimated a relative timescale for the intrusion and paleomagnetic ages of the dike swarms. The steep normal polarity dikes were emplaced at ~1143 Ma during the precursor stage of the MCR magmatism, followed by the emplacement of the Baraga, Mellen-Gogebic, and Central Wisconsin dikes. Finally, the Marquette reversely polarized dikes intruded ~1107 Ma during the early stage of magmatism.

The R-polarity dikes from Baraga and Marquette counties yielded consistent intensity values of ~14.4 µT and ~20.4 µT corresponding to a VDM of ~2 x 10^{22} Am^2 and 3.3 x 10^{22} Am^2, respectively. These values are less than half of the average field strength observed for the last 5 Ma, and are consistent with other paleointensity determinations from the Mesoproterozoic. Four additional sites from the Marquette shoreline are characterized by higher paleointensities, ~4.7 x 10^{22} Am^2, similar to the value obtained from the ~1090 Ma Lake Shore Traps and within the range of the present-day field. Taken at face value, these stronger paleointensity values could support the hypothesis of a compositionally-driven
stable geodynamo operating at ~1.1 Ga. However, low-temperature oxidation (magnetization) of these dikes may have resulted in an overestimation of the field strength.
3 Paleomagnetism and rock magnetism of the Greenstone Flow: Implications for the flow emplacement process and geomagnetic secular variation

3.1 Introduction

Investigation of large lava flows associated with continental flood basalt provinces (CFBP) has the great potential to improve the understanding of many physical phenomena such as the evolution of large igneous provinces (LIPs), the magma evolution and source, and the morphology of the geomagnetic field. The unusual volume and timing of large lava flow eruptions, amongst other aspects, have been well studied and show characteristics similar to those identified on other rocky planets including the Moon, Venus, Mars, and Io (e.g., Keszthelyi et al., 2006; Bryan et al., 2010). For example, a recent study by Keszthelyi and Jaeger (2015) used a model originally developed for circular structures found in the Roza member, a large lava flow of the Columbia Flood Basalt Plateau (e.g., McKee and Stradling, 1970; Hodges, 1978), to explain the formation of features identified on the eroded surface in Athabasca Valles on Mars. However, many aspects of the flow emplacement and solidification remain poorly understood as it has never been directly observed (e.g., Bryan and Ernst, 2008). The majority of our knowledge on the magmatic solidification is derived from analog modeling (e.g., Brandeis and Jaupart, 1986, 1987; Brandeis and Marsh, 1989; Huppert and Worster, 1985; Jaupart and Tait, 1995; Worster et al., 1993) and numeric modeling (e.g., Jaeger, 1968; Hort, 1997; Jaupart and Tait, 1995; Jellinek and Kerr, 2001; Mangan and Marsh, 1992; Marsh, 1988; Brandeis and Marsh, 1989; Marsh, 1996; Rüpke and Hort, 2004). Monitoring of Hawaiian pit craters (e.g., Kilauea Iki, Alae, and Makaopuhi; Wright et al., 1976) has also provided an analogue for the solidification of large lava flows. In this study, we used detailed paleomagnetic and rock magnetic measurements to analyze one of the most voluminous lava flows outcropping on the Earth, the Greenstone Flow, and to constrain a model for its cooling and emplacement.
The Greenstone Flow (GSF) was erupted at ~1094 Ma, during the main (extension) phase of activity of the Mesoproterozoic North-American Mid-continent rift (e.g., Bryan et al., 2010; Davis and Paces, 1990) (Figure 3.1). Outcropping on the Keweenaw Peninsula and Isle Royale island in Michigan (Figure 3.2), this large flow belongs to a series of Mesoproterozoic lava flows erupted during the main stage of magmatism of the MCR, the Portage Lake Volcanics (PLV; Figure 3.1) (Cannon and Nicholson, 2001). While continental flood basalts can display diverse characteristics, the GSF is particularly unique in many aspects, including its thickness, composition, and morphology.

Figure 3.1. Geological map showing the Mid-Continent Rift formations in Lake Superior area. The dashed frames a and b indicate the extent of the Figure 3.3 and Figure 3.4, respectively. The inset display the trace of the MCR formation and the extent of the main map (black outline).
Figure 3.2. Schematic cross-section of the North-American Mid-continent Rift. The green line represents the approximate trace of the Greenstone flow. This figure is modified from Huber (1973).

Unlike other large lava flows, the aggregated volume of the GSF (~1650 km$^3$) extends over a limited area (at its thickest at least ~5 x 10$^3$ km$^2$) (Longo, 1984). As a result, lava accumulated in a thick layer, up to ~450 m near Central, MI, producing the thickest lava flow found on Earth. In comparison, the Roza member flow of the Columbia River Basalt Plateau with its estimated minimum eruptive volume of 1300 km$^3$ (i.e. comparable to the GSF) covers an estimated area over five times larger (~51.8 x 10$^3$ km$^2$) and reaches an average thickness of only ~34 m (Bingham and Grolier, 1966; Bryan et al., 2010; Self et al., 1997; Tolan et al., 1989).

Due to its extraordinary thickness, the GSF cooled over much longer period of time in comparison to other lava flows allowing for the lava flow to differentiate and crystallize a thick pegmatitic layer (e.g., Cornwall, 1951a; Lindsley et al., 1971). Typically, lava flows solidify over periods of weeks to month (Schmincke, 2004) but it is not always the case. For example, the 1959 Kilauea Iki lava lake is still solidifying and is forecasted to be fully cooled below 540 °C in 19 years, about 78 years for a thickness of ~130 m (Gailler and Kauahikaua, 2017). Longo (1984) estimated the solidification time of the thickness of the GSF on Isle Royale (i.e. ~250 m) at 81yrs. This estimate appears to be an underestimate.
considering that it is comparable to the forecasted time for the Hawaiian example, which is almost two times thinner than the GSF thickness on Isle Royale.

Figure 3.3. Extent of the Greenstone Flow and sampling locations on the Keweenaw Peninsula. Simplified geological map showing the extent of the Greenstone Flow on the Keweenaw Peninsula. The letters represent the different sampling locations of this study (see text and Figure 3.4).
Figure 3.4. Simplified geological map showing the extent of the Greenstone Flow on Isle Royale. The letter corresponds to the location where Longo (1984) sampled the GSF.

Multiple paleomagnetic studies of the MCR have included portions of the GSF. Most of the previously studied sites were collected near Ahmeek on the Keweenaw Peninsula (i.e. point A on Figure 3.3) (DuBois, 1962; Books, 1972; Li and Beske-Diehl, 1993) but some sites were studied between Central and Delaware (i.e. points C and D on Figure 3.3) (Hnat et al., 2006) as well as at Blake Point on Isle Royale (i.e. point G on Figure 3.4) (Longo, 1984). The paleomagnetic data of the GSF from both sides of the Lake Superior syncline were used to test the correlation between Keweenaw Peninsula and Isle Royale. Li and Beske-Diehl (1993) found that the remanence directions reported from Isle Royale (Longo, 1984) were 10° steeper than the ones obtained from the Keweenaw Peninsula sites (Books, 1972). Li and Beske-Diehl (1993) suggested that this difference represented the bias imparted by a post-tilting secondary magnetization of the PLV. They also mentioned that the statistical interpretation and sampling scheme could have contributed to this disparity.
Namely, Longo (1984) averaged the results from 26 block samples whereas Books (1972) used only five sites. Li and Beske-Diehl (1993) also identified a difference of 8° in the lava flow just below the GSF. Upon closer inspection of the data, it appears that the Isle Royale sites were taken in a differentiated and thick portion of the GSF, while the Keweenaw Peninsula sites were collected at a point near Ahmeek where the flow abruptly thins and where no differentiation took place. Most likely, cooling of these two portions of the GSF occurred during two different periods and therefore the difference between the Keweenaw Peninsula and Isle Royale paleomagnetic directions can, in fact, reflect paleosecular variation of the geomagnetic field. Other factors, such as incomplete removal of secondary components carried by hematite (e.g., Li and Beske-Diehl, 1993), the extensive mineralization event responsible for the presence of native copper in the area (Browning and Beske-Diehl, 1987; Bornhorst et al., 1988), burial metamorphism (Livnat, 1983), or regional metamorphism (Stoiber and Davidson, 1959), may also affect these results.

In this chapter, we present the result of our paleomagnetic investigation on the differentiated part of GSF on the Keweenaw Peninsula. The aim of this study is to provide a more detailed comparison of the paleomagnetic directions between the different units of the GSF throughout the peninsula exposure and provide a better constraint on the cooling history of the flow and on the impact of alteration and metamorphism.

3.2 Geological setting

3.2.1 The Portage Lake Volcanics

The Portage Lake Volcanics (PLV) are a sequence of Mesoproterozoic aerial basaltic and basaltic andesitic lava flows (nearly 300 individual flows) interbedded with conglomerate layers. The PLV formed during the extension phase of the North-American Mid-Continent Rift (Cannon and Nicholson, 2001) and belongs to the Keweenaw series. Seismic reflection survey reveals that the PLV could be up to 7 km in thickness near the axis of the Lake Superior syncline (Figure 3.2) yet only 3 to 5 km of the sequence was exposed by tectonic activity mainly on the Keweenaw Peninsula and Isle Royale, Michigan, and to a small extent in northern Wisconsin (Behrend et al., 1988; Cannon et al., 1989). The PLV are
overlaid by the Copper Harbor conglomerate and the Lake Shore Traps, and bounded by
the Keweenaw fault and the Isle Royale fault, two thrust faults on the Keweenaw Peninsula
and Isle Royale, respectively (e.g., Cornwall, 1951b). Two of the PLV flows, the Copper
City flow, near the base of the sequence, and the Greenstone Flow, were dated at 1096.2 ±
1.8 Ma and 1094.0 ± 1.5 Ma (U-Pb zircon and baddeleyite ages; Davis and Paces, 1990),
respectively.

3.2.2 The Greenstone Flow

The Greenstone Flow is an important landmark on both the Keweenaw Peninsula and Isle
Royale. The large lava flow is predominant in the landscape forming massive ridges tilting
towards the axis of the MCR (Lane, 1898, 1911; Figure 3.2). The GSF can be traced for a
distance of over 86 km west of Keystone Bay on the Keweenaw Peninsula (Figure 3.3). Its
thickness increases from ~11 m southwest of Houghton to 76 m near Ahmeek. Northeast
of Ahmeek, the flow thickness abruptly increases and varies between 300 m and 450 m
(Broderick, 1935; Cornwall and Wright, 1954) (Figure 3.5). On Isle Royale, the GSF
extends for 71 km (Figure 3.4) between Washington Island and Passage Island and its
thickness fluctuates between 30 m and 244 m (Longo, 1984; Cornwall, 1954a; Huber,
1973).

Figure 3.5. Sketch showing the thickness variation of the Greenstone flow and the Allouez
conglomerate. This figure, not to scale, was redrawn from Longo (1984). The thickness of
the Allouez conglomerate was extracted from drill logs.

Early studies of the Keweenaw Peninsula and Isle Royale mention the GSF because of the
economical copper mineralization that can be mined at its lower border (Pumpelly, 1873;
Books, 1972). The GSF and its underlying conglomerate (i.e. the Allouez conglomerate on
the Keweenaw Peninsula and its correlative on Isle Royale) were used (along with other lava flows) as evidences for the Lake Superior Syncline by early studies of these areas (Lane, 1898; Broderick, 1935; Huber, 1973). Numerous detailed petrologic studies were performed on the flow (Broderick, 1935; Broderick and Hohl, 1935; Cornwall, 1951a, 1951b, 1951c; and reference therein). We should note that, due to the small variation in the chemical composition, most lava flows and units of the PLV are named for their textural characteristics (e.g., Lane, 1898). The GSF is an ophitic olivine basalt that can be described in four units. Most of the upper half of the flow and the entire lower half are composed of ophitic basalt called the ‘Upper-’ and ‘Lower Ophite’, respectively (Figure 3.6). Directly laying on top of the Upper Ophite, massive lava forms a cap between 0.3 m and 15 m of amygdaloidal lava usually called “amygdaloid” (Cornwall, 1954a). Fine-grained columnar basalt can be found locally in the GSF near the top of the flow (Cornwall, 1955).

In the upper half of the flow between the Upper and Lower Ophite and only present in the thickest part of the flow, the minerals are coarser and the rock is characterized by a pegmatitic facies (e.g., Davidson et al., 1955; Longo, 1984). Early studies of the GSF used the terms ‘doleritic’ or ‘dolerite’ to describe this unit due to the presence of coarse mineral, especially feldspars, and the pegmatitic texture of the rock (e.g., Lane, 1898, 1911; Broderick, 1935). These terms were later considered misleading and therefore the terms “pegmatoid” or “pegmatitic layers” were adopted (Cornwall, 1951a, 1954a; Huber, 1973; Longo, 1984) and, in this chapter, we refer to this layer as the “pegmatitic center”. This particular unit is parallel to the plane of the flow and contains interstitial granophyre in places or is cut by granophyritic dikes (Cornwall, 1954b). Evidences of the pegmatoid “autointrusion” into the Upper Ophite were also documented on Isle Royale (Longo, 1984). Some studies proposed that the pegmatoid layer resulted from the intrusion of a sill (e.g., Van Hise and Leith, 1911) but many detailed analyses showed that the layer originated from the slow cooling of a lava flow (e.g., Lane, 1898, 1911; Broderick, 1935; Broderick et al., 1946; Cornwall, 1951b; Huber, 1973).
Figure 3.6. Simplified composition of the Greenstone Flow at each studied location. The Ahmeek section was not sampled but is shown as an example of the thinning/undifferentiated part of the GSF. Sections were redrawn from the USGS maps for the Ahmeek, Phoenix, Eagle Harbor, Delaware, Lake Medora, and Fort Wilkins quadrangles (White et al., 1953; Cornwall, 1954c; Cornwall and Wright, 1954; Cornwall, 1954a, 1955, 1951c, 1954b). GS01-19 are the sites sampled for this study. The studied sites from previous studies and used later are also shown at their reported position; LH11 and LH10 from Li and Beske-Diehl, (1993), PL02-PL21 from Hnat et al., (2006). Sites from Blake Point (IR-1 – IR-4), on Isle Royale, are from Longo (1984).

The occurrence of pegmatoids in lava flows is unusual but is not unique to the GSF. Several other flows of the PLV such as the Mandan flow, the flow overlaying the Kearsage conglomerate, the Copper city “Big Trap” flow, or the Gratiot flow, contain pegmatitic lenses and/or facies (Paces, 1988; Cornwall, 1951a). Many other thick flows, sills and lava lakes elsewhere have been found to have pegmatoidal segregations. For example, the North Mountain Basalt flow (~175 m thick) in Nova Scotia, has differentiated layers composed of pegmatitic and rhyolite bands (Greenough and Dostal, 1992a, 1992b).

Finally, the abrupt change in thickness is particularly unusual for a lava flow (Figure 3.3 and Figure 3.5; Davidson et al., 1955). The underlying Allouez conglomerate is absent in
this area which suggest that the area was at high elevation at the time (Figure 3.5; Longo, 1984). Southwestward of this transition, the GSF is composed of massive lava (often referred as melaphyre) and the pegmatitic center is not present, which indicates that this part of the flow cooled more rapidly (Davidson et al., 1955). The thickness of the columnar-jointed facies that can be found in the uppermost Upper Ophite is unusually large at the abrupt thickening of the GSF, reaching up to 135m, while its typical thickness is ~55 m and ~70 m southwest and northeast of the transition respectively (Davidson et al., 1955). Broderick et al. (1946) estimated that the GSF could have had a relief of ~260 m after its formation, forcing an accumulation of lava flows against the southwest flank of its abrupt transition (Figure 3.5). Broderick et al. (1946) proposed two mechanisms to explain the formation of such elevation change. Their first model suggested that the increasing weight of later lava flows could have drawn out the lava from the southwest to northeast part of the transition (hereinafter referred to as the “injection northward” model, Figure 3.7). The second model involves a gradual thickening of the GSF by the addition of new material into the preexisting core of the flow and the weight northeast may have been isostatically compensated by the weight of the contemporaneous flow erupted on the southwest flank (Figure 3.7). In addition, Longo (1984) proposed that the abrupt thinning of the GSF could have resulted from a pond formed by the lava flows during the waning stage of the eruption from a combination of subsidence and arid runout of the lava towards the basin (hereinafter referred to as the “ponding” model, Figure 3.7).
Figure 3.7. Simplified models for formation of the Greenstone Flow proposed by (a and b), (c), and this study (d and e). The shades of color show similar cooling time, with darker/light shade for the early/late cooling time.
3.3 Methods

Seventeen sites were sampled throughout the Keweenaw Peninsula resulting in a total of 113 drill cores oriented using both magnetic and sun compass. Five sites (GS02-GS06) were collected in the Upper Ophite, the sites GS18-GS19 correspond to the pegmatitic layer, and the remaining ten sites were collected in the lower Ophite (Figure 3.3; Table 3.1). The relative position of sites within the GSF was estimated based on their location and the corresponding geological maps; the Ahmeek, Phoenix, Eagle Harbor, Delaware, Lake Medora, or Fort Wilkins quadrangles (White et al., 1953; Cornwall, 1954c; Cornwall and Wright, 1954; Cornwall, 1954a, 1955, 1951c, 1954b). The relative positions of the sites within the GSF were also combined into a single section to allow the comparison of our results within one stratigraphic column. The thickness of the location at Central was selected for the combined section as it is the thickest portion of the flow sampled (Figure 3.6; Table 3.1). Sample preparation, rock magnetic and paleomagnetic experiments were performed at the Earth Magnetism Laboratory at Michigan Technological University.
Table 3.1. Summary of the sampling sites. Lat/Long, latitude and longitude of the sampled sites; GSF units (UO: Upper Ophiolite, PC: Pegmatitic center; LO: Lower Ophiolite), estimated sections of the Greenstone Flow the site belong to; Normalized depth, distance from the approximate top of the GSF projected to the Central location; Tilt azimuth/dip, tectonic tilt of the flow at the different locations used for structural correction of the paleomagnetic data.

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<th>Lat [°N]</th>
<th>Long [°W]</th>
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<th>Depth [m]</th>
<th>Sampling location</th>
<th>Tilt Azimuth [°]</th>
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3.4 Magnetic mineralogy

Magnetic properties of each site were investigated using a set of rock magnetic experiments. An AGICO MFK1-FA Kappabridge was used to conduct thermomagnetic experiments and measure the bulk magnetic susceptibility. The mean alteration indice (Hrouda, 2003) represents the average difference between the heating and cooling magnetic susceptibilities on the thermomagnetic curves and was used to estimate the changes of magnetic susceptibility between the heating and cooling curves. The magnetic hysteresis properties of the different sections of the flow were measured using a Model 2900 Princeton Measurement Corporation Alternating Gradient Field Magnetometer.

In general, the heating curves display similar susceptibility variation throughout the flow with a major drop of susceptibility between 540°C and 580°C characteristic of low- to medium-Ti titanomagnetite (Figure 3.8; Figure 3.9). A reversible bump of susceptibility can be identified in most of the Upper and Lower Ophite (except the specimen GS06B), suggesting the presence of titanohematite (Figure 3.8; Figure 3.9.a and b). Hematite and maghemite are present in both Ophites (and two specimens in GS18) identified by irreversible susceptibility features at 700°C and between 590°C and 610°C, respectively (Figure 3.8; Figure 3.9). The Upper Ophite and the Pegmatitic center samples display fairly reversible thermomagnetic curves upon temperature cycling (Figure 3.8.a and b; Figure 3.9.a to d). The Lower Ophite exhibits a wide range of change in the magnetic mineralogy upon heating; these specimens are highly susceptible to thermochemical alteration (Figure 3.8; Figure 3.9.e and f; Figure 3.10.a). The variation of the bump on the cooling curves in the Lower Ophite suggests a decreasing Ti-content in the titanomagnetite after heating approaching its center (Figure 3.8).

The ratio of the remanent to induced magnetization value (the Koenigsberger ratio or Q) (Figure 3.10) are generally well constrained between 1 and 10. The Day plot (Day et al., 1977; Dunlop, 2002) shows that the magnetic hysteresis parameters are, on average, consistent with pseudo-single domain grains present throughout the flow (Figure 3.10.c). The Lower Ophite specimens display a higher Mr/Ms ratio and lower Hcr/Hc ratio than in
the Upper Ophite, nevertheless the two groups are undistinguishable. The hysteresis parameters from the Pegmatitic center are generally more consistent with the Lower Ophite but some of the specimens from GS19 deviate from the SD-MD magnetite mixing curves (Figure 3.10.c). These specimens show “gooseneck” hysteresis curves indicative of mixture of grains (Tauxe et al., 1996) (Figure 3.11.c). The grain size increases from the edges towards the pegmatitic center (Figure 3.11.d).
Figure 3.8. Variation of magnetic susceptibility with temperature throughout the different units of the Greenstone Flow. The Upper Ophite (a), the Pegmatitic center (b), and the Lower Ophite (c). The red lines represent the heating from room temperature to 700°C and the blue lines the cooling back to room temperature. Within each part, the thermomagnetic curves are ordered with respect to their position in the flow. The black dotted line on the cooling curves of the Lower Ophite highlights the variation of the susceptibility “bump”.
Figure 3.9. Examples of thermomagnetic curves measured from the different parts of the GSF. The Upper Ophite (a&b), the Pegmatitic center (c&d), and the Lower Ophite (e&f). These curves represent the variation of low-field magnetic susceptibility upon cycling from -190°C to 700°C (red and blue lines show heating and cooling, respectively).
Figure 3.10. Rock magnetic characteristics of the Greenstone Flow. Variation of the mean alteration indice (Hrouda, 2003) (a), the Mrs/Ms (saturation remanent magnetization/saturation magnetization) ratio (b), the low-temperature demagnetization (LTD) memory (c), the percentage of natural remanence magnetization left after thermal demagnetization at ~560°C (d). e. Weak-field magnetic susceptibility versus natural remanence magnetization (NRM) intensity plot. f. Day plot shows the ratio of Mrs/Ms versus the Her/He ratio (coercivity of remanence/coercive force). Abbreviations: SD, single domain; PSD, pseudo-single domain; MD, multi-domain; SD-MD line represents an example of mixture of SD-MD magnetite (Dunlop, 2002).
Figure 3.11. Examples of magnetic hysteresis curves measured from the different parts of the GSF. The Upper Ophite (a&b), the Pegmatitic center (c&d), and the Lower Ophite (e&f). These samples are the same as those representing the variation of the magnetic susceptibility in Figure 3.8.
3.5 Paleomagnetic directions

The characteristic remanent magnetization (ChRM) was isolated using both thermal and alternating field demagnetization. The magnetic remanence was measured throughout the demagnetization experiment on a three-axis DC-squid 2G Enterprises 760-R Superconducting Rock Magnetometer housed in magnetic shielded environment with an in-line (AF) demagnetizer. An ASC TD-48SC thermal specimen demagnetizer with controlled nitrogen atmosphere chamber was used to conduct the thermal demagnetization. Samples were subjected to a low-temperature demagnetization (LTD) prior demagnetization to eliminate the contribution of non-ideal magnetic grains (Schmidt, 1993). The LTD treatment was performed by immersing in liquid nitrogen the samples for at least fifteen minutes and consequently warm them back to room temperature in a magnetically shielded space.

A total of 93 specimens were selected for thermal demagnetization and 28 for alternating-field (AF) demagnetization. Between 40% and 81% of the NRM intensity remain after the LTD treatment (53% in average; Figure 3.10.c), which is consistent with the magnetic hysteresis results (Figure 3.10.f). Samples were demagnetized in 14 to 16 steps up to 555°C or 565°C or 12 to 13 steps until 80 mT or 100 mT. Most specimens display a component randomized by 300°C or 15 mT that we interpret as a viscous remanent magnetization (Figure 3.12). The majority of the remanences became erratic by 530°C-540°C or 60mT, or less than 12% of their NRM was left after the last thermal demagnetization (e.g., Figure 3.10.d, and Figure 3.12.b). Principal component analysis (Kirschvink, 1980) was used to isolate the remanent magnetization on the vector component diagrams and equal area plots (Figure 3.12). Generally, the components were identified within the thermal coercivity spectrum between 400°C and 540°C, and the AF coercivity spectrum between 10 mT and 60 mT. Each best-fit line included a minimum of 5 points (up to 15) and most also included the origin so no anchoring was necessary (Figure 3.12). Almost all the straight lines pointing towards the origin yielded the characteristic remanent magnetizations (ChRM) of normal polarity. Between 5 and 13 specimens were selected to compute the site means of all seventeen sites. The maximum angular deviation (MAD) for these specimens varied
between 0.6° and 5°, except for nine fits that possessed MAD between 5° and 8.8° (Fisher, 1953). All seventeen sites yielded interpretable results corrected for structural tilting based on the bedding information from Cannon and Nicholson (2001) (Figure 3.13; Table 3.1; Table 3.2). Table 3.3 compiles the results from previous paleomagnetic studies of the GSF that we used to complement and compare our paleomagnetic results. The majority of these sites were collected in the area near Ahmeek where the flow is thinner, but some were taken in the lower Ophite in the Central, Delaware, and Medora sections on the Keweenaw Peninsula, and from the different units of the GSF on Isle Royale (Table 3.3).

A mean discrimination test (McFadden and Lowes, 1981) was used to assess the statistical similarities between the corrected site-means of the Keweenaw Peninsula; all of our site-means and the seven site-means from previous studies (Table 3.4). Note that this test could not be performed on the previously reported paleomagnetic directions for which the number of selected specimen or precision parameter was not reported. Sites should share a common site mean direction if they have acquired their remanent magnetization at a similar time. Consequently, the discrimination test also has a potential to establish a relative cooling history between the various units of the GSF.

All sites share a common mean direction with at least one other site with the exception of GS11 which is discordant at 85.5% probability (the “grey” site in Table 3.4). No correlation between the geographic location of the sites and their site-mean has been found. In general, there is a good agreement between our site-mean directions from the upper-, lower- Ophite and the site GS19 from the pegmatitic center. These specimens were grouped to calculate the GSF-2 group mean. However, three sites (GS02, GS16, and GS17; the “blue” sites in Table 3.4) from the Phoenix section form a group isolated from all the other directions. Finally, the sites collected in the center of the flow (i.e. GS18, GS01, GS13, and GS14; the “orange” sites in Table 3.4) have site-mean directions very similar to that obtained from the thinner part of the flow in Ahmeek and from the previously investigated sites in a thicker part of the flow (Figure 3.13).
Figure 3.12. Examples of paleomagnetic results from the Greenstone Flow. Vector component diagrams and equal area plots in geographic coordinates show the characteristic remanent magnetizations (ChRMs) results from Upper Ophite (a), Pegmatitic center (b), and the Lower Ophite (c&d). Temperature (b-d) or AF field (a) steps are indicated. The arrows on the vector component diagram highlight the temperature or AF steps used for
the best-line fit. D: Declination, I: Inclination, MAD: Maximum angular deviation, Range:
The temperature or AF field range used for the ChRM calculation.

Figure 3.13. Paleomagnetic results for the Greenstone Flow. Equal-area projections showing the paleomagnetic site-mean directions, group mean direction and corresponding 95% confidence areas ($\alpha_{95}$).

Group-mean and corresponding virtual geomagnetic pole (VGP) were computed for the thin portion in Ahmeek, the differentiated portions on the Keweenaw peninsula (GSF-1 and GSF-2) and Isle Royale as well as an overall GSF mean (Table 3.5). The angular dispersions of the VGPs ($S$) were calculated using Equation 3.1, where $N$ is the number of individual VGPs and $\Delta i$ is the angle between the $i$-th VGP and the mean pole. The angular dispersion was also corrected for within-site dispersion ($Sw$) due to intrinsic variation within the lava flow and experimental uncertainties using Equation 3.2, where $Sb$ is the true-field (between-site) dispersion, $n$ is the average number of samples per site (Doell, 1970). The N-1 jackknife method was used to estimate the confidence interval on the angular dispersion (Efron, 1982).

$$S^2 = \frac{1}{N-1} \sum_{i=1}^{N} \Delta i^2 \quad (3.1) \quad S_{b}^2 = S^2 - S_{w}^2 / n \quad (3.2)$$
Figure 3.14. Variation of the paleomagnetic directions with depth into the Greenstone Flow. Both declination (a.) and inclination (b.) from this study and from literature are shown against their stratigraphic positions (Table 3.2; Table 3.3). The results from the Isle Royale and Ahmeek sites are arbitrary positioned within the plot.
Figure 3.15. Comparison of paleomagnetic results from different locations within the GSF and from the other PLV flows. Cropped equal-area projections showing the paleomagnetic site-mean directions, group mean direction and corresponding 95% confidence areas ($\alpha_{95}$). (a and b) The paleomagnetic directions and group-means for the thin part of the GSF on the Keweenaw Peninsula (a), and from the differentiated part on Isle Royale (b). (c) Distribution of the paleomagnetic site-mean directions above and below the GSF, and the overall group-mean from Kulakov (2014). (d) Summary of all the paleomagnetic group means calculated at different locations of the GSF, and the group mean for the PLV.
Table 3.2. Summary of the paleomagnetic and corresponding virtual geomagnetic pole (VGP) data from this study. n / N, number of selected / total samples per site; Dg, Ig, and Ds, Is, site mean declination and inclination in geographic and stratigraphic coordinates, respectively; α95, confidence interval of the site mean direction; Plat / Plong, latitude / longitude of the virtual geomagnetic pole (VGP); dp: semi-axis of the confidence ellipse (degrees) along a site to pole great circle; dm: Semi-axis of confidence ellipse (degrees) perpendicular to the great circle; k, best estimate of (Fisher) precision parameter for the site mean. Sites marked with an asterisk were excluded from the group mean. Sites included in the GSF-2 group-mean are marked by †.

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<th>Ds</th>
<th>Is</th>
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Table 3.3. Paleomagnetic results from previous studies on the Greenstone Flow. Lat/Long, Latitude and Longitude estimated or reported for the sites. Rf, reference from which the data was extracted from; a, Longo (1984); b, DuBois (1962), c, Books, 1972; d, Li and Beske-Diehl (1993); e, Hnat et al. (2006); IR, Isle Royale; KP, Keweenaw Peninsula; Th, the thin part of the GSF near Ahmeek. n / N, number of selected / total specimens per sites, the asterisk (*) indicates that the authors reported the number of selected/total oriented block samples used to compute the site-mean instead of the number of specimen; Ds, Is, site mean declination and inclination in stratigraphic coordinates; α95, confidence interval of the site mean direction; k, best estimate of (Fisher) precision parameter for the site mean.

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Table 3.4. Summary of the mean discrimination test performed on the paleomagnetic site means from the GSF. UO, Upper Ophite; PC, pegmatitic center; LO, Lower Ophite; Th, Thin part of the GSF near Ahmeek. The “†” indicates the site-means from a previous study. See the text for the “blue”, “orange”, “purple”, and “grey” background colors. Each numbers (in percentage) shows the statistical similarity between site and when sites are similar below the 80% confidence level not numbers are shown.

| Site    | GS 03 | GS 04 | GS 05 | GS 06 | GS 07 | GS 08 | GS 09 | GS 11* | GS 14 | GS 17 | GS 18 | GS 19 | PL 14 | PL 20 | PL 21 | LH 11 | LH 10 | PL 02 | PL 06 | Top  |
|---------|-------|-------|-------|-------|-------|-------|-------|--------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-----|
|         | UO    | PC    | LO    | UO    | PC    | LO    | UO    | PC     | LO    | UO    | PC    | LO    | UO    | PC    | LO    | UO    | PC    | LO    | UO   |
| GS03    | -     | 90    | -     | 90    | -     | 90    | -     | 90     | -     | 90    | 80    | 95    | 90    | 90    | 80    | 90    | 90    | 95    | 90   |
| GS04    | 90    | -     | 95    | -     | 90    | 95    | 90    | 90     | 90    | 80    | 90    | 95    | 90    | 95    | 90    | 80    | 90    | 90   |
| GS07    | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90     | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90   |
| GS08    | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90     | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90   |
| GS09    | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90     | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90   |
| GS11    | 80    | 80    | 80    | 80    | 80    | 80    | 80    | 80     | 80    | 80    | 80    | 80    | 80    | 80    | 80    | 80    | 80    | 80   |
| GS12    | -     | 90    | -     | 90    | -     | 90    | -     | 90     | -     | 90    | -     | 90    | -     | 90    | -     | 90    | -     | 90    | -    |
| GS13    | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90     | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90   |
| GS14    | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90     | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90   |
| GS15    | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90     | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90    | 90   |
| GS16    | 80    | 80    | 80    | 80    | 80    | 80    | 80    | 80     | 80    | 80    | 80    | 80    | 80    | 80    | 80    | 80    | 80    | 80   |

See the text for the “blue”, “orange”, “purple”, and “grey” background colors. Each numbers (in percentage) shows the statistical similarity between site and when sites are similar below the 80% confidence level not numbers are shown.
Table 3.5. Summary of the group means calculated for the Greenstone Flow. n / N, number of selected / total sites for the group mean; Ds, Is, site mean declination and inclination in stratigraphic coordinates; α95 / A95, confidence interval of the site mean direction/VGP assuming a circular distribution; Plat / Plong, latitude / longitude of the group-mean virtual geomagnetic pole (VGP); k/K, best estimate of (Fisher) precision parameter for the site mean/VGP; R, resultant vector length; S/±ΔS, estimated angular standard deviation of VGPs and corresponding Jackknife estimated error (1 σ). ND, non-differentiated.

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<th>Is [°]</th>
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<th>k</th>
<th>R</th>
<th>Plat [°N]</th>
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<td>19</td>
<td>27.3</td>
<td>191.9</td>
<td>5.6</td>
<td>36.7</td>
<td>13.4 ± 3.0</td>
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<td>288.1</td>
<td>50.6</td>
<td>3.7</td>
<td>231</td>
<td>8</td>
<td>34.4</td>
<td>193.0</td>
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<td>47.5</td>
<td>9.2</td>
<td>100</td>
<td>4</td>
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<td>7.6</td>
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<td>6.7 ± 2.2</td>
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<tr>
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<td>289.2</td>
<td>33.9</td>
<td>5.6</td>
<td>75</td>
<td>10</td>
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<td>5.1</td>
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<td>65</td>
<td>28.3</td>
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<td>3.6</td>
<td>25</td>
<td>12.7 ± 2.1</td>
</tr>
</tbody>
</table>
3.6 Discussion

3.6.1 Stability of the remanence direction

Several lines of evidence confirm the primary origin of the characteristic remanent magnetization isolated from the Greenstone Flow rocks analyzed in this study. First, the paleomagnetic signal in all sampled sections of the GSF is mainly carried by stable pseudo-single-domain low- to medium Ti-content titanomagnetite grains (Figure 3.8, Figure 3.9, and Figure 3.10). Second, the ChRM has high unblocking temperatures between 400°C and 540°C and its demagnetization trajectory converges towards the origins on vector endpoint diagrams (Figure 3.12). Third, the primary origin of ChRM is supported by a positive baked contact test against the Allouez conglomerate directly under the differentiated (thick) part of the GSF, and by a positive conglomerate test observed at more distant sites of the conglomerate (i.e. indicating the absence of a pervasive regional remagnetization) (Li, 1987). We further note that while the thermomagnetic curves show the presence of some (titano)hematite and maghemite in some samples, suggesting that the rocks experienced some alteration, no remanence component corresponding to these minerals has been found by thermal demagnetization. This observation is consistent with the previous results from the GSF using thermal demagnetization (Li and Beske-Diehl, 1993; Hnat et al., 2006); a hematite component was found in only one of the seven studied sites (i.e. PL21 near the bottom of the Central section) (Figure 3.6; Hnat et al., 2006). These results indicates that the GSF was much less affected by the hydrothermal alteration responsible for the acquisition of a thermoviscous remanent magnetization (TCVM) in the other PLV flows (Li and Beske-Diehl, 1993).

3.6.2 Paleosecular variation

The VGP angular dispersion of 13.4° ± 3.0° calculated for the differentiated part of the Greenstone Flow (GSF-1 in Table 3.5) is consistent with the expected dispersion of 12.5° ± 2.4° estimated for rock formed at this latitude between 1.1 Ga and 2.2 Ga (Figure 3.16; Smirnov et al., 2011). Taken at face value, this result suggests that the GSF paleomagnetic
data have recorded the full range of paleosecular variation and represent the time-averaged geomagnetic field which implies that the flow may have cooled over a period as long as 10000 years. However, the analysis of the Kilauea Iki lava lake cooling, when roughly extrapolated to the maximum thickness of the GSF (i.e. ~450 m), suggests that it took less than 450 years for most of the flow to be fully cooled below its Curie temperature. Furthermore, the angular dispersion of VGPs within the differentiated part is nearly equal to the S value obtained for 65 PLV flows (PLV in Table 3.5). Together, these observations suggest that the scatter of GSF directions does not represent the time-averaged field but is caused by another mechanism.

Figure 3.16. Analysis of the Virtual Geomagnetic Pole (VGP) dispersion. Angular dispersion of the VGP from the means calculated for the GSF at various locations; Isle Royale (IR) and the Keweenaw Peninsula thick portion (GSF-1 and GSF-2) and non-differentiated portion (ND). The paleomagnetic pole for the Portage Lake Volcanics is also shown by a purple square based on the revised value from Kulakov (2014). The paleosecular variation (PSV) model fitted for 1.0 to 2.2 Ga are shown with its corresponding standard deviation (dashed and dotted lines, Smirnov et al., 2011). The black dots shows the angular dispersion and associated standard deviation used by Smirnov et al., (2011) to compute the PSV model.
An inadequate structural correction of paleomagnetic data is unlikely to be the source of the scatter. The GSF data appear to be adequately corrected for the tectonic tilting because of (1) lack of a systematic relationship between the remanence directions and their sampling locations, and (2) the statistical similarity between some sites-means from distinct locations (e.g., GS01 and GS18 from Phoenix and Lake Medora respectively).

A closer look at the directional data from the differentiated part of the GSF (Figure 3.13.b and Figure 3.14) show that the data from the upper and lowermost parts of the flow (i.e. the sites included in the GSF-2 group-mean) are noticeably less scattered than the data from the center (i.e. the upper Pegmatitic Center site GS18 and the uppermost Lower Ophite sites). The corresponding values of S are 5.3° and 12.9°, respectively. Thus the large scatter calculated from the entire sequence is mainly due to the scatter in the center. One potential mechanism that may explain this contrast is a difference in the cooling times. Thermal modelling shows that the upper and lower part of the flow would solidify first while the center part would remain in partly molten state for a longer time. It is possible that the pressure from the colder upper part has resulted in small differential motions (e.g., subsidence) within the lower part that resulted in a larger scatter of paleomagnetic directions/VGPs. We note that in order to affect the paleomagnetic record, these motions must have occurred after the rocks cooled below their Curie temperatures (i.e. below 700°C).

3.6.3 Implications for the Greenstone Flow formation

Our results are overall consistent with the results from previous studies. The Isle Royale direction from Longo (1984) are in good agreement with our site-means retrieved from the Upper Ophite and in lowermost part of Lower Ophite (Table 3.4) despite their use low AF field demagnetization and the minimum dispersion criteria instead of the least-square fit to identify the remanence direction. Figure 3.15 show the different group mean calculated for the section of the GSF on both sides of the MCR indicating that the Isle Royale and GSF-2 mean are nearly indistinguishable. These results corroborate our previous observation that the disparity between the Keweenaw Peninsula and Isle Royale paleomagnetic means.

118
highlighted by Li and Beske-Diehl (1993) was mainly a result of paleosecular variation and differences in solidification time. Interestingly, the variation of the remanence direction in the different sections of the flow shows a progression towards shallower inclination from the border towards the center of the flow. Two directional groups can be identified by the mean discrimination analysis; the first one consisting of the Upper and Lower Ophite data from the Keweenaw Peninsula and the results from the Isle Royale, and the second group composed of the sites in the center of the flow. Only two site-means appear to have directions similar to site means from each group (i.e. GS05 site and one of the Isle Royale sites; Table 3.4).

The most interesting feature in our results comes from the statistical similarity between the site in the center of the flow (i.e. the “orange” group in Table 3.4) and the sites from the non-differentiated. The Ahmeek section with its ~76m thickness must have cooled more rapidly than the Medora or Delaware section that are over five times thicker. Nevertheless, the paleomagnetic direction suggested that the Ahmeek portion of the flow has acquired its remanence at the same time as the center of the flow, which most likely indicate that this portion was erupted later, during the cooling of differentiated part of the GSF. A later emplacement of the southwest (thin) part of the GSF could explain the lack of pegmatitic facies in it while on the Isle Royale section of similar thickness experienced the latent heat of the thick, slowly cooling unit which resulted in a longer cooling and the formation of pegmatitic facies (Figure 3.6).

Figure 3.7 illustrates the expected cooling time of the different portion of the GSF according to the various emplacement mechanism. The paleomagnetic results are also incompatible with the “injection northward” model proposed by Broderick et al. (1946) and the “ponding” model proposed by Longo (1984) (Figure 3.7.a and c). The inflation of the northeast portion of the GSF resulting from the injection of lava from the southwest proposed in the “injection northward” model would result in a tilting of the topmost part of the Upper Ophite (Figure 3.7.a and b). We have already established the agreement between the paleomagnetic direction of sites of the Upper and Lower Ophite therefore it is unlikely this model can explain the formation of the GSF. In the “ponding” model, the
southwest portion of the GSF would have acquired its remanence prior the pegmatitic center (Figure 3.7.c). Finally, the second model proposed by Broderick et al. (1946) could explain the formation of the relief of the GSF however in this model again, the southwestern part of the GSF would not have acquired its remanence at the same time as the pegmatitic center in the northeastern part (Figure 3.7.b).

Two scenarios could be proposed to explain this phenomenon. One hypothesis is that the thin portion of the GSF, in fact, represents another flow that was emplaced during the final stages of cooling of the GSF (Figure 3.7.d). However, no evidence of such relationship indicating a contact between two flows can be identified in the field. Another possible explanation could be that some of the autointruding dikes, such as the one identified by Longo (1984) on Isle Royale, have reached the surface of the GSF, permitting some of the lava to flow along the edge of the flow (Figure 3.7.e). The mechanism referred to as “flowout” is used to describe lava escaping from autointrusive dikes, as well as lava tunnels in basaltic flows (Macdonald, 1967) and appears to adequately explain the remanence direction and the field observations. The flow-out could have occurred in several episodes which may explain a relatively large value of VGP scatter (Thin in Table 3.5) observed from a relatively thin flow.

Based on our results and observations from previous studies, we can envision that the slow eruption of the GSF must have progressively filled the rift basin and formed a thick flow that was constrained by an elevation high, highlighted by the lack of Allouez conglomerate near Ahmeek. Towards the latest stage of the cooling of the flow, autointruding dike permitted the lava to flowout of the center of the flow that overcame the elevation high and flowed over tens of kilometers on the edge of the basin.
3.7 Conclusions

Paleomagnetic investigation of the GSF provided us with useful information to constrain a cooling history and emplacement mechanism of a large lava flow. Our results suggest that the GSF was less affected by the regional metamorphism than other lava flows of the PLV. Our detailed analysis revealed that, contrary to previous observation, the difference in the paleomagnetic directions between the GSF from Isle Royale and Keweenaw Peninsula was not a result of a magnetic overprint or an artefact due to the use of outdated methods of paleomagnetic research. The careful comparison between the differentiated part of the flow on both sides of the Lake Superior Syncline shows that the remanence directions vary mainly due to paleosecular variation and difference in cooling time between units; from the edge towards the center of the flow. The dispersion of the VGP appeared to be consistent with the flow cooling for a period (~10000 years) sufficient to adequately average the geomagnetic field. However, based on the solidification of Lava Lake such as Kilauea Iki, the GSF most likely cooled over a shorter period of time (< 450 years) yet an impressively long cooling compared to normal flows. Therefore, the dispersion of the VGP is most likely the results of deformations affecting the center portion of the flow. Finally, similar remanence directions were found simultaneously in the center of the differentiated northeastern part and the southwest portion of the flow. These results could support the occurrence of at least one flowout event towards the end of the flow cooling and explain the unusual morphology of the GSF.
4 Calibration of the pseudo-Thellier paleointensity determination for Precambrian Rocks

4.1 Introduction

Data on the strength of ancient magnetic field (paleointensity) are crucial for understanding the origin and evolution of planets as well as our solar system. The principal challenge to overcome during paleointensity investigation is that many rocks undergo magneto-mineralogical alteration during the successive heating steps required by the original Thellier double-heating protocol (Thellier and Thellier, 1959). Several modifications of the Thellier original protocol have been proposed to reduce the effects of the experimental alteration by decreasing the number of heating steps (e.g., the IZZI protocol, Tauxe and Staudigel, 2004, and the quasi-perpendicular method, Kono and Ueno, 1977), using microwave radiation (Walton et al., 1992, 1993), using alternating-field (AF) demagnetization in place of thermal demagnetization (e.g., Shaw method, Shaw, 1974), using single silicate crystals instead of bulk rock samples (Cottrell and Tarduno, 2000; Tarduno et al., 2006), or multiple sister specimens (e.g., multi-specimen method, Dekkers and Böhnel, 2006). Other alternative approaches involving no heating treatment at all have been developed to include a broader spectrum of rocks (e.g., sediments, meteorites) to paleointensity determination. These non-heating (normalization) techniques use an anhysteretic remanent magnetization (ARM) or a saturation isothermal remanent magnetization (SIRM) in place of laboratory thermoremanent magnetization (TRM) to retrieve relative paleointensity estimates (e.g., the ARM method, Markert and Heller, 1972; Banerjee and Mellema, 1974a; Stephenson and Collinson, 1974), the ratio of equivalent magnetization method, (Cisowski et al., 1975; Gattacceca and Rochette, 2004; Acton et al., 2007), and the pseudo-Thellier method (Tauxe et al., 1995).

The pseudo-Thellier (PT) method experimental protocol is similar to that of the original Thellier method but uses a laboratory ARM instead of laboratory TRM to replace the natural remanent magnetization (NRM) thus involving no heating. The method is frequently used for sedimentary rocks, providing relative paleointensity estimates. The use
of ARM as an analogue of TRM relies on three rock magnetic characteristics (Yu et al., 2003a): (1) the coercivity spectra of alternating field (AF) demagnetization of ARM and TRM are similar, (2) the intensity of ARM and TRM is linearly proportional to the induced field (for small Earth-like fields), and (3) both magnetizations have similar magnetic grain-size dependence (except for magnetite grains smaller than 1µm) (Yu et al., 2003a).

In its most recent form, the PT method protocol consist in progressively removing the natural remanent magnetization using alternating-field (AF) demagnetization before imparting a laboratory ARM in a known magnetic bias field and demagnetizing it using the same AF sequence (de Groot et al., 2013). Analogously to the Thellier method, the relative paleointensity in a specimen is determined by the slope of a linear segment ($b_{PA}$; equation 1) on the pseudo-Arai plot (NRM lost versus ARM gained).

$$b_{PA} = \frac{\text{NRM lost}}{\text{ARM gained}}$$ (3)

The success of the pseudo-Thellier method relies on the fact that the ARM obeys the laws of additivity, reciprocity, and independence equivalent to the corresponding Thellier’s laws for TRM. The law of additivity states that the total ARM corresponds to the sum of partial ARMs induced over different non-overlapping AF coercivity windows. Yu et al. (2002a) demonstrated that the law of additivity was universally obeyed by single-domain (SD), pseudo-single-domain (PSD), and multi-domain (MD) grains with compositions ranging from pure magnetite to TM60 titanomagnetite. The law of reciprocity requires that any ARM induced over a certain AF coercivity window must be demagnetized completely within the same AF coercivity window. Yu et al. (2002b) showed that, while SD and PSD grains obeyed this law, large PSD and MD grains exhibited a nonlinear behavior with the AF demagnetization of these grains generally outweighing the ARM acquisition resulting in curved pseudo-Arai plots. The use of low-temperature demagnetization (LTD) during the experiment can improve the accuracy of the field estimates, reduce the effect of MD magnetite, and consequently reduce the curvature on the pseudo-Arai plots (Yu et al., 2003b). Finally, the law of independence requires that ARM induced over mutually
exclusive coercivity windows remain independent from one another. SD grains obey this law while the large PSD and MD grains violate the independence law (Yu et al., 2003c).

The value of pseudo-Thellier slope ($b_{pA}$) cannot be used directly to calculate the absolute paleointensity value (as in the Thellier method) due to different acquisition efficiencies of ARM and TRM in the same bias field. In addition, the slope depends on the grain-size distribution and composition of magnetic minerals in the sample (e.g., Dunlop and Özdemir, 2001; Yu et al., 2003a; de Groot et al., 2015). An absolute paleointensity in a specimen can be calculated by normalizing the relative pseudo-Thellier estimate by a calibration factor ($c$) to account for the different acquisition efficiencies of TRM and ARM (equation 2; Yu et al., 2003a).

$$B_{ANC} = \frac{b_{pA}}{c} B_{ARM} \quad (4)$$

The calibration of the relative pseudo-Thellier estimate for lavas has been the focus of several studies over the recent years (e.g., Yu et al., 2002b, 2003a, de Groot et al., 2013, 2014, 2015; Paterson et al., 2016 and references therein). Determination of the calibration factors is challenging and is associated with large uncertainties (e.g., Yu et al., 2003b; Gattacceca and Rochette, 2004; Yu, 2010; de Groot et al., 2013; Lappe et al., 2013; Lerner et al., 2017). The uncertainty estimate ($s_B$, i.e. 1σ) for the average pseudo-Thellier paleointensity ($m_B$) depends on the mean and uncertainties of both the average pseudo-Thellier slope ($m_{pA}$ and $s_{pA}$) and the calibration factor ($m_c$ and $s_c$) (equation 3 and 4). Previous studies showed that the pseudo-Thellier method can provide a good estimation of the average field intensity with 25 per cent uncertainties which is however greater than the acceptable range of error for traditional methods (Yu, 2010; Paterson et al., 2016).

$$m_B = \frac{m_{pA}}{m_c} \quad (5)$$

$$s_B = m_{pA} \left( \frac{s_{pA}}{m_{pA}} \right)^2 + \left( \frac{s_c}{m_c} \right)^2 \right)^{1/2} \quad (6)$$
de Groot et al. (2013) found an empirical relationship between the pseudo-Thellier slope and the known reference field values from historical Hawaiian lavas flows. This empirical relationship (equation 5, with $a = 7.371$ and $b = 14.661$) was found to adequately estimate the magnetic field intensity for the specimens for which the field necessary to impart half of the saturation ARM (i.e. a half-saturating ARM field, $B_{1/2\text{ARM}}$) was between 23 mT and 63 mT (de Groot et al., 2013). The calibration relationship was originally based on a narrow range of known Earth’s magnetic field intensities (between 34.7 µT and 37.7 µT, Figure 4.1). Later, de Groot et al. (2015) used well-established paleointensity determinations (up to 80 µT) using the thermal and microwave methods from Canary islands lava flows to refine the empirical formula. Finally, de Groot et al. (2016) further extended the range of field strengths (down to 20 µT) based on the results from lava flows from Terceira and suggested the coefficients $a = 7.718$ and $b = 14.600$ in the calibration formula.

$$B_{abs} = a \times |b_{pA}| + b \quad (7)$$

While the empirical relationship in equation 5 provides absolute intensity estimates comparable to the estimates from the historical or well-constrained thermal data used to derive the relationship, some of its aspects are controversial. In theory, the linear relationship between the pseudo-Thellier slope and the laboratory/historical field should go through the origin of the $B_{abs}(b_{pA})$ plot (i.e. $b$ in equation 5 should be equal to zero). However, the non-zero y-axis intercept in the calibration equation remains even after the refinement provided by de Groot et al. (2016). The second issue comes from the fact that even best paleointensity estimates based on heating methods used by de Groot et al. (2015; 2016) could have been affected by systematic bias thus affecting the calibration formula. Nevertheless, the refinement of the relationship has not changed radically since its original estimation (Figure 4.1).

More recently, Paterson et al. (2016) refined the calibration of the pseudo-Thellier method (equation 5, with $a = 12.195$ and $b = 0$) using natural rocks with a wide range of lithologies from the Emeishan large igneous province, Vesuvius, and Mont Saint Helen. In this study, the natural rock specimens were given a TRM in a known laboratory field (ranging from
10 µT to 100 µT) to model NRM and thus served as synthetic laboratory specimens. The combination of the data from these “natural synthetic” specimens with the results from the historical lava flows from de Groot et al. (2013) permitted to refine the calibration formula to better agree with the theoretical basis of the method (e.g., solving the non-zero intercept problem, Figure 4.1).

Figure 4.1. Calibration of the pseudo-Arai slope from previous studies. Circles are from Paterson et al. (2016, PT16) and diamonds are from de Groot et al. (2013, DG13). The solid lines represent the mean calibrations and dashed lines denote the standard deviation.

The calibrated pseudo-Thellier method represents a valuable tool to assess paleointensity estimates from rocks that cannot be evaluated using the conventional heating methods as they are highly susceptible to chemical alteration upon heating. Consequently, better understanding of the physical foundations and relative efficiency of the method require continued effort. Currently, the calibration factor for the PT method has been only determined from very recent rocks. Finding the calibration factor for non-historical rocks
is critical to expand the calibrated pseudo-Thellier method to a wider range of rocks such as Precambrian or extraterrestrial rocks that are generally affected by laboratory heating induced alteration and/or are available in limited amount of material for experiments (e.g., Yu, 2010; Lappe et al., 2013). In this chapter, we investigate the variation of the calibration factor using a combination of the pseudo-Thellier and Shaw double-heating methods (Shaw, 1974) (Figure 4.2). Both methods use the coercivity spectra of alternative-field (AF) demagnetization of NRM and ARM. Consequently, the LTD-DHT (LTD-double heating) Shaw protocol (Shaw, 1974; Tsunakawa and Shaw, 1994; Tsunakawa et al., 1997; Yamamoto et al., 2003) was implemented to include the progressive acquisition of an ARM (Figure 4.2). As a result, each one of the three cycles provides an estimate of the relative pseudo-Thellier paleointensity (from NRM and two laboratory TRMs – TRM1 and TRM2).

4.2 Methods

All experiments were performed at the Earth Magnetism Laboratory at Michigan Technological University. The pseudo-Thellier-Shaw protocol detailed in Figure 4.2 was used on both synthetic and natural specimens. The synthetic samples are composed of reduced W3006 magnetite (~1.5µm grain size) in non-magnetic matrix of calcium fluoride (CaF₂). The specimens were first air dried in the magnetically shielded room for 24 hours and then gradually heated to 400°C in a nitrogen atmosphere for 5 hours. All the natural specimens were taken from mafic dikes of the ~1.1 Ga Baraga-Marquette swarm that provided stable remanence. The paleomagnetic and rock magnetic characteristics of these specimens are described in Chapter 2.

TRMs were imparted along the z-axis by heating the samples to 610°C in an ASC TD-48SC thermal specimen demagnetizer with controlled nitrogen atmosphere chamber in laboratory bias fields ranging from 30 µT to 50 µT. Magnetic remanence was measured using a 2G Enterprises 760-R Superconducting Rock Magnetometer. Different ARM DC bias fields were used on our specimens: 40, 50, 60, 80, and 100 µT. The results obtained from the natural specimen using the Shaw method were presented in Chapter 2. For the
pseudo-Thellier results, each experimental step on a synthetic specimen was performed in a known laboratory field. The two laboratory induced thermoremanent magnetizations (TRM1 and TRM2) from the Baraga-Marquette specimens are used as ‘natural synthetic’ specimens while the pseudo-Thellier slope from the natural remanent demagnetization (NRM) was considered separately (to determine the ancient field strength).

In order to assess the rock magnetic properties of the Baraga-Marquette sites used as “natural synthetic” specimens, additional sample chips were prepared from at least two specimens per site upon completion of the pseudo-Thellier-Shaw protocol and from non-heated (sister) specimens. Magnetic hysteresis parameters and IRM acquisition curves were measured using a Model 2900 Princeton Measurement Corporation Alternating Gradient Field Magnetometer. During our experiments, the maximum applied AF field was constrained to 100 mT, therefore some of our estimations of the half-saturating ARM\textsubscript{max} fields (B\textsubscript{1/2 ARM}) may underestimate the true value of B\textsubscript{1/2 ARM} used as a grain-size selector for the empirical formula shown in equation 5. Consequently, we complemented our analysis by calculating the half-saturating field of SIRM (B\textsubscript{1/2 SIRM}) from the normalization of the IRM acquisition curves by the SIRM obtained from the hysteresis measurements.

The pseudo-Thellier data were reduced and selected using the approach by Paterson et al., 2016 (Figure 4.3). Vector subtraction was used to reduce the effect of potential presence of high coercivity remanence components. The relative paleointensity estimates were accepted if obtained from at least five data points \( n \) representing at least 45% of the NRM fraction \( f \) with a scatter \( \beta \leq 0.1 \) on the pseudo-Arai plot. In addition, in each of the three used plots (demag-demag, pseudo-Arai, and ARM loss-gain), the selected AF segment should be linear (i.e. \( R^2 \geq 0.9 \)) and, on the NRM-ARM demagnetization plot, the selected trend should trend toward the origin (i.e. \( f_{RESIDUAL} \leq 0.15 \)). Finally, to exclude the data based for non-ideal behavior, the slope on the ARM loss-gain (b\textsubscript{AA}) was required to be -1.00 ± 0.15 (Figure 4.3).
Figure 4.2. Schematic representation of the pseudo-Thellier-Shaw protocol used in this study. The solid black outlined frames show the typical LTD-DHT Shaw protocol and the grey frame represent the pseudo-Thellier protocol.
Figure 4.3. Examples of pseudo-Thellier plots for synthetic and natural specimens: NRM-ARM demagnetization (a&d), pseudo-Arai (b&e), and ARM acquisition-demagnetization (c&f) plots. The black line shows the best-fit line and the solid/hollowed points correspond to the data selected/unselected data for interpretation.
4.3 Rock magnetic properties

Rock magnetic experiments were conducted to characterize the magnetic carrier in our specimens and compare their characteristics with the pseudo-Thellier results. Detailed rock magnetic characterization of the Baraga-Marquette specimens used for the paleointensity experiments can be found in Chapter 2. The thirteen sites selected for the experiment described here are dominated by single-domain to pseudo-single-domain grains. The variation of the low-field magnetic susceptibility with temperature for these specimens yielded two types of thermomagnetic behavior (e.g., type 1 and type 2, Figure 4.4.a and b). Both thermomagnetic curves indicate the presence of a low Ti-content titanomagnetite/magnetite and some medium Ti-titanomagnetite. Type 2 curves feature an additional magnetic mineral most likely titanomaghemite resulting from the maghemitization (low-temperature oxidation) of titanomagnetite. The synthetic Wright 3006 (W3006) powder used for the synthetic sample is composed of ~1.5 µm pseudo-single-domain magnetite. Figure 4.4.c shows the thermomagnetic curve for the W3006 magnetite powder reduced in argon atmosphere. The thermomagnetic curves of type 1 measured from the natural specimens and those from the synthetic specimen appear consistent with the “type H” from de Groot et al. (2016), which has been identified as favorable for the use of their empirical formula.

The Day plot (Day et al., 1977) shows that the “natural synthetic” Baraga-Marquette specimens are composed of PSD grains being well grouped between 20% and 40% on the theoretical mixing SD-MD lines (Figure 4.5). The magnetic hysteresis parameters of the “natural synthetic” are consistent with the results from the natural specimens before experiments. The average half-saturating ARM$_\text{max}$ field value increases from 36 ± 6 mT to 40 ± 6 mT for the natural specimen throughout the experiments for the natural specimens, and is 30 ± 7 mT for the synthetic specimens (Figure 4.6). The half-saturating IRM field values are more variable with averages of 49 ± 15 mT and 65 ± 11 mT before and after the experiments, respectively (Figure 4.6). These average values are statistically indistinguishable due to their overlapping confidence intervals, however, we can estimate
that the $B_{1/2,ARM}$ values are overestimated by 14 and 25 mT, respectively, before and after heating (Figure 4.6).

Figure 4.4. Variation of the low-field magnetic susceptibility with temperature. (a&b) Thermomagnetic curves of type 1 (a) and type 2 (b) measured from the natural specimens. (c) Thermomagnetic curve measured from the W3006 magnetite powder (c).
Figure 4.5. Comparison of the magnetic hysteresis parameters and IRM acquisition curves before and after paleointensity experiments. (a) The Day plot. Saturation remanent magnetization (M_Rs), saturation magnetization (M_s), coercive force and coercivity of remanence (H_c and H_cr, respectively). The dashed lines show SD-MD mixing models from Dunlop (2002). (b) and (c) Examples of hysteresis, remanence, and IRM acquisition curves for natural rock specimens before (b) and after (c) experiment.
Figure 4.6. Variation of the half-saturating ARM fields. The light green area highlights the acceptable $B_{1/2 \text{ ARM}}$ window to calibrate the pseudo-Thellier slopes to absolute paleointensity estimates using the empirical equation (5) from de Groot et al. (2013) (a) Boxplot diagram showing the statistical distribution of the half-saturated ARM$_{\text{max}}$ field in the natural specimens (NRM, TRM1, and TRM1) before correction and synthetic specimens. (b) Examples of half-saturating ARM and IRM fields before and after
experiments. The black contour lines show the difference between $B_{1/2IRM} - B_{1/2ARM}$ (labelled $\Delta$ in mT) for the cases where both half-saturating fields are equal (solid lines) and the average $\Delta$ (dashed) for the specimens before and after the experiments (i.e. 14 and 25 mT, respectively)

4.4 Pseudo-Thellier results

Synthetic and natural samples have low mean residual NRM ($7 \pm 4 \% \,(1\sigma)$ and $9 \pm 10 \% \,(1\sigma)$, respectively) and a good reproducibility of the total ARM before vector subtraction (4% and 22% on average). Both of these results are in agreement with our rock magnetic results and show that the majority of the specimens contain soft minerals (e.g., magnetite, high Ti titanomagnetite, or maghemite) with good thermal stability for synthetic samples and a wider range of thermal stability for the natural sample. Most of the $B_{1/2ARM}$ values (~80%) are within the acceptable ranges after correction for the underestimation.

177 out of 226 natural specimens and 37 out of 72 synthetic specimens yield at least one paleointensity result (linear fit) passing the criteria. In case of multiple fits satisfied the criteria, we selected the fits that would contain the most points, cover the biggest fraction of NRM, and the $b_{AA}$ closest to -1. Most of the rejected results were excluded due to (1) an ARM lost-gain slope ($b_{AA}$) between -0.61 and -0.84, (2) the residual fraction on the demag-demag plot between 0.23 and 0.54, and/or (3) the fraction of NRM providing the best-fit between 0.25 and 0.45. Table 4.1 summarizes the selection criteria values for the accepted specimens.

The accepted pseudo-Thellier slopes for the three synthetic groups (e.g., synthetic and natural synthetic TRM1 and TRM2) plotted against their respective $B_{TRM}/B_{ARM}$ ratio form well-defined linear relationship (with $R^2$ between 0.9813 and 0.999) trending toward the origin (Figure 4.7). As shown by equation 3, the slope of these linear relations is the average calibration factor $m_c$. Overall, the average calibration factor is better defined than the average calibration factors from the $B_{TRM}/B_{ARM}$ ratio data for individual experimental groups (Table 4.2). The calibration factors calculated for the two natural synthetic groups (e.g., TRM1 and TRM2) are indistinguishable (Figure 4.7). Based on the linear
relationships between the pseudo-Thellier slopes and the $B_{TRM}/B_{ARM}$, we can convert each pseudo-Thellier slope to the corresponding value for an ARM bias field of 40μT as it is the most commonly used in the calibrated pseudo-Thellier studies.

Figure 4.7. Average pseudo-Thellier slope results for our synthetic specimens according to the $B_{TRM}/B_{ARM}$ ratio.

Figure 4.7. Average pseudo-Thellier slope results for our synthetic specimens according to the $B_{TRM}/B_{ARM}$ ratio.
Table 4.1. Summary of the selection criteria statistics for the accepted specimens. The number of specimens accepted for each specimen group is shown in bold with the total number of specimen in parentheses. \( n \), number of data points used for the best-fit; \( R^2_{dd} \) and \( R^2_{pA} \), the correlation coefficients for the selected segment of the demag-demag plot and pseudo-Arai plot; \( f_{\text{Residual}} \), the intercept of the best-fit line on the NRM-ARM plot with the vertical axis; \( f_{\text{NRM}} \), fraction of NRM used for the best-fit; \( b_{AA} \), ARM loss-gain slope on the best-fit segment.

<table>
<thead>
<tr>
<th></th>
<th>( n )</th>
<th>( R^2_{dd} )</th>
<th>( f_{\text{Residual}} )</th>
<th>( f_{\text{NRM}} )</th>
<th>( \beta )</th>
<th>( R^2_{pA} )</th>
<th>( b_{AA} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>NRM</td>
<td>52(77)</td>
<td>5</td>
<td>0.95</td>
<td>0.00</td>
<td>0.52</td>
<td>0.00</td>
<td>0.95</td>
</tr>
<tr>
<td>minimum</td>
<td>5</td>
<td>0.92</td>
<td>0.00</td>
<td>0.49</td>
<td>0.00</td>
<td>0.92</td>
<td>-1.05</td>
</tr>
<tr>
<td>maximum</td>
<td>13</td>
<td>1.00</td>
<td>0.14</td>
<td>1.04</td>
<td>0.09</td>
<td>1.00</td>
<td>-0.85</td>
</tr>
<tr>
<td>mean</td>
<td>8</td>
<td>0.99</td>
<td>0.06</td>
<td>0.85</td>
<td>0.02</td>
<td>0.99</td>
<td>-0.96</td>
</tr>
<tr>
<td>TRM1</td>
<td>60(76)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>minimum</td>
<td>5</td>
<td>0.92</td>
<td>0.00</td>
<td>0.49</td>
<td>0.00</td>
<td>0.92</td>
<td>-1.05</td>
</tr>
<tr>
<td>maximum</td>
<td>15</td>
<td>1.00</td>
<td>0.15</td>
<td>1.03</td>
<td>0.05</td>
<td>1.00</td>
<td>-0.87</td>
</tr>
<tr>
<td>mean</td>
<td>9</td>
<td>0.99</td>
<td>0.05</td>
<td>0.86</td>
<td>0.01</td>
<td>0.99</td>
<td>-0.99</td>
</tr>
<tr>
<td>TRM2</td>
<td>65(73)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>minimum</td>
<td>5</td>
<td>0.93</td>
<td>0.00</td>
<td>0.48</td>
<td>0.00</td>
<td>0.93</td>
<td>-1.07</td>
</tr>
<tr>
<td>maximum</td>
<td>14</td>
<td>1.00</td>
<td>0.14</td>
<td>1.02</td>
<td>0.03</td>
<td>1.00</td>
<td>-0.99</td>
</tr>
<tr>
<td>mean</td>
<td>10</td>
<td>0.99</td>
<td>0.06</td>
<td>0.88</td>
<td>0.01</td>
<td>0.99</td>
<td>-1.00</td>
</tr>
<tr>
<td>Synthetic</td>
<td>37(72)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>minimum</td>
<td>6</td>
<td>0.96</td>
<td>0.00</td>
<td>0.47</td>
<td>0.00</td>
<td>0.95</td>
<td>-1.05</td>
</tr>
<tr>
<td>maximum</td>
<td>13</td>
<td>1.00</td>
<td>0.14</td>
<td>1.00</td>
<td>0.02</td>
<td>1.00</td>
<td>-0.86</td>
</tr>
<tr>
<td>mean</td>
<td>10</td>
<td>0.99</td>
<td>0.05</td>
<td>0.74</td>
<td>0.01</td>
<td>0.99</td>
<td>-0.96</td>
</tr>
</tbody>
</table>
Table 4.2. Pseudo-Thellier results for the synthetic specimens. $m_{c40}$ is the average calibration factor for $B_{ARM} = 40$ µT and $m_c$ the average generalized calibration factor.

<table>
<thead>
<tr>
<th>$B_{TRM}/B_{ARM}$</th>
<th>Synthetic</th>
<th>TRM1</th>
<th>TRM2</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Average pseudo-Thellier slopes</td>
<td></td>
</tr>
<tr>
<td>0.38</td>
<td>-</td>
<td>2.22 ± 0.10</td>
<td>2.33 ± 0.54</td>
</tr>
<tr>
<td>0.50</td>
<td>2.17 ± 0.48</td>
<td>2.60 ± 0.68</td>
<td>2.70 ± 1.30</td>
</tr>
<tr>
<td>0.67</td>
<td>-</td>
<td>4.13 ± 0.67</td>
<td>3.88 ± 0.73</td>
</tr>
<tr>
<td>0.75</td>
<td>3.47 ± 0.04</td>
<td>4.71 ± 0.86</td>
<td>4.17 ± 0.90</td>
</tr>
<tr>
<td>1.00</td>
<td>-</td>
<td>6.27 ± 1.43</td>
<td>5.42 ± 1.22</td>
</tr>
<tr>
<td>1.25</td>
<td>5.77 ± 0.83</td>
<td>7.44 ± 0.84</td>
<td>7.42 ± 0.78</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Average calibration factors</td>
<td></td>
</tr>
<tr>
<td>0.38</td>
<td>-</td>
<td>5.92 ± 0.28</td>
<td>6.22 ± 1.43</td>
</tr>
<tr>
<td>0.50</td>
<td>4.33 ± 0.95</td>
<td>5.19 ± 1.36</td>
<td>4.92 ± 1.19</td>
</tr>
<tr>
<td>0.67</td>
<td>-</td>
<td>6.19 ± 1.00</td>
<td>5.82 ± 1.10</td>
</tr>
<tr>
<td>0.75</td>
<td>4.62 ± 0.05</td>
<td>6.28 ± 1.15</td>
<td>5.56 ± 1.20</td>
</tr>
<tr>
<td>1.00</td>
<td>-</td>
<td>6.27 ± 1.43</td>
<td>5.42 ± 1.22</td>
</tr>
<tr>
<td>1.25</td>
<td>4.61 ± 0.66</td>
<td>5.95 ± 0.67</td>
<td>5.94 ± 0.63</td>
</tr>
<tr>
<td>$m_{c40}$</td>
<td>0.21 ± 0.01</td>
<td>0.16 ± 0.01</td>
<td>0.17 ± 0.01</td>
</tr>
<tr>
<td>$m_c$</td>
<td>8.37 ± 0.26</td>
<td>6.30 ± 0.35</td>
<td>6.83 ± 0.40</td>
</tr>
</tbody>
</table>
4.5 Selection of data based on magnetic grain size and thermomagnetic behavior

The natural and synthetic specimens exhibit both half-saturating ARM field value (Figure 4.6) and thermomagnetic behavior (Figure 4.4) that suggest that the use of the pseudo-Thellier calibration formula from de Groot (2016) should adequately estimate the acquisition field of TRM imparted on most of our specimens. We note that while the natural specimens from sites A1, H1, H3, and A10 were included in our analysis, they show evidence of maghemitization, which may influence the outcome of the experiments by overestimating or underestimating the field.

All the natural-synthetic and synthetic results were calibrated using the calibration formulas from both de Groot et al. (2016) and Paterson et al. (2016). The calibrated results were then normalized by the laboratory field used for the corresponding specimen to evaluate the efficiency of each pseudo-Thellier formula. Figure 4.8 shows the variation of the ratio of calibrated pseudo-Thellier field estimates over the laboratory field as a function of the half-saturated ARM field values. Overall, both techniques overestimate the field strength, however, the de Groot et al. (2016) formula provided the best predictions (Table 4.3). The means of the calibrated pseudo-Thellier estimates included within the half-saturating ARM fields window from de Groot et al. (2013) are also statistically indistinguishable from the means including all the accepted results.

These experimental results show that despite the suitable grain size and magnetic mineral identified in our synthetic specimens both calibration formulas provided overestimated field intensity.

No correlation could be identified between the calibration factor and the rock magnetic properties of our specimens (e.g. magnetic hysteresis parameters, or the residual remanence remaining after alternating-field demagnetization, Figure 4.9).
Table 4.3. Efficiency of the calibration formulas to predict the acquisition field of the laboratory TRM induced in the natural synthetic specimens. The mean ratio of the estimated field and expected field is shown with the standard deviation and the fraction of the standard deviation over the mean value in percentage. DG16, (de Groot et al., 2016), PT16, (Paterson et al., 2016).

<table>
<thead>
<tr>
<th>Specimens</th>
<th>DG16 TRM1</th>
<th>DG16 TRM2</th>
<th>DG16 Synthetic</th>
<th>PT16 TRM1</th>
<th>PT16 TRM2</th>
<th>PT16 Synthetic</th>
</tr>
</thead>
<tbody>
<tr>
<td>All</td>
<td>1.50 ± 0.27 (18%)</td>
<td>1.45 ± 0.37 (25%)</td>
<td>1.26 ± 0.16 (13%)</td>
<td>1.78 ± 0.39 (22%)</td>
<td>1.70 ± 0.56 (33%)</td>
<td>1.41 ± 0.24 (17%)</td>
</tr>
<tr>
<td>Filtered</td>
<td>1.53 ± 0.25 (17%)</td>
<td>1.51 ± 0.40 (27%)</td>
<td>1.27 ± 0.16 (13%)</td>
<td>1.82 ± 0.37 (20%)</td>
<td>1.78 ± 0.64 (36%)</td>
<td>1.41 ± 0.25 (18%)</td>
</tr>
</tbody>
</table>

Figure 4.8. Comparison between the ratio of calibrated pseudo-Thellier field estimates over the laboratory field, and the half-saturating ARM field. The light green area highlights the acceptable $B_{1/2\text{ARM}}$ window to calibrate the pseudo-Thellier slopes to absolute paleointensity estimates using the empirical equation (de Groot et al., 2013).
Figure 4.9. Correlation between the generalized correlation factor and rock magnetic characteristics; the magnetic hysteresis parameters $H_{cr}/H_{c}$ (a), $H_{c}$ (coercivity, b), $M_{rs}/M_{s}$ (squareness ratio, c), the half-saturated IRM field ($B_{1/2 \text{IRM}}$, d), the half-saturated $A_{R_{max}}$ field ($B_{1/2 \text{ARM max}}$, e), and the residual TRM left after AF demagnetization (f). Dotted lines mark the linear regressions and the corresponding correlation coefficient is shown in the lower corner of each plot.
4.6 Calibrated pseudo-Thellier results on Precambrian rocks

Nine out of the thirteen of the Baraga-Marquette sites yielded at least four acceptable paleointensity determinations (Table 4.4). The average of the acceptable pseudo-Thellier slope for an ARM field was calibrated using four different ways: (1) with the calibration formula from Paterson et al., (2016), (2) with the calibration formula from de Groot et al. (2016), (3) with the average calibration factor estimated from our synthetic specimens (i.e. $m_{c40} = 0.21 \pm 0.01$) and (4) the calibration factor estimated from our natural synthetic specimen (i.e. $m_{c40} = 0.16 \pm 0.1$). The obtained paleointensities were compared with the results obtained using the Shaw method. The uncertainty estimates for the Paterson et al., (2016) formula and for our natural synthetic calibration were calculated using equation 4. Equation 5 was used to estimate the uncertainties of the results obtained with the de Groot et al. (2016) formula.

Figure 4.10. Boxplot diagram showing the statistical distribution of the average paleointensity estimates from the different calibration techniques and the Shaw results for the accepted Baraga-Marquette sites. The average associated with each technique is marked by a diamond and labelled (also see Table 4.4).
Table 4.4. Summary of the average pseudo-Thellier slopes for the natural specimens from the Baraga-Marquette dikes. N/n, number of specimen/site total/accepted for the average; \( b_{pA40} \), average pseudo-Thellier slope for an ARM field of 40\( \mu \)T, PT16/DG16/pTs/pTNS/Shaw, average paleointensity estimates and uncertainty (in \( \mu \)T) from the calibration from Paterson et al. (2016), de Groot et al. (2016), our synthetic (pTs) and natural synthetic (pTNS) specimens and the Shaw method, respectively. The Shaw results are reported from Chapter 2. Sites B14, B26, A6, and A10 did not yield a sufficient number of accepted pseudo-Thellier slopes (i.e. n>4) and therefore were not included in this table.

<table>
<thead>
<tr>
<th>NRM</th>
<th>N</th>
<th>n</th>
<th>( b_{pA40} )</th>
<th>PT16 [( \mu )T]</th>
<th>DG16 [( \mu )T]</th>
<th>pTs [( \mu )T]</th>
<th>pTNS [( \mu )T]</th>
<th>Shaw [( \mu )T]</th>
</tr>
</thead>
<tbody>
<tr>
<td>B12</td>
<td>6</td>
<td>6</td>
<td>1.6 ±0.3</td>
<td>15.7 ±5.2</td>
<td>26.6 ±2.6</td>
<td>13.0 ±2.8</td>
<td>8.2 ±1.8</td>
<td>14.3 ±4.7</td>
</tr>
<tr>
<td>B28</td>
<td>6</td>
<td>4</td>
<td>3.2 ±0.3</td>
<td>16.7 ±4.5</td>
<td>39.0 ±2.1</td>
<td>26.4 ±2.4</td>
<td>8.7 ±0.9</td>
<td>22.4 ±6.7</td>
</tr>
<tr>
<td>A4</td>
<td>7</td>
<td>4</td>
<td>1.5 ±0.2</td>
<td>15.3 ±4.2</td>
<td>26.3 ±1.3</td>
<td>12.7 ±1.4</td>
<td>8.0 ±1.0</td>
<td>12.9 ±3.1</td>
</tr>
<tr>
<td>B24</td>
<td>6</td>
<td>6</td>
<td>2.5 ±0.3</td>
<td>20.4 ±5.8</td>
<td>34.1 ±2.5</td>
<td>21.2 ±2.8</td>
<td>10.6 ±1.5</td>
<td>14.8 ±2.5</td>
</tr>
<tr>
<td>B17</td>
<td>6</td>
<td>4</td>
<td>1.6 ±0.3</td>
<td>18.6 ±5.6</td>
<td>27.0 ±2.0</td>
<td>13.4 ±2.2</td>
<td>9.7 ±1.6</td>
<td>12.1 ±3.6</td>
</tr>
<tr>
<td>B16</td>
<td>7</td>
<td>4</td>
<td>1.3 ±0.4</td>
<td>17.1 ±6.9</td>
<td>24.3 ±3.0</td>
<td>10.5 ±3.3</td>
<td>8.9 ±2.8</td>
<td>12.2 ±5.7</td>
</tr>
<tr>
<td>A1</td>
<td>6</td>
<td>6</td>
<td>3.3 ±0.3</td>
<td>18.1 ±4.9</td>
<td>39.8 ±2.6</td>
<td>27.3 ±3.0</td>
<td>9.4 ±1.1</td>
<td>26.3 ±4.1</td>
</tr>
<tr>
<td>H1</td>
<td>7</td>
<td>5</td>
<td>3.3 ±1.2</td>
<td>22.2 ±10</td>
<td>39.8 ±9.4</td>
<td>27.3 ±10.3</td>
<td>11.5 ±4.4</td>
<td>23.6 ±4.9</td>
</tr>
<tr>
<td>H3</td>
<td>6</td>
<td>5</td>
<td>2.6 ±0.3</td>
<td>12.9 ±3.5</td>
<td>35.1 ±1.9</td>
<td>22.2 ±2.2</td>
<td>6.7 ±0.7</td>
<td>28.8 ±9.3</td>
</tr>
<tr>
<td><strong>Average</strong></td>
<td><strong>9</strong></td>
<td><strong>9</strong></td>
<td><strong>2.3 ±0.8</strong></td>
<td><strong>17.5 ±2.6</strong></td>
<td><strong>32.4 ±6.0</strong></td>
<td><strong>19.3 ±6.6</strong></td>
<td><strong>9.1 ±1.4</strong></td>
<td><strong>18.6 ±6.2</strong></td>
</tr>
</tbody>
</table>
Of the four pseudo-Thellier calibration methods used, only the calibration formula from Paterson et al. (2016) yielded the mean paleointensity value similar to that obtained by the Shaw method (Figure 4.10), whereas the de Groot et al. (2016) formula resulted in a noticeably higher value and the calibration factors based on our synthetic and natural samples resulted in lower paleointensity values. We note however that the uncertainty intervals of all four determinations overlap with the uncertainty interval of the Shaw results (Figure 4.10). Interestingly, the calibration factor based on the synthetic specimens provides results more consistent with the Shaw estimates than the calibration factor based on the natural specimens does. This disparity suggests that the natural specimens could have undergone a change in their mineralogy sufficient to affect the calibration factor but still not strong enough so that they can be adequately corrected using the LTD-DHT Shaw method procedure.

Finally, we should note that the use of the Shaw method paleointensity results to discriminate between the various calibration techniques cannot be considered as a strict validation of the calibration from Paterson et al. (2016) for Precambrian rocks since the true field intensities recorded by these rocks are unknown. However, the perfect correlation between the results of these two paleointensity methods implies a strong cross-validation.

### 4.7 Implications for the paleointensity of the Baraga and Marquette dikes

The paleointensity values (2-4 x 10^{22} Am^2) obtained using both LTD-DHT Shaw and pseudo-Thellier methods suggest that the field strength during the formation of the Baraga-Marquette dikes in the Mesoproterozoic was significantly (by 2-3 times) lower than the average Earth’s magnetic field strength (~7.5 x 10^{22} Am^2) for the last 10 millions of years. The paleointensity values from the Baraga-Marquette dikes are also lower than the average field strength (~ 6 x 10^{22} Am^2) obtained using the LTD-Thellier method from a sequence of slightly younger lava flows erupted during the late stage of the Midcontinent Rift magmatic activity – the ~1087 Ma Lake Shore traps (Kulakov et al., 2014) (Figure 2.27). On the other hand, the measured paleointensities are consistent with other paleointensity
results reported for the Neo- and Mesoproterozoic periods that vary between 1 and \(4 \times 10^{22}\) Am\(^2\) (Yu and Dunlop, 2001, 2002; Macouin et al., 2003, 2006; McArdle et al., 2004; Celino et al., 2007; Sprain et al., 2018) (Figure 2.27). Taken at face value, these and our results suggest that the field intensity was low during this time period, and this was interpreted as supporting a “Proterozoic dipole low” (Biggin et al., 2009). We note that all of these paleofield are lower than the critical field value of approximately \(4 \times 10^{22}\) Am\(^2\) which is characteristic for transitional or excursional geomagnetic field recorded in 0.03-10 Ma volcanic rocks (Tanaka et al., 1995) and may be associated with the emergence of large non-dipole fields at many locations (Guyodo and Valet, 1999; Tarduno and Smirnov, 2004).

However, our paleodirectional data indicate that the paleointensity results from the Baraga-Marquette dikes represent a non-transitional geomagnetic field. Furthermore, large long-term nondipole components of the geomagnetic field have not been identified in searches of data from the late Archean to Proterozoic interval (e.g. Smirnov and Tarduno, 2004; Evans, 2006; Smirnov et al., 2011). Therefore, if true, the long-term low field intensities would imply a Proterozoic geomagnetic field that is significantly different from its modern counterpart, i.e. the geodynamo able to produce low field strength during stable polarity epochs. This exciting possibility requires an additional investigation including numerical modelling of the geodynamo.

It is noteworthy, however, that the studies resulting in low paleofield values are mostly based on slowly cooled intrusive rocks. It was suggested that the low paleointensities recorded by intrusive rocks are likely to be an artifact due to the presence of a thermochemical remanent magnetization (TCRM) (Smirnov and Tarduno, 2005). A TCRM is imparted if the oxyexsolution process during initial lava cooling continues at temperatures below the Curie point of magnetite. Estimates of the TCRM/TRM ratio show that the Thellier data could underestimate the true field value by a factor of 4. Therefore, at this stage, we cannot rule out that the Mesoproterozoic geodynamo was similar to the recent compositionally-driven dynamo.
4.8 Conclusions

The calibrated pseudo-Thellier method has a great potential to improve our understanding of the magnetic field recorded by rocks highly susceptible to thermochemical alteration. However, reliable field estimates necessary to verify if the calibrations available in literature are appropriate for such Precambrian or extraterrestrial rocks are very sparse or absent. In this chapter, we studied the variation of the pseudo-Thellier slope on synthetic and natural specimen using a combination of the pseudo-Thellier and Shaw protocols. The rock magnetic experiments performed on the specimens before and after the experiment showed that their magnetic minerals are well suited to employ the calibration formulas proposed for historical and recent rocks in previous studies. The analysis of the prediction efficiency on the synthetic samples revealed that the both calibration formulas generated overestimate field estimates. Further comparison revealed that the generalized calibration factors of the synthetic specimens were inconsistent with the calibration factors of the specimens used to elaborate the calibration formulas. Consequently, we conclude that the grain-size selector and the thermomagnetic behavior may not always adequately select specimens suitable for paleointensity determination.

The combination of the pseudo-Thellier and Shaw protocols allowed us to compare the absolute intensity Shaw results with the pseudo-Thellier paleointensity results calibrated using the formulas from previous studies (e.g., de Groot et al., 2016 and Paterson et al., 2016) as well as using the calibration factors we estimated from our synthetic and natural synthetic specimens. Our results show that the calibration formula from Paterson et al. (2016) provides paleointensity estimates consistent with the results from the Shaw method. These results not only suggest that the slight changes in the magnetic mineralogy during the heating involved in the Shaw protocol were adequately corrected but they also indicate that the pseudo-Thellier method can provide reliable absolute intensity estimates from Precambrian rocks.
5 Conclusions

The new results presented in this dissertation demonstrate that paleomagnetic and paleointensity investigations represents a powerful means to obtain information about the long-term evolution of the intensity, morphology, and stability of the Earth’s magnetic field. The geomagnetic field characteristics are intrinsically related to the Earth’s deep interior processes and thus provide important insights into the thermal history and structure of our planet. Equally important, the paleomagnetic data are crucial for deciphering the tectonic and geological histories at the global and regional levels. The paleomagnetic and paleointensity investigations accomplished in the course of this PhD work have provided new valuable data and new insights into the geodynamo and inner core evolution, the early evolution of the North American Midcontinent Rift, and the formation processes of large lava flows.

The detailed paleomagnetic investigation of the numerous mafic dikes exposed in the Baraga and Marquette counties have resulted in several important achievements (Chapter 2).

First, the existence of three pulses of dike intrusion in the Baraga-Marquette area have been established based on comparison of the primary paleomagnetic directions recorded by the dikes. Each dike emplacement group is characterized by a statistically different group-mean paleomagnetic direction. Two of the groups were emplaced during two different reversed geomagnetic polarity epochs, and one was emplaced during a normal polarity epoch.

Second, the emplacement ages for each group have been estimated by correlation of the corresponding group-mean paleomagnetic poles with the North American apparent polar wander path (APWP). According to these analyses, the reversely magnetized dikes south of the Fall River thrust fault in the Baraga County were emplaced at ~1160 Ma, followed by the intrusion of the normally magnetized dikes in the Marquette County at ~1140 Ma, and finally the reversely magnetized dikes in the Marquette County intruded at ~1110 Ma.
The latter age is corroborated by a radiometric age determination at ~1107 Ma from the Yellow Dog intrusion genetically related to the investigated dikes.

Third, the new paleomagnetic results indicate that the ~1160 Ma dikes in the Baraga County represent the oldest known magmatic event associated with the Midcontinent Rift formation, replacing in this capacity the ~1142 Ma Abitibi/lamprophyre magmatism in northern Ontario (Canada).

Fourth, two new reliable estimates of the angular dispersion of virtual geomagnetic poles (VGP) have been obtained from the reversely magnetized dikes in the Baraga and Marquette areas. These estimates indicate the presence of a stable and strongly dipolar geomagnetic field at ~1.1 Ga. More importantly, these data fill a gap in the data on latitudinal dependence of the angular dispersion of VGP for high latitudes (Figure 2.23). In addition, these analyses indicate that the group-mean paleomagnetic directions obtained from both reversely magnetized groups represent the time-averaged geomagnetic field.

Future work on the Baraga-Marquette dikes should include detailed geochemical analyses to characterize the magma sources for the different dike emplacement groups, and radiometric geochronological analyses to confirm their ages obtained by the paleomagnetic dating. Additional dikes exposed along the Lake Superior shoreline north of Marquette need to be sampled to increase the statistical significance of the group-mean paleomagnetic direction and pole corresponding to the normally magnetized dikes.

The new paleointensity data obtained from the reversed Baraga-Marquette dikes (Chapter 2 and 4) using the Shaw and calibrated pseudo-Thellier method constitute an important contribution to the paleointensity database for the Precambrian, which is currently very scarcely populated (Chapter 1). The new paleointensity values (2-4·10^{22} Am^2) suggest the time-averaged field strengths that is 2-3 times weaker than the average field strength during the last 200 millions of years. However, these values are within the field variability range for the Phanerozoic (Figure 5.1).
Figure 5.1. New group-mean paleointensity data from the Baraga (red diamond) and Marquette (red square) areas. Also shown are the site-mean paleointensity data (dipole moment) from the global database for the 500-3500 Ma period (the green circles are determinations from bulk rock samples, the red diamonds are determinations from single silicate crystals, the open circles/diamonds are the group-mean values calculated from three or more sites). The grey oblique crosses show the site-mean values for the Phanerozoic (0.05-500 Ma) (grey oblique crosses). The grey dashed line corresponds to the mean paleointensity value calculated from the data for the 0-200 Ma period. The errors shown are 1σ. The rectangles outlined with blue dashed lines schematically show the approximate duration and field strength corresponding to the three stages of geodynamo evolution in the model by Ziegler and Stegman (2013): 1) a relatively strong field generated in the basal mantle ocean; 2) the period of generation of a weak field in the absence of an inner core; 3) the period of generation of a strong field in the presence of the inner core (compositionally-driven geodynamo). The red dashed line shows the field strength level at $4 \cdot 10^{22}$ Am$^2$ indicating the threshold between a stronger stable dipolar field and a weaker transitional and less stable/dipolar field (see text).

These relatively low values are consistent with the paleointensity data reported for the Proterozoic by other authors. Taken alone, the new data are consistent with the basal mantle ocean model (Ziegler and Stegman, 2013; Chapter 1) that suggests a very young (age <500 Ma) solid inner core (Figure 5.1). However, this interpretation contradicts the data on the angular dispersion of VGPs (Figure 2.23 and Chapter 2) that indicate a stable and strongly dipolar field in the Proterozoic. According to the current paradigm, the latter would require
a compositional geodynamo with the inner core formed in the Archean time. Furthermore, the low field intensity values in the Proterozoic are below the $4 \times 10^{22}$ Am$^2$ level (Figure 5.1), which, according to the current understanding of the geodynamo processes, represents the critical value below which the field is in the less stable (transitional) and less dipolar state (e.g., Tarduno and Smirnov, 2004). However, this also contradicts the VGP angular dispersion data suggesting a stable and strongly dipolar field.

The contradictions between the Proterozoic paleointensity and paleodirectional data discussed above may indicate that the current geodynamo paradigm may need to be reconsidered. A low-strength but dipolar and stable field is not consistent with the modern-style compositional geodynamo. If confirmed by subsequent investigations, such a field behavior may require development of a different geodynamo model for the Proterozoic.

The paradoxes described above emphasize the importance of obtaining new high-quality paleointensity determinations for the Precambrian. However, this task is extremely challenging because most Precambrian rocks are not suitable for the paleointensity determinations using the conventional double-heating Thellier method due to heating-induced laboratory alteration (Chapter 1). The methodological investigation of the suitability of reduced/non-heating paleointensity determination methods (Chapter 4) has indicated that the calibrated pseudo-Thellier method represents the best alternative to the conventional method if the calibration procedure by Paterson et al (2016) is used. As demonstrated by our paleointensity investigation for the Baraga-Marquette dikes, the application of this technique opens new opportunities to substantially expand the paleointensity database for the Precambrian.

The detailed paleomagnetic investigation of the Greenstone Flow (Chapter 3) allowed us to constrain its formation mechanism. Specifically, the obtained data are best explained by the "flowout" mechanism in which the slow eruption of the flow first filled the rift basin and formed a thick flow constrained by an elevation high. During a later stage of the flow cooling, an autointruding dike allowed the lava to flow-out of the center of the flow that overcame the elevation high and flowed tens of kilometers on the edge of the basin.
All the results described in this dissertation have been presented at several international and national scientific meetings of the American Geophysical Union, the International Association of Volcanology and Chemistry of the Earth’s Interior, the Castle meeting and the Institute for the Lake Superior Geology. Several papers to be submitted to peer-reviewed journals are now in preparation.
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166
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