Mineral Magnetic Studies of Archaeological Samples: Implications for Sample Selection for Paleointensity Determinations

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Samples with a significant fraction of multidomain magnetic grains, hard secondary components and thermally unstable magnetic phases have been shown to be unreliable for paleointensity studies. However, mineral magnetic screening is rarely performed before paleointensity determinations are made even though non-ideal magnetic properties are the main reasons for rejecting data after the work has been completed. We have conducted a detailed mineral magnetic investigation of 23 archaeological samples from Bulgaria which yielded both satisfactory and unsatisfactory paleointensity results. Our study demonstrates how the non-ideal magnetic properties lead to unacceptable paleointensity results. We have used our findings to develop a simple and practical sample selection procedure which requires only two specimens from each sample and which can be done with conventional paleomagnetic equipment. We suggest that any sample which fails to pass this screening should not be subjected to time-consuming Thellier-Thellier experiments.

1. Introduction

The majority of high quality absolute paleointensity data have been obtained from volcanic rocks and archaeological materials. Almost all of these determinations are based on the method of Thellier and Thellier (1959) or on subsequent modifications of this method (Nagata et al., 1963; Coe, 1967; McElhinny and Evans, 1968; Shaw, 1974; Kono and Ueno, 1977; Kono, 1978; Senanayake et al., 1983; Rolph and Shaw, 1985; Walton, 1986, 1987; Hoffman et al., 1989).

The method of Thellier and Thellier (1959) involves a comparison of the natural remanent magnetization (NRM) of a sample with the thermal remanent magnetization (TRM) acquired in a known laboratory field. The theoretical equation is:

\[
\frac{\text{NRM}}{\text{TRM}} = \frac{F_a}{F^*}
\]

where \(F_a\) is the paleointensity and \(F^*\) is the intensity of the laboratory magnetic field. In the Thellier-Thellier experiment, one measures the NRM remaining after a sample has been heated to a particular temperature as well as the partial thermal remanent magnetization (pTRM) acquired by the sample as it is cooled from the same temperature in a known laboratory field. The assumptions implicit in the method are (Collinson, 1983): 1) the intensity of the pTRM is proportional to the applied field, 2) the pTRM acquired by cooling through a certain temperature interval is lost when the sample is heated through the same interval, 3) the pTRMs are additive, and 4) the TRM acquired is independent of cooling rate.

It is customary to determine the paleointensity from a plot of the NRM remaining versus the pTRM gained after each temperature step. The primary criterion for reliability is linearity of the NRM-TRM curve. If the curve is non-linear, the data are rejected except when the only non-linearity is at low temperatures and can be interpreted as arising from a viscous component. Because each sample is heated many times, alteration of the magnetic minerals can become a serious problem at higher temperature steps.
Although detailed mineral magnetic investigations are seldom employed in paleointensity (and archaeointensity) studies, such investigations can be useful in two ways. First, the method of Thellier and Thellier (1959) is time-consuming and labor-intensive, and the number of samples from which useful data are obtained can be quite low. Thus it would be helpful to be able to screen the samples before the Thellier-Thellier experiments are performed. The goal of the screening would be to identify samples with an NRM that is a primary TRM and with magnetic minerals that are thermally stable and in a single domain (SD) and/or pseudo-single domain (PSD) state. A second reason to conduct mineral magnetic studies is that even if the behavior of the sample is acceptable by the Thellier-Thellier criteria, the results may not be reliable (Prevot et al., 1983, 1985). Detailed mineral magnetic studies could help to resolve issues that have arisen in this regard (Walton, 1984, 1988; Merrill, 1987).

Several previous studies have addressed the question of sample selection. For example, Senanayake and McElhinny (1981) used low-temperature susceptibility data to help select samples, and they reported an increase in the success rate for paleointensity determinations (Senanayake et al., 1982). However, later researchers reported that the approach did not work very well (Rolph, 1984; Thomas, 1993). There have also been contradictory interpretations of the low-temperature susceptibility data (Senanayake and McElhinny, 1981; Radhakrishnamurty, 1993). Prevot et al. (1985) and Derder et al. (1989) used three criteria for sample selection: 1) that the stable NRM directions should be very close to the average direction of the cleaned remanence of the sample, 2) that the viscosity indices should be less than 5%, and 3) that the magnetic mineralogy must be dominated by a single phase with a high Curie temperature and reversible $J_\alpha$-$T$ curves. These criteria are based on measurements of the stable NRM, the magnetic viscosity, the magnetic susceptibility, the temperature dependence of a saturation-induced magnetization, and values of the Koenigsberger coefficient. In the work of Derder et al. (1989), only 8% of the 220 samples originally collected were found to meet the stated criteria. As a result, the criteria have been criticized as being too strict and as leading to the rejection of samples that were actually acceptable (Prevot, 1985; Thomas, 1993).

Thomas (1993) used multiple mineral magnetic techniques to develop sample selection criteria for Precambrian basalts containing pure magnetite with a mixture of grain sizes. Both thermal and non-thermal methods were used to categorize the magnetic behavior. The different types of behavior were compared with results of paleointensity experiments, and selection and rejection criteria were defined. This approach seems to be more thorough than previous ones; however, it is probably only applicable to basalt samples with mixed grain size assemblages of pure magnetite.

The relationship between mineral magnetic properties and the success or failure of the Thellier-Thellier experiment in archaeointensity studies has been explored by Kovacheva and Toshkov (1994). Although the mineral magnetic methods used in that paper are limited, the work demonstrated that even for young archaeological materials, the relationship can be quite complex. Cui and Verosub (1995) used mineral magnetic parameters to develop simple and practical sample selection criteria for a small collection of pottery samples whose only magnetic mineral was magnetite. In the present paper, we use a similar approach to study a larger and different group of archaeological samples. This group includes materials from which good and bad paleointensity results had previously been obtained.

2. Description of Samples

The current study is based on a suite of 23 archaeological samples collected from sites in Bulgaria. The samples vary considerably in nature and age (Table 1). The purpose of this study was to determine why some samples gave acceptable paleointensity results and why others gave unacceptable results. The original paleointensity experiments were done in the Paleomagnetism Laboratory of the Geophysical Institute of the Bulgarian Academy of Sciences at Sofia, Bulgaria. The data were interpreted by the fourth author as acceptable and unacceptable (good and bad), based on standard criteria for the method of Thellier and Thellier (1959) as implemented by the Sofia laboratory (Kovacheva and Kanarchev, 1986; Kovacheva and Toshov, 1994). Mineral magnetic properties were then classified as positive and negative based on
Table 1. Age and description of samples used in this study.

| Sample # | Age       | Nature and source                  | Results of paleointensity |
|----------|-----------|-----------------------------------|---------------------------|
| KPH 4a   | 6000 BC   | Hearth                           | Good                      |
| CpH11a   | 5800 BC   | Burnt dwelling wall               | Good                      |
| CpH1     | 5800 BC   | Burnt dwelling wall               | Bad                       |
| Ko13g    | 5500 BC   | Burnt wall                        | Good                      |
| Ko5a     | 5500 BC   | Hearth                           | Conditionally good        |
| 1069     | 5 c. AD   | Brick from Roman town            | Good                      |
| 1371     | 14 c. AD  | Brick from Medieval church       | Good (?)                  |
| 1948b    | 12 c. AD  | Burnt wall of a Medieval pit     | Good                      |
| 1949a    | 12 c. AD  | Burnt wall of a Medieval pit     | Good                      |
| 2072     | 4 c. BC   | Destruction (wall) of the Thracian town after an ancient fire | Good |
| 2073     | 4 c. BC   | Destruction (wall) of the Thracian town after an ancient fire | Good (?) |
| 2067     | 4 c. BC   | Destruction (wall) of the Thracian town after an ancient fire | Good |
| 2098     | 4 c. BC   | Insufficiently burnt floor of Thracian house | ? |
| 33       | 4500 BC   | Prehistoric hearth               | Bad                       |
| 43       | 4500 BC   | Prehistoric hearth               | Bad                       |
| 65       | 4500 BC   | Prehistoric hearth               | Bad                       |
| 703-6    | 6 c. AD   | Brick from Roman town            | Bad                       |
| 896a     | 10 c. AD  | Medieval hearth                  | Bad                       |
| 957      | 2 c. AD   | Brick from a Roman fortress      | Bad                       |
| 1174     | 2 c. AD   | Brick from a Roman town          | Bad                       |
| 1290b    | 18 c. AD  | Brick from Turkish mosque        | Bad                       |
| 1508     | 7 c. AD   | Roman furnace                    | Bad                       |
| 2028     | 17 c. AD  | Brick                            | Good (?)                  |

their empirical and theoretical relationship to the method of Thellier and Thellier (1959). Sample selection criteria were developed on the basis of these properties.

3. Mineral Magnetic Methods

Because no single method can provide complete and accurate information on the mineral magnetic properties of a sample, several techniques were used in this study. In all cases, the measurements were done using fresh material. For each sample, the following properties were measured: AF demagnetization of NRM, ARM and SIRM, thermal demagnetization of NRM and thermal demagnetization of a three-axis IRM, IRM acquisition behavior, hysteresis parameters, temperature-dependent susceptibility, and frequency-dependent susceptibility. These measurements usually required four specimens from each sample. The first specimen was used for stepwise thermal demagnetization of the NRM, with measurement of susceptibility at each temperature step. This specimen provided information about the thermal stability of the sample, the unblocking temperature distribution, the median destructive temperature, the existence of secondary remanence components and the magnetic mineralogy. Frequency-dependent susceptibility was measured before the thermal demagnetization measurements in order to provide information about the presence of grains near the single domain/superparamagnetic (SD/SP) boundary. The second specimen was used for AF demagnetization of the NRM and ARM, and for studies of the acquisition and AF demagnetization of an IRM (Lowrie, 1990). This specimen was then subjected to thermal demagnetization of a three-component IRM. The third specimen was used for magnetic hysteresis measurements. The measurements on these three specimens were intended to determine the median destructive field (MDF), the median destructive temperature (MDT), and the dominant magnetic minerals, as well as their domain states. The results of the AF demagnetization of the NRM and SIRM were used to perform the Lowrie-Fuller test for determining the domain state (Lowrie and Fuller, 1971). The fourth specimen was used for determination of the temperature-dependent susceptibility ($\chi$ vs. $T$) which provided information...
about magnetic phases and their Curie points. The amount of material required for the third and fourth specimens is small: about from 5 to 10 mg for the third specimen and about 2 g for the fourth specimen.

The IRMs were imparted with an ASC Impulse Magnetizer. Susceptibility was measured with a Bartington Instruments MS2 magnetic susceptibility meter. Temperature-dependent susceptibility was measured with a Kappabridge KLY-2 magnetic susceptibility meter with a CS-2 furnace. Magnetic hysteresis parameters were obtained with a Princeton Measurements Corporation Micro Mag alternating gradient magnetometer. Most remanence measurements were made with a 2G Enterprises Model 760 cryogenic magnetometer in a magnetically shielded room. Some remanence measurements for strongly magnetized samples (e.g., IRM) were made with a Schonstedt Model SSM-1 Spinner Magnetometer. All of these experiments were done in the Paleomagnetism Laboratory of the Department of Geology at the University of California at Davis.

4. Results and Discussion

The mineral magnetic properties of these samples vary considerably and are summarized in Table 2. The MDT ranges from 270°C to 560°C, and the MDF ranges from 12 to 90 mT. By the standard interpretation of the Lowrie-Fuller test (Lowrie and Fuller, 1971), many of the samples appear to be dominated by SD grains. However, Xu and Dunlop (1995) recently showed that under certain circumstances MD grains can exhibit SD-behavior in the Lowrie-Fuller test so the interpretation of the Lowrie-Fuller test for these samples is problematic. On the other hand, non-SD behavior in the Lowrie-Fuller text still appears to be a valid indicator for the predominance of MD grains (Xu and Dunlop, 1995).

Under these circumstances, it is not surprising that samples which exhibit SD-type behavior in the Lowrie-Fuller test have magnetic hysteresis parameters indicative of pseudo-single domain (PSD) grains on the plot of Day et al. (1977). However, for samples that contain a significant fraction of magnetic minerals other than magnetite, the hysteresis parameters cannot be used to determine the domain state. Many of the samples had wasp-waisted hysteresis loops (cf. Roberts et al., 1995). The significance of these hysteresis loops will be discussed in the next section.

In this study, those samples which produced satisfactory paleointensity results always had the following magnetic properties: 1) SD behavior in the Lowrie-Fuller test, 2) a negligible secondary remanence component, 3) a broad unblocking temperature distribution with a median destructive temperature between 270–460°C for magnetite-dominated samples (except for sample 2072, which had an MDT of 550°C), and 4) no large susceptibility changes below about 500°C. Samples which produced unsatisfactory paleointensity results had one or more of the following negative characteristics: 1) non-SD type behavior in the Lowrie-Fuller test, 2) a significant secondary remanence component, 3) a narrow unblocking temperature distribution with MDT greater than 500°C for magnetite-dominated samples, and 4) large susceptibility changes beginning at moderate (300°C) temperatures. Some of these samples also had wasp-waisted hysteresis loops. As discussed below, the reliability of the paleointensity data is related to whether wasp-waisted hysteresis loops represent the mixture of a primary magnetic mineral with secondary minerals from processes such as weathering.

It is not surprising that a sample with SD grains, a thermally stable mineralogy and a primary TRM provides good paleointensity results because these characteristics meet the theoretical requirements of the Thellier and Thellier (1959) method. A broad unblocking temperature distribution is also beneficial because it increases the temperature range from which high quality data can be retrieved (Cui and Verosub, 1995). However, it is also important to understand how the negative mineral magnetic characteristics affect the paleointensity studies.

4.1 Non-SD behavior in the Lowrie-Fuller test

It is well known that a sample with a TRM carried by SD grains is an ideal recorder of the direction and intensity of the geomagnetic field. However, multi-domain (MD) grains are regarded as poor recorders of paleointensity because the additivity of partial TRMs, which is needed for the Thellier and
Fig. 1. Data from sample 1290b. (a) Decay of NRM intensity with stepwise thermal demagnetization. (b) Vector component diagram from stepwise thermal demagnetization of NRM. Open (closed) symbols indicate projections onto the vertical (horizontal) plane. (c) Susceptibility change (%) during stepwise thermal demagnetization. (d) Susceptibility versus temperature (heating and cooling curves are indicated with arrows). (e) NRM-TRM plot from the Thellier-Thellier experiment. The NRM and the TRM are normalized to the value of the NRM at room temperature. (f) Lowrie-Fuller test with AF demagnetization of NRM and SIRM.
Fig. 1. (continued).
Thellier method, only holds approximately (Dunlop and Xu, 1994). Several studies (Coe, 1967; Levi, 1977; Collinson, 1983; Rubin and Moskowitz, 1994) have shown that samples with a significant fraction of MD magnetite do not give linear NRM-TRM plots. Despite these results, MD behavior has rarely been used as a rejection criteria in Thellier-Thellier experiments because it is difficult to determine the domain states for natural samples, particularly when the magnetic assemblage consists of a mixture of grain sizes. Our work indicates that the Lowrie-Fuller test can be used for this purpose. While the work of Xu and Dunlop (1995) implies that SD-type behavior on the Lowrie-Fuller does not necessarily indicate the predominance of SD grains, any non-SD behavior can be conservatively taken as evidence for the presence of MD grains. Additional magnetic methods such as low-temperature demagnetization (Dunlop and Argyle, 1991) can then be used to confirm this result.

One example of a sample which seems to have a significant fraction of MD grains is samples 1290b. All of the other magnetic properties of this sample are positive. A broad unblocking temperature distribution is evident in the thermal demagnetization curve (Fig. 1a) and no significant secondary component is seen in the vector component diagram (Fig. 1b). Susceptibility measurements indicate that there are no major changes in mineralogy below 500°C (Fig. 1c), and the \( \chi_T \) curve (Fig. 1d) indicates a Curie point of about 570°C, which suggests that magnetite is the dominant magnetic mineral. However, the NRM-TRM plot is non-linear and useful paleointensity results were not obtained (Fig. 1e). The Lowrie-Fuller test (Fig. 1f) shows that during AF demagnetization most of the remanence is removed at low fields, which is still a valid criterion for MD grains (Xu and Dunlop, 1995).

As discussed by Kovacheva and Toshkov (1994), the presence of MD grains is probably the explanation for the failure of the paleointensity experiments for two other bricks, samples 1286 and 1287, which belong to the same collection as sample 1290b although sample 1290b is the only one of these samples that shows non-SD behavior in the Lowrie-Fuller test. This suggests that samples 1286 and 1287 may be examples of MD grains that show SD-behavior in the Lowrie-Fuller test (Xu and Dunlop, 1995). There are other samples (33, 703-6 and 2028) which also display SD behavior and which produced unsatisfactory paleointensity results. Whether or not these samples fall in the same category is not clear. Thus, non-SD behavior on the Lowrie-Fuller test can be used to identify samples that are likely to produce unsatisfactory paleointensity results. However, the test cannot be used to identify all samples for which unsatisfactory paleointensity results arise from the presence of MD grains.

4.2 Persistent secondary components

The effect on a paleointensity determination of a secondary remanence component depends on the stability of that component or on its maximum unblocking temperature as well as the nature of the remanence. If the secondary component can be removed below 200°C, the Thellier-Thellier experiment may not be affected. However, if the secondary component persists above 350°C, the quality of the paleointensity data will be affected. If, in addition, mineralogical changes occur at high temperatures, the window of unblocking temperatures from which useful paleointensity results can be obtained could be quite narrow. This situation is likely to lead to rejection of a sample because the NRM-TRM plot must be linear over a certain range of temperatures. Sample 33 is an example of a sample with a significant secondary remanence component (Figs. 2a and 2b). The low temperature component is not removed until 500°C while the high temperature component persists until 600°C. Thus, the paleointensity value could only be deduced from data in the interval of 500–600°C. Generally, it is difficult to obtain a high quality paleointensity result in this temperature interval, as the data from sample 33 demonstrate (Fig. 2c).

Sample 2028 is interpreted as having three remanence components (Fig. 3a). One of these components has a maximum unblocking temperature of 200°C while the second has a maximum unblocking temperature of 450°C. Although there is a linear interval in the NRM-TRM plot between 230°C and 430°C (Fig. 3b), a paleointensity result from this interval would be unreliable because it is carried by a secondary component. After these components have been removed at 450°C, there is no linear interval on the NRM-TRM plot from which the paleointensity can be deduced. Thus the paleointensity data obtained from the temperature interval on the NRM-TRM plot between 230°C and 430°C should be
Fig. 2. Data from sample 33. (a) Decay of NRM intensity with stepwise thermal demagnetization. (b) Vector component diagram from stepwise thermal demagnetization of NRM (symbols are the same as in Fig. 1). (c) NRM-TRM plot from the Thellier-Thellier experiment.
Fig. 3. Data from sample 2028. (a) Vector component diagram (symbols are the same as in Fig. 1). (b) NRM-TRM plot from the Thellier-Thellier experiment.

treated with caution because the nature of this component is not known. Sample 65 also has two secondary components (Fig. 4a). One component is removed at 150°C while the other persists to 500°C. The primary component has a maximum unblocking temperature between 550°C and 600°C. It is unlikely that it is possible to obtain a reliable paleointensity data from such a narrow temperature window (Fig. 4b).

In addition to samples 33, 2028 and 65 which have been discussed above, samples 1174, 703-6 and 1508, also have persistent secondary components. The NRM-TRM plots for these samples are shown in Fig. 5. From these results, it appears that the presence or absence of a persistent secondary remanence component is a critical factor in determining the acceptability of the paleointensity data in this suite of samples, especially if the persistent secondary component is not carried by the primary TRM.

In principle, a secondary remanence component can be detected by AF and thermal demagnetization if the primary direction is different from the secondary direction. However, for some magnetic minerals, the secondary component may be highly resistant to AF demagnetization. For example, the AF demagnetization of sample 33 does not reveal a secondary component even though thermal demagnetization (Fig. 2b) of the same sample clearly isolates a strong secondary remanence component. In addition to being more effective in discriminating persistent secondary components, thermal demagnetization also
provides information about: 1) the thermal stability, as determined by susceptibility changes with temperature, 2) the unblocking temperature spectrum, and 3) the number of magnetic phases present in the sample. All of this information is of value in assessing the suitability of samples for the Thellier and Thellier method.

4.3 Susceptibility changes with temperature

Thermal alteration during heating is a major problem for the Thellier and Thellier method. Measurements of changes in susceptibility with temperature are one way to monitor for thermal alteration. Six samples that gave unsatisfactory paleointensity results also display large susceptibility changes below 500°C (Table 2). For example, the susceptibility of sample 33 changed 17.5% between room temperature and 350°C. The susceptibility of sample 703-6 changed 18% between 100°C and 450°C. Samples 43, 65, CpH1&1, and 1371 also showed greater than 10% susceptibility change during heating. Except for sample 2073, all of the well-behaved samples showed less than 10% susceptibility change. The susceptibility of sample 2073 increased by about 37% between room temperature and 550°C (Fig. 6), and we believe that its paleointensity determination should be rejected even though the result was originally rated as...
Fig. 5. NRM-TRM plots from the Thellier-Thellier experiment on samples: (a) 1174; (b) 703-6; and (c) 1508.
acceptable. We suggest that any sample that undergoes a susceptibility change of more than 10% between 100°C and 450°C should be discarded, regardless of its other magnetic properties.

4.4 Narrow unblocking temperature distribution

Cui and Verosub (1995) recently showed that for pottery samples, a broad unblocking temperature distribution can arise from a grain size distribution that spans the SP/SD boundary and that such an unblocking temperature distribution has a good chance of producing high quality paleointensity data. On the other hand, in principle, a narrow unblocking temperature distribution and its associated high MDT should not have an adverse effect on paleointensity determinations. However, as discussed above, the presence of a persistent secondary component and the occurrence of mineral alteration at high temperatures can significantly narrow the range of temperatures over which the NRM-TRM plot is linear. Thus, a sample with a MDT above 500°C has a poorer chance of producing high quality paleointensity data. In practice, acceptable paleointensity results are often obtained from the intermediate unblocking temperature interval (200°C–400°C) (Prevot et al., 1985; Pick and Tauxe, 1993; Cui and Verosub, 1995). For samples having magnetite as the only magnetic carrier, Cui and Verosub (1995) showed that the frequency-dependent susceptibility can be used to estimate the narrowness of the unblocking temperature distribution, provided there is no secondary component. However, in archaeomagnetic samples, magnetite is seldom the only magnetic carrier.

In this study, the MDT values are estimated from thermal demagnetization of the NRM. Due to the presence of secondary remanence components, some MDTs may be overestimated. High MDTs can indicate: 1) a uniformly fine grain size distribution, 2) the presence of secondary remanence components, 3) mixtures of magnetite and hematite, or 4) a combination of the above factors. Samples 1508 and 33 have MDTs of 500°C and 560°C, respectively. In both cases, secondary remanence components are responsible for the high values. For example, sample 1508 has a secondary component with unblocking temperatures up to 250°C (Fig. 7a). Because the secondary component is anti-parallel to the primary component, the true MDT is lower than 500°C. The presence of this strong secondary component explains why the high frequency-dependent susceptibility does not correspond to a broad unblocking temperature distribution and a relatively low MDT. For this sample, the susceptibility started to change at low temperatures (100°C) (Fig. 7b), possibly indicating thermal alteration, and there is no linear interval on the NRM-TRM plot (Fig. 5c). Another example of a sample with a narrow unblocking temperature distribution is sample 896, which has a high MDT (525°C) and which shows susceptibility changes above 500°C (Fig. 8a). Even though this sample has other magnetic properties that seem to be positive for paleointensity determination, it produced
unsatisfactory data, and the Thellier-Thellier experiment on this sample was aborted at 350°C because there was no evidence of linearity in the NRM-TRM plot (Fig. 8b).

A high MDT alone is not sufficient reason to reject a sample because determination of the MDT can be influenced by the presence of a strong secondary component. However, a sample with high MDT and with susceptibility changes at moderate temperatures is unlikely to produce high quality paleointensity data, especially when there is no secondary component and only one magnetic mineral is present. One exception to this statement would be a sample with a significant fraction of magnetic minerals with a high Curie temperature. Thus, MDT should be used with caution as a criterion for sample selection, and it should always be used in conjunction with other mineral magnetic parameters.

4.5 Wasp-waisted hysteresis loops

A wasp-waisted hysteresis loop requires the coexistence of two contrasting coercivity components. These two components can result from a mixture of two different grain size assemblages of the same magnetic mineral or from a mixture of two different magnetic minerals (Roberts et al., 1995). In some cases, samples with wasp-waisted hysteresis loops have produced high quality paleointensity data (Pick...
Fig. 8. Data from sample 896. (a) Susceptibility changes (%) during stepwise thermal demagnetization. (b) NRM-TRM plot from the Thellier-Thellier experiment.

Mineral magnetic studies of these samples indicate that the wasp-waisted loops are due to a mixture of SD and SP magnetite grains. In the present study, however, samples with wasp-waisted hysteresis loops proved unsatisfactory for paleointensity determinations. For these samples, thermal demagnetization of a three-axis IRM indicates a large decrease in remanence between room temperature and 240°C for the high and intermediate coercivity components (Fig. 9a). The weak remaining remanence held by these components persisted to temperatures between 650°C and 700°C, which indicates the presence of hematite. The low coercivity component has a maximum unblocking temperature between 550°C and 600°C which indicates the presence of magnetite. A likely candidate for the third component of the mineralogy is goethite. The maximum unblocking temperature of goethite ranges from 60°C to 120°C (Strangway et al., 1968; Dekkers, 1988, 1989), but goethite dehydrates during heating at temperatures as low as 50°C (Goss, 1987), leading to complete transformation into hematite at about 250°C (Wolska and Schwertmann, 1989). We interpret the thermal unblocking of the hard and intermediate components at temperatures up to 240°C to be due to unblocking of goethite, accompanied by goethite dehydration, hematite formation, and partial unblocking of hematite (see Roberts et al., 1995). Above 240°C, the hard component appears to be fully converted to hematite, which
has a maximum unblocking temperature between 650° and 700°C (Fig. 9a). Lack of saturation even at 2.5 T (Fig. 9b) and high stability against AF demagnetization are additional evidence for the dominance of a high-coercivity mineral, which based on the unblocking temperature data, is most likely to be goethite.

Most of the samples with wasp-waisted hysteresis loops listed in Table 1 are bricks. Goethite can be expected as a product of weathering in these samples. Three of the samples with wasp-waisted hysteresis loops clearly have secondary magnetization components, and in two cases (2028 and 703-6) the secondary components are likely to be carried by goethite because this component is removed below 240°C (Fig. 10a) and the high and medium coercivity IRM components decrease significantly below 240°C (Fig. 10b). For the other samples with wasp-waisted hysteresis loops, it may be that the secondary overprint is in the direction of the primary component. In addition, goethite has a weak magnetic moment, and a significant volume of goethite must be present with magnetite in order to produce a wasp-waisted hysteresis loop (Roberts et al., 1995). This conclusion is consistent with the fact that the high coercivity IRM component dominates the total IRM at low demagnetization temperatures.

If it is correct that the secondary low-temperature remanence component is due to the presence of goethite, then we are dealing with a chemical alteration that occurred before the Thellier-Thellier experiment was conducted. The paleointensity data from sample 1371 should therefore be unreliable even
though it has a linear interval on the NRM-TRM plot. As mentioned previously, a linear NRM-TRM plot does not guarantee the reliability of the data (Prevot et al., 1985). Partial TRM checks should be used to detect chemical changes due to heating of goethite because it will be unstable when heated in air. Although there are tests to monitor for chemical changes during the Thellier-Thellier experiment, no method has been devised to check for alteration that occurred prior to the experiment. Additional work is needed to understand the degree to which such alteration will affect the paleointensity determination.

The above results and discussion indicate that materials which yield wasp-waisted hysteresis loops will provide unreliable paleointensity data if the wasp-waisted hysteresis behavior is related to secondary, thermally unstable, magnetic phases. This observation provides additional evidence for the importance of mineral magnetic studies for screening samples for paleointensity investigations.

5. Summary

Our results indicate that any sample with a significant fraction of MD grains or a significant secondary component is unlikely to be suitable for paleointensity determination. In addition, samples with
narrow unblocking temperature distributions and moderately high MDTs are also likely to be a problem if changes in susceptibility occur at high temperatures. In order to screen paleointensity samples for these undesirable characteristics, three experimental tests need to be conducted: 1) stepwise thermal demagnetization of the NRM, with susceptibility measurement after each thermal step, 2) a Lowrie-Fuller test (or determination of hysteresis parameters), and 3) thermal demagnetization of a three-axis IRM. These tests give information above the domain state, number and nature of magnetic mineral phases, unblocking temperature distributions (including MDT), and the thermal stability of the magnetic mineral(s). All of these tests can be carried out with conventional paleomagnetic equipment.

To perform the tests, two specimens are required from each sample. One specimen can be used for thermal demagnetization (and the associated susceptibility measurements), the other can be used for the Lowrie-Fuller test and then for the thermal demagnetization of the three-axis IRM. Any sample with one or more of the following magnetic properties should be rejected: 1) non-SD behavior in the Lowrie-Fuller test, 2) persistent secondary components with high (above 350°C) unblocking temperatures, 3) significant susceptibility changes beginning at moderate temperatures (300°C), 4) narrow unblocking temperature distribution with MDT greater than 500°C and significant susceptibility change below 500°C, and 5) magnetic minerals resulting from secondary processes such as weathering. While these criteria will not eliminate all bad samples, we believe that they will significantly improve the success rate of the method of Thellier and Thellier (1959), and that they will be aid in the interpretation of the paleointensity data. Further work should be done on larger collections to refine these simple criteria.

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