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Palaeomagnetic cleaning strategies

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ABSTRACT

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Cleaning is a term used in palaeomagnetism to refer to laboratory methods designed to demagnetize preferentially the less stable components of magnetization. Although the diversity of methods suggests that there is a wide choice available, this is illusory. Except in simple cases, the wrong cleaning strategy may yield misleading results. Cleaning methods include thermal, magnetic (alternating field or a.f.), low-temperature, chemical, shock and microwave cleaning. A cleaning strategy involves one or more cleaning methods applied in a specific sequence. The design of a strategy is made easier with the a priori knowledge of magnetic mineralogy and granulometry, or in other words, rock magnetic properties. Although not a substitute for other rock magnetic experiments, the variation of low-field susceptibility with temperature (k–T) not only provides information on magnetic mineralogy and granulometry, but it also draws attention to chemical alteration that may occur during thermal cleaning.

Apart from the rapid, though reversible, decrease of k associated with Curie temperatures, other diagnostic k–T features include the low-temperature (about −140°C) peak typical of multidomain (MD) magnetite, the high-temperature (Hopkinson) peak typical of fine-grained magnetite (and other magnetic minerals) and the classic lepidocrocite–maghemite–haematite profile, which is irreversible.

Examples are given where the remanence caused by lightning can be effectively eliminated only by applying a.f. or low temperature (LT) pre-treatment. Such remanence may be an isothermal remanent magnetization (IRM), or a combination of IRM and anhysteretic remanent magnetization (ARM). As the unblocking temperature of relatively low-coercivity MD grains may extend to high-temperature, it is often desirable to suppress MD remanence. An example is given of palaeointensity determination, with and without LT pre-treatment. The nature of the LT transition is briefly addressed in the light of a rare double low-temperature peak, which may reflect both the isotropic point and the Verwey transition.

1. Introduction

In palaeomagnetic studies, cleaning is the process whereby components of natural remanent magnetization (NRM) are selectively demagnetized. The resolution of these components depends critically on choosing the most efficacious cleaning technique. Traditionally, magnetic cleaning using an alternating field (a.f.) was used for (titano)magnetite- and pyrrhotite-bearing samples whose coercivities were attainable with standard equipment (As and Zijderveld, 1958). Samples containing high-coercivity minerals, such as haematite and goethite, were thermally (Irving et al., 1961) or chemically (Collinson, 1965) demagnetized. These are usually referred to as thermal or chemical cleaning respectively. Most workers have abandoned this over-simple approach for more judicious procedures to recover as much information as possible. Considerable effort is spent experimenting with various schemes to improve data quality. To this end, the a priori knowledge of magnetic mineral type(s) and grain size distribution is of great benefit in designing a cleaning strategy. This information can be acquired by combining results of a number of rock

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magnetic experiments and observations. For instance, a.f. demagnetization, anhysteretic remanent magnetization (ARM) and high-field hysteresis are particularly useful for determining domain structure, or granulometry, in (titano)magnetite bearing rocks (Dunlop, 1983). Thermal demagnetization of orthogonal isothermal remanent magnetizations (Lowrie, 1990) is a useful technique for identifying magnetic phases, particularly magnetically weak minerals such as haematite or goethite. A technique which has the advantage of determining both granulometry and mineral species is the monitoring of low-field susceptibility (k) from liquid nitrogen temperature to the Curie temperature. This method also alerts the investigator to chemical alterations which may interfere with thermal cleaning. The purpose of this paper is to discuss k–T analyses in the context of palaeomagnetic cleaning and to suggest that they should become a routine practice before committing valuable sample collections to cleaning processes.

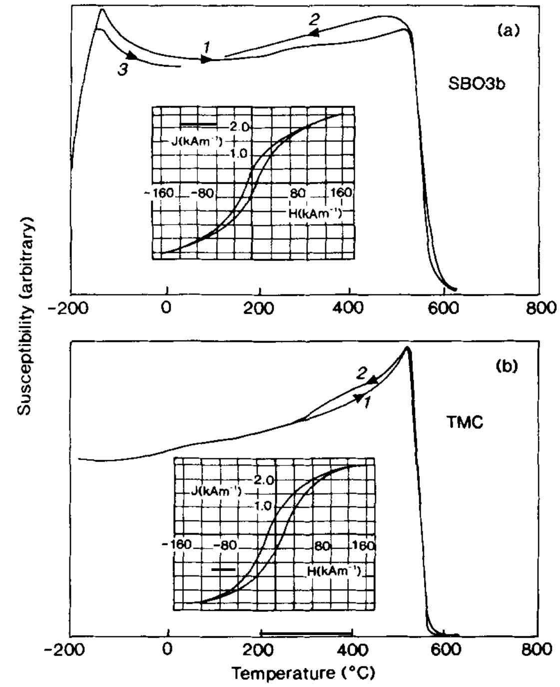

Fig. 1. Low-field susceptibility variation with temperature ($k-T$) with hysteresis as inserts. (a) MD magnetite in dolerite of the Stuart Dykes (central Australia); (b) SD magnetite produced by heating sandstone containing siderite from Tahmoor (NSW). The numerals refer to heating sequence.

2. Thermal variation of low-field susceptibility (k–T)

In the late 1960s and 1970s, Radhakrishnamurty and coworkers utilized the variation of susceptibility with temperature (k–T) to infer granulometry (e.g. see Radhakrishnamurty et al., 1977). They showed that there was indeed useful information in k–T analyses, although some of their interpretations were often questioned and have since been shown to be erroneous (Senanayake and McElhinny, 1981; Clark and Schmidt, 1982).

The instrument used here to measure k–T is an adaptation of the transformer bridge reported by Ridley and Brown (1980). The original instrument yields precisions of better than 1% in the range 10^{-4}–1 SI (about 8×10^{-6} to 0.08 G Oe^{-1}), decreasing to 10% at 5×10^{-6} SI (4×10^{-7} G Oe^{-1}). The addition of a water-jacket and furnace, and the necessarily smaller sample size (about 1 cm^3), effectively reduce the usable range to susceptibilities above 5×10^{-4} SI (4×10^{-5} G Oe^{-1}). The temperature is measured with a resistive thermal device (RTD) with a linear output from −200°C to 800°C. Both temperature and susceptibility are recorded on a personal computer which also controls the heating rate. From liquid nitrogen (LN$_2$) temperature (−197°C) to room temperature the sample warms naturally, although the holder can be pre-cooled to slow the warming if required.

Figure 1 shows two k–T curves for almost pure magnetite, both being essentially reversible, indicating little chemical change, and displaying sharp Curie temperatures close to 580°C. Another conspicuous feature is that one sample shows a pronounced peak at low temperature whereas the other sample shows a pronounced peak at high-temperature. The curve showing the peak near −130°C to −150°C (Fig. 1(a)) is for a dolerite sample from the Precambrian (1050 Ma) Stuart Dykes from central Australia. This peak is usually attributed to the isotropic point at about −140°C, where the first magnetocrystalline anisotropy constant K_1 for magnetite changes sign. Because of thermal gradients it is difficult to obtain the temperature accurately with the present set-up. Nevertheless, anisotropy of magnetocrystalline origin implies the presence of multidomain (MD) grain sizes. However, the inset in Fig. 1(a) shows that the hysteresis is dominated by single-domain/pseudo-single-domain (SD/PSD) grains. In a field of 160 kA m^{-1} (2000 Oe), ratios of near saturation remanence to saturation magnetization (J_{rs}/J_s) of 0.17 and coercivity of remanence to bulk coercivity (H_{cr}/H_c) of 1.9 are SD type (Dunlop, 1983). Although full hysteresis loops have not been measured in higher fields, J_{rs}/J_s and H_{cr}/H_c have been estimated as 0.11 and 2.0, respectively, in a field of 1.6 MA m^{-1} (20000 Oe) using a cryogenic susceptometer. These values are more hybrid in character and suggest a mixed population of PSD and MD grains.

Recently, Hodych (1991) suggested that the low-temperature peak is primarily due to the crystallographic Verwey transition, about 14°C below the magnetocrystalline isotropic point. The Verwey transition is the change that magnetite crystals undergo from cubic to a lower symmetry form. It should also be noted that although Hodych observed these changes via the temperature variation of saturation isothermal remanent magnetization (SIRM), the properties of IRM are well known to be different from those of thermoremanent magnetization (TRM) and its laboratory analogue, anhysteretic remanent magnetization (ARM). Although Hodych did not discuss the implications of the predominance of the Verwey transition in terms of grain size, the above conclusion that a susceptibility peak at about −140°C indicates MD grains remains valid. This is because the shape anisotropy of the SD fraction is thermally insensitive. The subject of the isotropic point vs. the Verwey transition is encountered again below in Section 4. The minor Hopkinson peak at temperatures just below the Curie temperature is also a diagnostic feature of MD magnetite, for which variations in intrinsic susceptibility with changing temperature are suppressed by self-demagnetization. That the Hopkinson peak is smeared on cooling indicates that some small chemical change or annealing has occurred at T_C.

The susceptibility peak at temperatures just below T_C in Fig. 1(b) is for a sample of fine sandstone that had been heated previously to 600°C in the laboratory. A copious amount of very fine-grained magnetite was produced by this heating, presumably through the breakdown of the siderite and the buffering action of organic material present (the sample is from a colliery near Tahmoor, New South Wales). The absence of a low-temperature peak indicates that shape anisotropy is dominant. The steady increase of susceptibility with temperature, culminating in a well-defined unblocking or Hopkinson peak, reflects the presence of superparamagnetic (SPM) grains. The susceptibility of SPM particles increases greatly at their unblocking temperature. These properties are indicative of very fine grained magnetite, mostly SD at room temperature. The inset of Fig. 1(b) showing near-saturation (at 160 kA m^{-1} or 2000 Oe) hysteresis supports the evidence for SD grains from k–T analysis. Ratios of J_{rs}/J_s (0.25) and H_{cr}/H_c (1.6) are typically SD (Dunlop, 1983). At higher fields of 1.6 MA m^{-1} (20000 Oe), using a cryogenic susceptometer, J_{rs}/J_s was estimated as 0.19 and H_{cr}/H_c as 1.5, again typically SD.

To a casual observer, the hysteresis loops displayed in Figs. 1(a) and 1(b) appear to be similar. However, this similarity is superficial, and a closer examination suggests that sample SDO3b is mixed SD and MD, whereas sample TMC is predominantly SD. The k–T curves suggest that the former is mainly MD, whereas the latter is mixed SD and SPM. It is apparent that the susceptibility is dominated by the softer fractions whereas (room temperature) hysteresis is biased towards the harder fractions. This emphasizes the complementary roles of k–T analysis and hysteresis loops in rock magnetism. Nevertheless, it is worth noting that both the type of magnetic mineral present and a qualitative notion of the grain sizes present can be gained from k–T analysis.

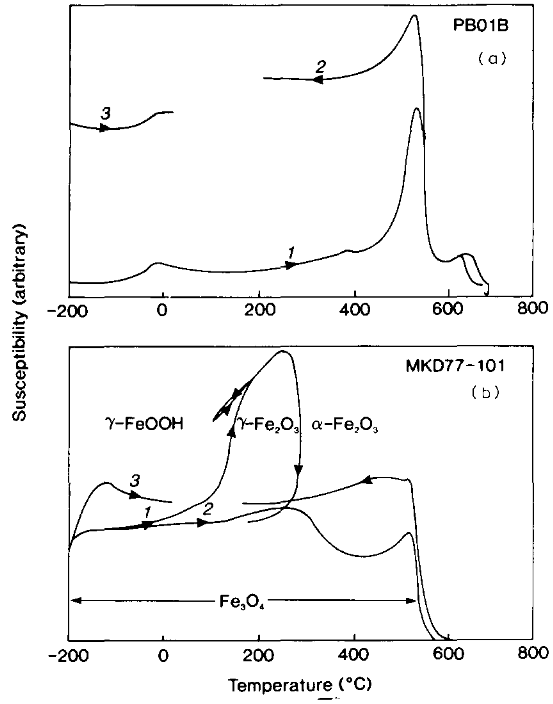

Fig. 2. k–T curves: (a) haematitic iron ore from Paraburdoo (Pilbara, W.A.); (b) partially weathered greenstone from Mt. Keith (Yilgarn, W.A.). Numerals as for Fig. 1.

The usefulness of k–T analysis for detecting chemical alteration during heating is graphically displayed in Fig. 2. The first example is of an iron ore (Paraburdoo, Western Australia, Fig. 2(a)). This ore is composed of coarse haematite, which is perfectly antiferromagnetic with a very weak susceptibility, below the Morin transition at about −20°C. Above the Morin transition a small but detectable susceptibility appears which remains fairly level until above 400°C, when an irreversible reaction occurs producing SD (±PSD) magnetite. It should be noted that the enhanced peak during heating reflects both Hopkinson unblocking of fine-grained magnetite and the actual formation of the magnetite. The Curie temperature of the haematite is attained at about 675°C. The thermal hysteresis displayed by the k–T curve at this temperature is thought to be due to more rapid cooling of the RTD sensor as compared with the sample, rather than a chemical change of the haematite. On cooling the sample displays a characteristically well-defined Hopkinson peak at about 550°C. The susceptibility remains flat on further cooling, although the susceptibility is well above that on heating. Apart from the Morin transition, no low-temperature features are present on the repeat warming from LN_2 temperature. This is consistent with the magnetite being SD. Considerable problems would be anticipated during thermal cleaning of this sample if special precautions to shield the sample from extraneous magnetic fields were not taken. The magnetite is magnetically much stronger than the haematite and it is fine grained, probably containing sufficient 'SPM' grains to acquire spurious magnetization components with decay times at least as long as the measurement period (it is pedantic to differentiate between SPM grains and those carrying viscous remanent magnetization (VRM) when discussing phenomena on the laboratory time-scale). Although easily detected by variations in k–T, the magnetite produced on heating was not detectable by X-ray diffraction (XRD). The precise origin of the magnetite is unknown.

More spectacular than the production of magnetite described above is the creation and inversion of maghemite in a drill-core sample of partially weathered greenstone from Mt. Keith (Yilgarn, W.A.), shown in Fig. 2(b). On warming from LN_2 temperature the susceptibility increases steadily to about 125°C, reminiscent of SD magnetite of Fig. 1(b). However, above 125°C the slope suddenly rises until 200°C, where it suddenly decreases to a slope comparable with that before the sudden rise. This increment of susceptibility can be shown to be due to the creation of magnetic material by cooling from 200°C to about 100°C and observing that the curve is irreversible. On reheating, the curve is reversible and has a constant slope up to 270°C. Notwithstanding the susceptibility increment, the early slope and the slope of the cooling–reheating loop appear to be sub-parallel, suggesting that the heating has created more of the material that existed before heating. At about 270°C the susceptibility begins to plummet. This is not a T_C but an irreversible change, as shown by the cooling from 300°C to

200°C, where the susceptibility continues to decrease.

On recooling the sample to LN_2 temperature and reheating continuously to above 600°C, the irreversible change recommences at about 300°C and is complete at 400°C, leaving fine-grained magnetite as indicated by the unblocking peak at about 550°C. On cooling, more magnetite is produced, as indicated by the increased susceptibility, and the Hopkinson peak is suppressed, suggesting a greater effective grain size. Finally, a third warming from LN_2 temperature reveals a low-temperature peak, confirming the presence of MD magnetite.

On XRD analysis maghemite peaks are much stronger in material that has been heated to about 200°C than in unheated material. Evidently, the irreversible increase corresponds to the creation of maghemite and the irreversible decrease corresponds to the inversion of maghemite to much less magnetic haematite. Although the maghemite precursor could not be identified confidently, from XRD it may be somewhat impure lepidocrocite. Similar alteration has been suggested as the cause of non-linear Thellier palaeointensity data (Barbetti et al., 1977). Core samples taken from greater depths in the same drill-hole were found to contain only MD magnetite. It is concluded therefore that although the present sample was taken from over 100 m depth, it contained lepidocrocite related to magnetite that has oxidized and hydrated during weathering. The lepidocrocite dehydrates to maghemite between 120 and 200°C, and this in turn inverts to haematite above 270°C. In our laboratory we now recognize this shaped curve as the arch-typical lepidocrocite–maghemite–haematite curve, invariably observed in samples collected from outcrop or from (relatively) shallow depth from drill-core.

Recently, other workers have reported similar maghemite creation/inversion curves. Orlicky (Fig. 6, 1990) showed a very similar curve to Fig. 2(b) between 0 and 400°C. Although he did not discuss maghemite creation, or its relationship to weathering, Orlicky identified the abrupt decrease with inversion to haematite. Thomas (1991) also identified this type of curve with maghemite.

3. Cleaning strategies

Although of intrinsic interest from a purely rock magnetic point of view, the foregoing information is also of critical importance regarding remanence cleaning, and whether to use a.f., thermal or chemical cleaning, or some combination. Having identified the magnetic mineralogy and grain size ranges, the next step is to examine NRM direction and intensity and susceptibility distributions. Planar NRM distributions suggest partial overprinting. Unimodal NRM distributions usually suggest almost complete overprinting, or the complete absence of partial overprinting. Of course, there are exceptions to all rules, as workers in the Appalachians have learned recently (Kent and Opdyke, 1985). Many redbeds may be almost completely remagnetized through chemical processes that almost obliterate the characteristic remanence. A unimodal distribution is attained through standard thermal cleaning, but only with small temperature increments just below T_C is the characteristic remanence isolated. Thus, it is with some trepidation that I attempt to set out below some guidelines for cleaning strategies. There will always be exceptions.

3.1. Thermal cleaning and thermal pre-treatment

The remanence in igneous rocks is usually thermally related. The initial TRM may be partially overprinted during burial and uplift. This overprint may be blocked on cooling, in which case it would be best removed by thermal demagnetization, or it may be related to chemical alteration, in which case there are no firm rules on which technique is best. Ouliac (1976) found that thermal and not a.f. cleaning was effective in removing a VRM. Another example of the success of thermal cleaning and failure of a.f. cleaning to remove what is essentially a thermal overprint was given by Schmidt and Embleton (1981) for the Jurassic(?) Hornsby Breccia (N.S.W.). On the other hand, the nearby Dundas Breccia, which has suffered a similar burial/uplift history, has undergone overprinting related to both thermal and chemical processes (Schmidt, 1982). Thermal cleaning of some samples from the Dundas Breccia yields Z-shaped orthogonal projections, with the first and third leg related to overprinting and the middle leg related to the characteristic magnetization. This Z signature has been found elsewhere and attributed to similar causes (Schwarz and Buchan, 1989; Mushayandebvu, personal communication, 1990; Schmidt, 1990). That thermal demagnetization works at all in these cases is largely fortuitous. Although chemical processes are renowned for producing grain populations with smeared unblocking temperatures, their coercivity spectra may be even more smeared, leaving thermal cleaning as the best prospect.

Other special cases can arise that call for partial thermal cleaning. A limestone that contains secondary goethite and minor detrital magnetite may be best treated by breaking down the high-coercivity goethite at 120°C, then continuing with a.f. cleaning, thus avoiding chemical alteration common to limestones at higher temperatures (B. McClelland, personal communication, 1991).

3.2. a.f. cleaning and a.f. pre-treatment

Clearly, samples whose magnetic minerals display gross chemical or structural changes demand particular care on thermal cleaning. Such samples may respond better to a.f. and, as a general rule, at least some samples of any particular collection ought to be magnetically cleaned. Dinarès-Turell and McClelland (1991) have found a pathological

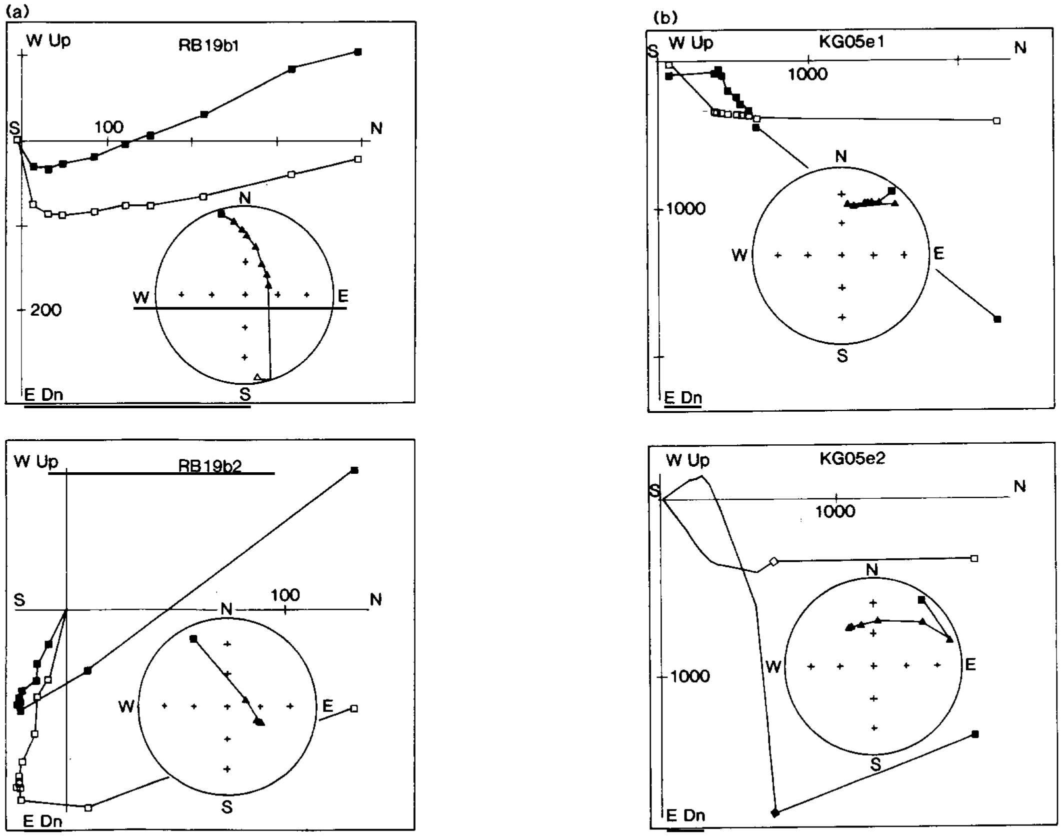

Fig. 3. Orthogonal and stereographic demagnetization plots for untreated (upper) and pre-treated (lower) (a) Mt. Roe Basalt (Pilbara, W.A) and (b) dolerite of the Kulgera Dykes (central Australia). The Mt. Roe Basalt sample had a.f. pre-treatment and the Kulgera Dyke sample had a low-temperature (LT) pre-treatment. Solid symbols refer to the horizontal, and open symbols refer to the vertical. (Units are in mA m^{-1} (μG).)

case where the unblocking temperature spectra of two components were identical, and on thermal cleaning gave the appearance of a single component. The overlapping of unblocking temperature spectra is typical of chemical remanent magnetization processes, although not usually to this extreme.

High intensities and, more particularly, high Königsberger ratios may indicate the effects of lightning. In these cases, although *k–T* behaviour is ideal (Fig. 1(a)), thermal cleaning by itself is not recommended. Pre-treatment using a.f. cleaning is called for. Figure 3(a) compares thermal vs. a.f./thermal cleaning of two specimens from the same lightning-affected sample (Schmidt and Embleton, 1985). Clearly, an endpoint is reached only if the sample is pre-treated with a.f., in this case to 25 mT (250 Oe). As *k–T* analysis showed no irreversible chemical changes, thermal cleaning was used after the a.f. Finishing with thermal demagnetization avoids problems associated with gyroremanent magnetization (GRM) or rotational remanent magnetization (RRM). Although the lightning induced component has a discrete, or limited coercivity spectrum, its associated unblocking temperature is broad. This is typical of MD grains (McClelland and Suguira, 1987) and would suggest that low-temperature (LN_2) cleaning may be just as effective as a.f. cleaning for lightning-affected samples, or indeed for any case where the broad unblocking temperature spectrum of MD magnetite grains obscures the fidelity of the SD/PSD record. This is discussed below.

3.3. *Low-temperature (LT) pre-treatment*

Overprints carried by MD magnetite may be eliminated or suppressed by exploiting low-temperature transitions. That is, cooling to LN_2 temperature and rewarming in zero field selectively removes remanence carried by MD magnetite. Kobayashi and Fuller (1968) have described in some detail how remanence of MD material behaves on thermal cycling. Figure 3(b) compares thermal vs. LT/thermal cleaning of two specimens from another lightning-affected sample (Camacho et al., 1991). Here an endpoint is reached only if the samples are given LT pre-treatment. It should be noted that specimen KG 05e1 in Fig. 3(b) not only does not reach an endpoint, but actually retraces its path on the stereonet. On the orthogonal projection this is manifested as a Z signature, as discussed above in Section 3.1. The difference here is that the *k–T* behaviour is ideal (similar to that depicted in Fig. 1(a)), and the Z signature results from lightning effects rather than chemical effects.

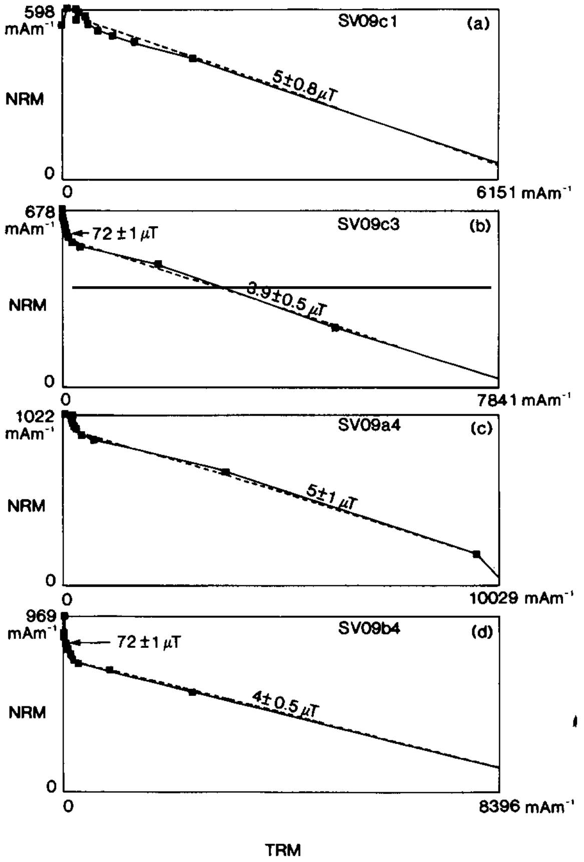

Fig. 4. Modified Thellier palaeointensity NRM/TRM plots for Early Devonian Snowy River Volcanics (Victoria): (a) and (c) untreated samples; (b) and (d) samples with low-temperature (LT) pre-treatment. (Units are in mA m^{-1} (μG) and μT (0.01 Oe).)

Palaeointensity determinations may also benefit from LT pre-treatment by suppressing the MD contribution. This was attempted using some Early Devonian volcanics which displayed ideal *k–T* behaviour (i.e. similar to Fig. 1(a)) and showed no effects of lightning. Although the volcanics were known to possess a thermal overprint, the characteristic component is pre-folding and is likely to be primary TRM (Schmidt et al., 1987). Therefore, multicomponent palaeointensity determinations might be possible. Devonian palaeointensities are especially important, as some workers have determined very low values for this time (Didenko and Pechersky, 1989). The modified Thellier method was used on separate specimens that were both untreated and LT pretreated. Of course, once treated at LT, specimens were retreated after each partial TRM acquisition stage. The results for specimens from two samples are plotted in Fig. 4. The untreated specimens both give very non-linear segments for the lower-temperature portions and no palaeointensity associated with the overprint could therefore be determined. The high-temperature portions, however, yield very low palaeointensities of 5 ± 1 μT (0.05 ± 0.01 Oe) especially considering the fairly steep palaeoinclinations of 58°; this result supports the claims of Didenko and Pechersky (1989). The pre-treated specimens also yield very low palaeointensities of 4 ± 0.7 μT (0.04 ± 0.007 Oe), but in addition yield well-determined palaeointensities associated with the overprint values of 72 ± 1 μT (0.72 ± 0.01 Oe). This value is close to present-day polar values, consistent with the very steep palaeoinclinations of 75°. Although confirmation using historic lava flows is required, the suppression of MD magnetization by LT pre-treatment appears to produce better linear behaviour of partial TRM demagnetization/acquisition during modified Thellier palaeointensity determination.

Traditionally, it is asserted that the mechanism of LT demagnetization is related to the magnetite isotropic point, K_1, changing sign at about −140°C. As mentioned above, Hodych (1991) recently questioned this mechanism on the basis of temperature of demagnetization of SIRMs, which seems to occur closer to −150°C, and is more consistent with the Verwey crystallographic transition. During routine k–T analysis of dolerite dykes from central Australia, some very unusual and rare behaviour has been observed which may have some bearing on this issue. Occasionally, a double LT peak appears, which is reproducible even after heating to above T_C for magnetite (Fig. 5). The lower temperature peak, supposedly associated with the Verwey transition, is small compared with the higher-temperature peak, supposedly associated with the isotropic point. The inference from these k–T observations is that most of the remanence unblocking (and hence demagnetization if in zero field) is associated with the isotropic point. On closer inspection of other LT peaks (e.g. Fig. 1(a)), a slight inflection is discernible on the low-temperature side which now seems likely to be due to the Verwey transition. Therefore, most LT peaks observed in k–T curves are probably unresolved Verwey transitions and isotropic points. However, by far the largest amount of unblocking is associated with the isotropic point. The k–T observations and Hodych's IRM observations are yet to be reconciled.

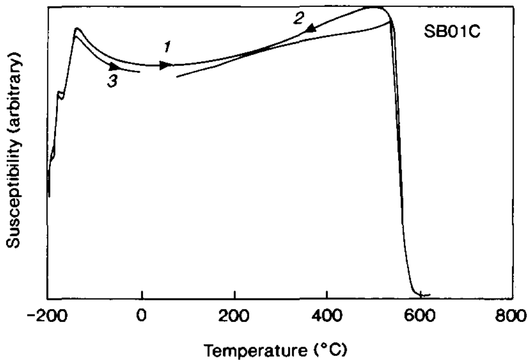

Fig. 5. k–T curve for MD magnetite in dolerite of the Stuart Dykes (central Australia). This sample displays the rare resolution of the Verwey transition and the isotropic point (compare Fig. 1(a), where the peaks are merged).

4. Conclusions

Palaeomagnetic cleaning is laborious and time consuming, and every effort to reduce this burden should be encouraged. A better knowledge of the rock magnetic properties of minerals is invaluable. One of the most rewarding techniques is k–T analysis, which not only identifies minerals but also yields unblocking temperatures, chemical alteration products and qualitative information on grain size ranges.

Chemical alteration may produce grains with broad unblocking spectra, but even broader a.f. spectra. Lightning may activate a relatively narrow coercivity range, but a very broad unblocking temperature range. The Z signature on orthogonal projections probably indicates an inappropriate cleaning procedure, and particular care should be exercised. For samples collected in areas prone to lightning strikes, a.f. pre-treatment is strongly recommended.

Low-temperature (LT) pre-treatment is also useful for cleaning lightning-affected samples, or whenever remanence carried by MD grains is to be suppressed. This appears to improve palaeointensity determinations. The problem of which plays the greater role in LT demagnetization, the isotropic point or the Verwey transition, seems to be one of degree rather than kind. Both may be seen to play a role, although, unlike results of IRM demagnetization, results of k–T analysis show that the isotropic point is dominant.

Acknowledgements

Largely through the inspiration of David Clark, the application of rock magnetic properties to palaeomagnetic studies have become routine practice in our laboratory. Mark Huddleston interfaced the k–T bridge to a personal computer, which led directly to increased productivity and allowed the k–T bridge to run without continual operator attendance. David Clark and Mark Lackie are thanked for their suggested improvements to the draft. The referees are also thanked for their constructive criticisms. Bruce Simons and Alfredo Camacho (formerly of the NTGS) are thanked for providing the samples of the Stuart Dykes, the Kulgera Dykes and other dolerites. I am grateful to David French for providing the XRD analyses.

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