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Low-temperature demagnetization of volcanic rocks containing multi-domain magnetic grains: Implications for the Thellier paleointensity determination

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RESUMEN

Seleccionamos trece muestras de unidades volcánicas previamente estudiadas del Cáucaso, Islandia y las Filipinas para usarse en este estudio de estructura de dominio magnético y desmagnetización de baja temperatura. Se aplicó el tratamiento de baja temperatura a las magnetizaciones termoremanentes parciales para remover la parte de magnetización remanente portada por granos multi-dominio. En general, 10 a 40% de la 'termoremanencia parcial multi-dominio' fue removido con este tratamiento, lo que puede ayudar a incrementar el éxito de las mediciones de paleointensidad. Las muestras de Filipinas provienen de la erupción del volcán Pinatubo y muestran un fenómeno de auto-inversión total debido a la presencia de ilmeno-hematitas y una auto-inversión parcial entre 500° y 575°C debido a las titanomagnetitas. La desmagnetización de baja temperatura aplicada por primera vez a remanencia auto-inversa muestra que el pico de alta temperatura es casi eliminado por el tratamiento.

PALABRAS CLAVES: Magnetismo de rocas, desmagnetización de baja temperatura, estructura de dominio.

ABSTRACT

Thirteen samples of volcanic rocks from Caucasus, Iceland and Philippines were subjected to low-temperature treatment in order to remove the part of remanent magnetization carried by multi-domain grains. Ten to 40% of multi-domain partial thermoremanence were removed, which may help to increase the success of paleointensity experiments. A sample from the 1991 eruption of Pinatubo volcano shows a total self-reversal due to ilmeno-hematites and partial self-reversal between 500° and 575 °C due to titano-magnetites. Low temperature demagnetization applied to self-reversed remanence suggest that the high-temperature peak is almost suppressed by this treatment.

KEY WORDS: Rock-magnetism, low-temperature demagnetization, domain structure.

INTRODUCTION

Reliable absolute paleointensity results are generally much more difficult to obtain than reliable directional data. Only volcanic rocks that satisfy certain specific magnetic criteria can be used for paleointensity determination (Kosterov and Prévot, 1998). Thellier and Thellier (1959) provided a method to determine the intensity of the geomagnetic field on volcanic rocks and archeological materials carrying thermoremanent magnetization (TRM). This method is considered to be reliable (Goguitchaichvili *et al.,* 1999a). However, several conditions have to be obeyed to ensure the significance of the paleointensity results:

1. The primary remanent magnetization must be a TRM and must not have decayed.

2. The secondary components must be weak with respect to the primary component and must be removed at relatively low temperatures.

3. The remanence must be carried mainly be single-domain magnetic grains to ensure the independence of the partial thermoremanent magnetizations (pTRM) (Thellier and Thellier, 1944, 1959).

4. No chemical/magnetic changes during laboratory heatings.

Conditions 1 and 2 are fulfilled by a significant fraction of volcanic rocks. The magnetic carriers in volcanic rocks selected for paleointensity experiments is commonly a Ti-poor titano-magnetite spinel formed from spinodal decomposition of a original Ti-rich titano-magnetite. The grain size of this spinel phase is generally larger than single-domain/pseudo-single-domain (SD/PSD) threshold (Kosterov and Prévot, 1998). This makes impossible the determination of geomagnetic paleointensity for most natural volcanic rocks.

In order to increase the number of paleointensity determinations, Kono and Ueno (1977) proposed applying a field perpendicular to the natural remanent magnetization (NRM) direction. It is possible to extract both NRM and TRM by performing a single heating at each step. This technique requires exceptionally stable components and involves large experimental errors. Hoffman *et al.* (1989) suggested to use sub-samples, heated to different temperatures. Sherwood (1991) evaluated this approach and found no improvement compared with the Thellier technique. Pick and Tauxe (1993) used submarine basalt glasses but no paleodirections are known for these rocks. Herrero-Bervera and Valet (2000) performed paleointensity experiments involving alternating field demagnetization on seven contemporary lava flows from Hawaii. Thirty per cent of NRM-TRM curves were perfectly linear and thus appropriate for absolute intensity experiments.

The present work is an effort to try to eliminate the fraction of remanence carried by multi-domain magnetic grains and thus increase the success of Thellier paleointensity determination by low-temperature demagnetization.

THEORETICAL BACKGROUND

Low-temperature demagnetization (LTD) is the process of cooling a sample to the isotropic temperature T_I = 120-135 K of magnetite (Bickford *et al.*, 1957; Syono, 1965). Usually the sample is cooled to liquid nitrogen temperature (77 K) and then reheated to room temperature in zero field. This is an effective means of erasing lower coercivity remanence (Nagata, 1961; Ozima *et al.*, 1964; Merrill, 1970). However, LTD cannot be carried out in stepwise fashion to yield a record of the progressive removal of soft component magnetization.

In theory, LTD should destroy the remanence carried by multi-domain grains of magnetite or Ti-poor titano-magnetite (Markov *et al.*, 1983). Around 130 K, the magnetocrystalline anisotropy K_1 changes its sign and the easy magnetization axis changes orientation (Bickford *et al.*, 1957). This transition is accompanied by abrupt changes in coercivity, remanence and susceptibility (Aragon, 1985, 1992). The magnetic memory (the fraction of TRM surviving after LTD) is much more resistant to AF demagnetization than original TRM before LTD (Heider *et al.* 1992). This hypothesis is supported by McClelland and Shcherbakov (1995). They demonstrated that LTD could destroy the component of multi-domain remanence. In this work, we examined whether technique involving low-temperature demagnetization could be used to remove the remanence carried by multi-domain grains in natural volcanic rocks.

EXPERIMENTAL RESULTS

Remanence measurements of different rock samples was recorded on 'Orion LtD' vibrating sample thermomagnetometer (so-called VSTM) at the paleomagnetic laboratory of the University of Montpellier. This magnetometer allows the acquisition and demagnetization of TRM and pTRM (partial thermoremanent magnetization), and the measuring of magnetic moment during thermal demagnetization in a continuous way along one axis. The sensitivity of the magnetometer is $5*10^{-10}$ Am^2, the maximum field H which can be applied is $4*10^3$ Am^{-1} and residual field after turning off the magnet is < 0.1 Am^{-1}. For our experiments, small cylindrical specimens were cut. Their magnetization was measured and fields were applied along their axis of maximum magnetization.

We selected twelve representative volcanic (basalts) sample coming from Southern Caucasus and Iceland. They yielded a stable remanence and reversible behavior during continuous susceptibility vs temperature measurements with Curie points near pure magnetite as showed by previous studies (Goguitchaichvili *et al.*, 1997, 1999a). One more sample belong to Pinatubo (Philippines) 1991 eruption. It contains two magnetic phases: ilmeno-hematite with y=0.53 and titano-magnetite with x=0.09 (Goguitchaichvili and Prévot, 2000a).

Determination of the viscosity index (Thellier and Thellier, 1944; Prévot, 1983) is useful to obtain information about paleomagnetic stability of the samples. We placed all samples during 15 days with one axis aligned with Earth's magnetic field. After measuring magnetization ($\mathbf{M_d}$), samples were placed for 15 days in a field-free space, and the magnetization ($\mathbf{M_0}$) was measured again. The viscosity index is $V = [(Z_d – Z_0) : M_{nrm}] \times 100$, where Z_d and Z_0 are the magnetization components of M_d and M_0 which are parallel to the magnetizing field and $\mathbf{M_{nrm}}$ is the intensity of natural remanent magnetization. The samples did not present a large capacity for viscous remanence acquisition. Viscosity index was generally less than 5%, a value which is low enough to obtain precise measurements of the remanence during the process of thermal demagnetization (Prévot *et al.*, 1985).

The samples belong to sites with very low angular dispersion. All values of α_{95} were within 5° of cleaned natural remanent magnetization (NRM) directions. They carry essentially a stable, single-component magnetization, observed upon both thermal and alternating field (AF) treatments (Figure 1). A minor secondary component, probably of viscous origin, is easily removed by low temperatures / AF fields. The median destructive fields range mostly in the 20-25 mT interval, suggesting the existence of pseudo-single domain (PSD) grains as remanence carriers (Dunlop and Ozdemir, 1997). This behavior might also be due to a mixture of single-domain (SD) and multi-domain (MD) magnetic particles. The major part of remanence is destroyed at 500-550°C, suggesting low-Ti titanomagnetite as responsible for magnetization.

Low-field continuous susceptibility measurements were performed in vacuum using a Bartington MS2 susceptibility

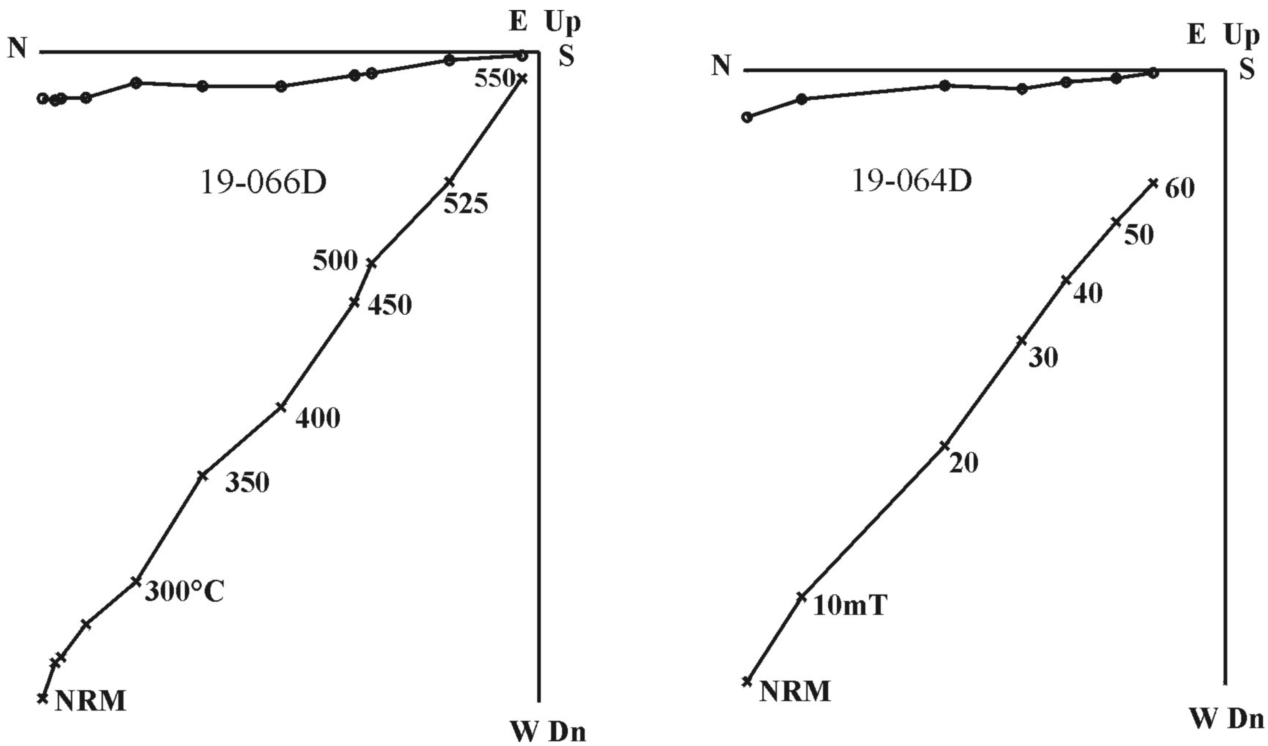


Fig. 1. Orthogonal vector plots of stepwise thermal or alternating field demagnetisation of selected samples. The numbers refer either to temperatures in °C or to peak alternating fields in mT. o – projections into the horizontal plane, x – projections into the vertical plane.

meter with furnace. The selected samples yield a single ferrimagnetic phase with a Curie point compatible with Ti-poor titanomagnetite (Figure 2a). Some of the cooling and heating curves are not perfectly reversible.

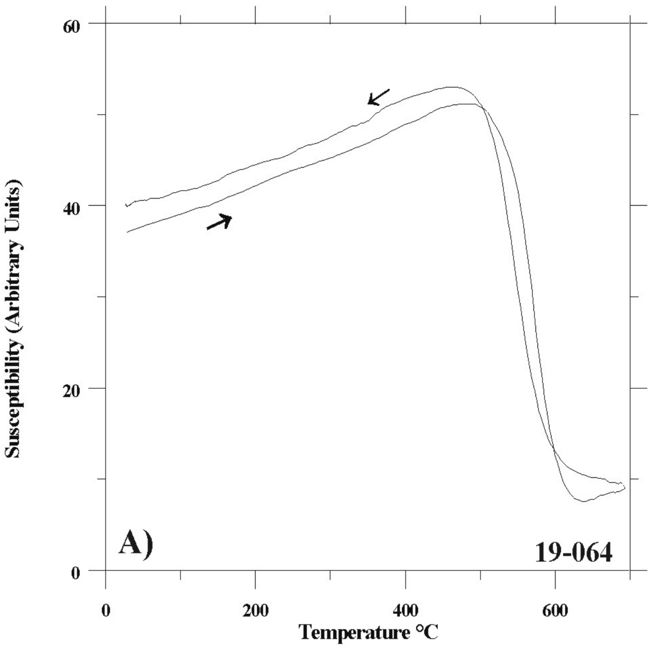
Hysteresis measurements at room temperature using an AGFM-Micromag apparatus were carried out in fields of up to 1.4 Tesla. The hysteresis parameters (saturation remanent magnetization Jrs, saturation magnetization Js, and coercive force Hc) were calculated after correction for the paramagnetic contribution. Coercivity of remanence Hcr was determined by applying a progressively increasing back field after saturation. IRM (isothermal remanent magnetization) curves show that saturation is reached in moderate fields of the order of 150-200 mT (Figure 2C), which points to some spinels as remanence carriers. Judging from the ratios of hysteresis parameters (Figure 2B), it appears that the samples fall in the PSD grain size. This probably indicates a mixture of MD and SD grains. If some superparamagnetic grains are also present, the measured coercive force and saturation magnetization may be underestimated.

As blocking and unblocking temperatures of multidomain grains are not the same, the presence of such grains can be detected by means of pTRM acquisition and demagnetization experiments. A pTRM acquired between 300°C and room temperature, would not be completely demagnetized below the Curie temperature (Bolshakov and Shcherbakova, 1979; Worm *et al.* 1988). This method is more efficient than regular hysteresis measurements to detect multidomain grains, but requires much more laboratory time (Goguitchaichvili *et al.,* 2001).

First, the NRM of the selected samples was demagnetized using VSTM (Figure 3, curve 1). Next they were cooled to room temperature in a 50 μT field, so that a TRM was acquired. Afterwards, this TRM was demagnetized (curve 2). Subsequently, pTRM was given to each of the samples between 300 and 25°C, which was subsequently thermally demagnetized (curve 3). The amount of remanent magnetization still present after heating above the highest pTRM acquisition temperature can provide information about the fraction of multidomain grains in a sample (Shcherbakova *et al.,* 1996). In a final step, the same pTRM was given again to those samples, where a significant multi-domain-grain fraction seemed to be present, and they were then placed into liquid nitrogen and left in free magnetic space approximately for two hours. Subsequently, they were thermally demagnetized (Figure 3, curve 4).

DISCUSSION

In five of twelve curves, pTRM blocking temperatures were quite similar to their blocking temperatures. Thus the



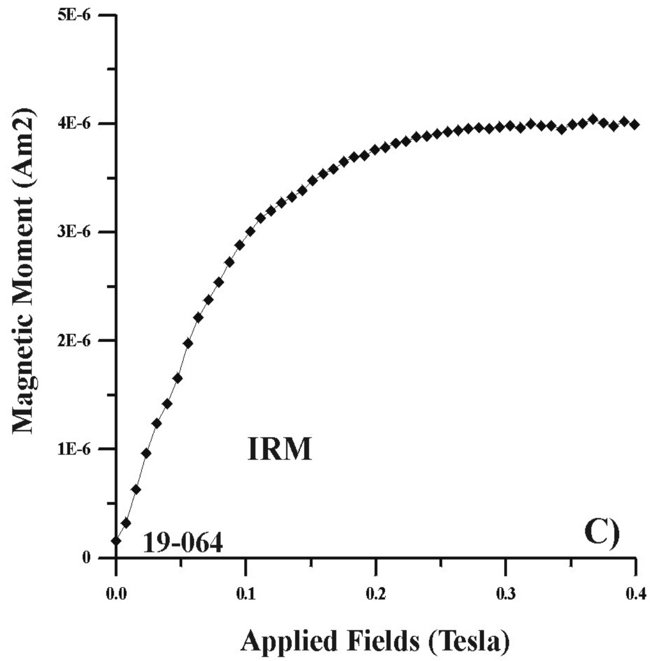


Fig. 2. (Continued).

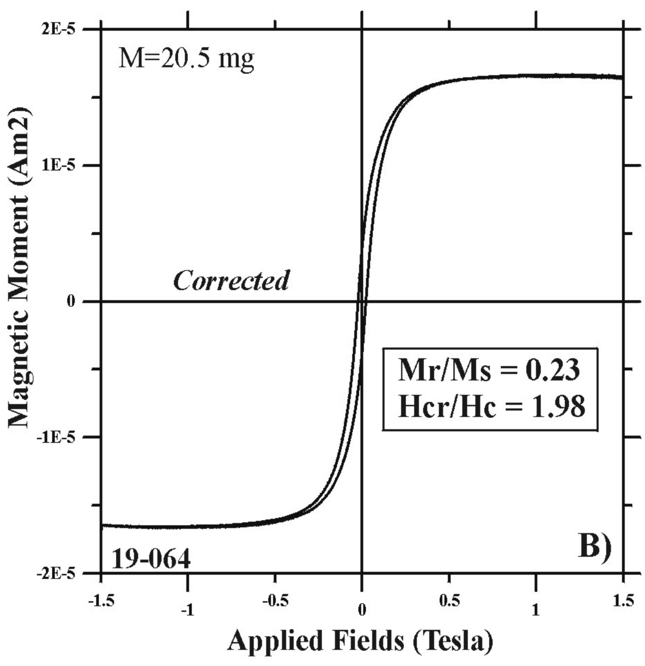


Fig. 2. a) Representative susceptibility versus temperature curves. The arrows indicate the heating and cooling curves (see also text). b) Typical examples of hysteresis loop (corrected for dia/paramagnetism) of small chip samples and c) isothermal remanence (IRM) acquisition curve.

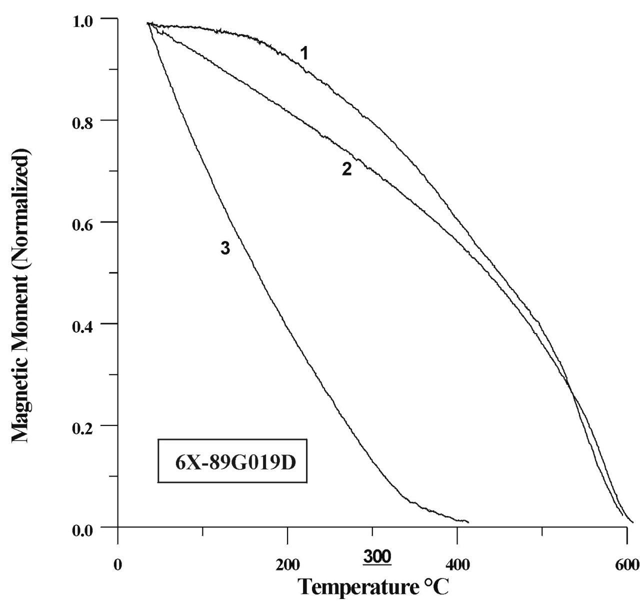
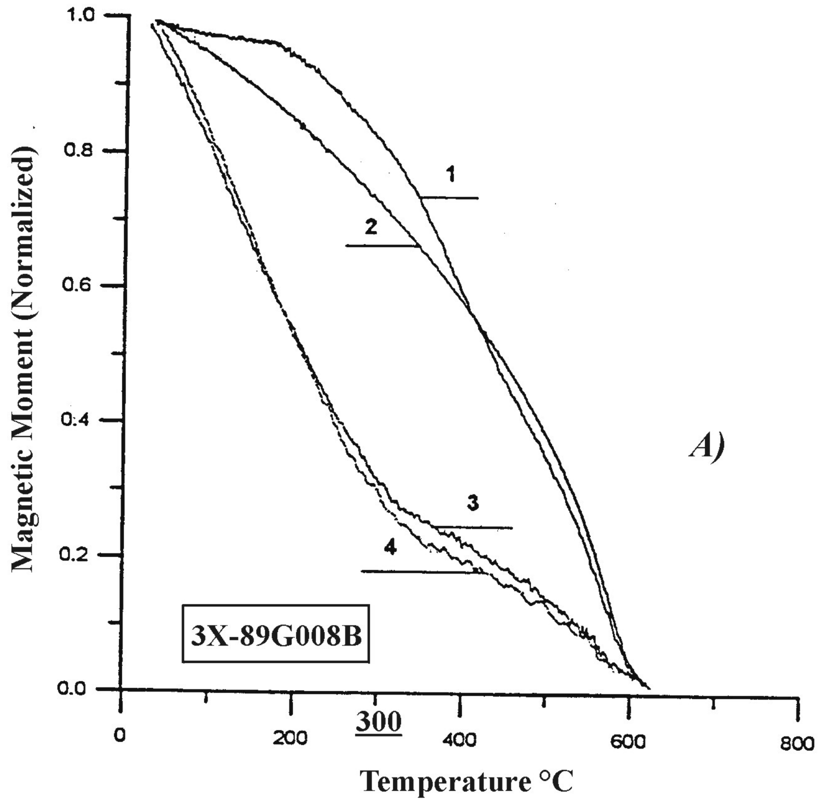


Fig. 3. Continuous thermal demagnetization of natural remanent magnetization (curve 1), thermoremanence (curve 2) acquired in a field of 50 μT, and partial thermoremanence (curve 3) acquired in a field of 50 μT, cooling down from 300°C to room temperature (from Goguitchaichvili *et al.,* 2000b).



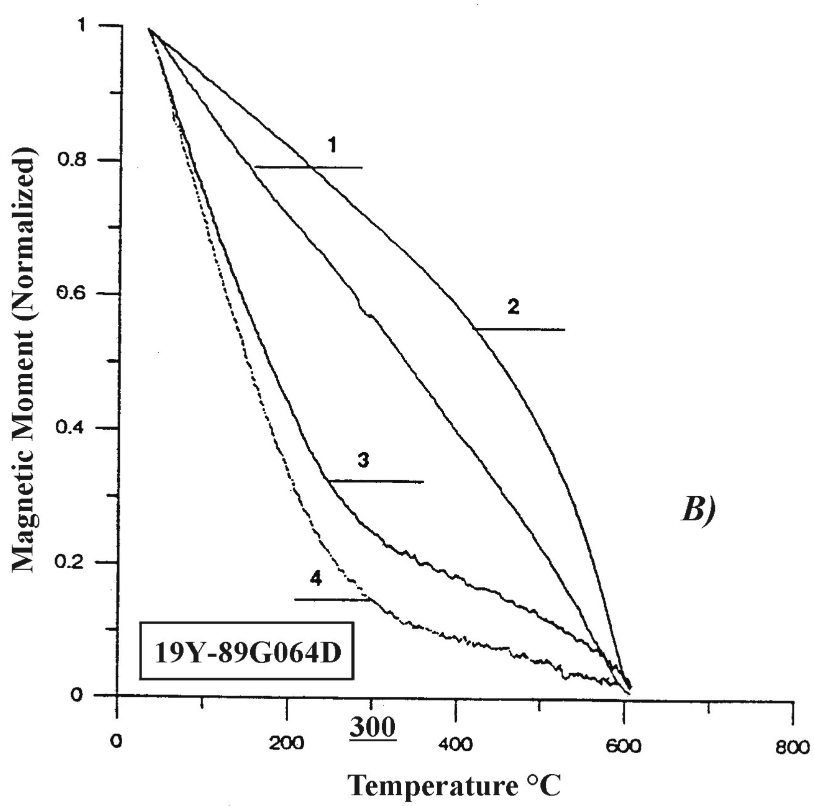


Fig. 4. Notation as in Figure 1. Curve 4 is a continuous thermal demagnetization of partial thermoremanence acquired in a field of 50 µT, from 300°C to room temperature after treatement with liquid nitrogen (See also text).

Table 1

The magnetic parameters of partial thermoremanent magnetization (pTRM) of samples which yielded 'multi-domain like' behaviour (see also text). $pTRM_1$ – intensity of partial thermoremanent magnetization formed from 300°C to room temperature in a field of 50 μT. $pTRM_2$ – *idem*, after LTD treatment (see also text). pTRM tail – the amount (in %) of pTRM (300°C, 50 μT, T_0) remainig undestroyed above 300°C.

Sample	pTRM tail (%)	$pTRM_1$(300°C) (A/m)	$pTRM_2$ (300°C) (A/m)	$pTRM_2$/$pTRM_1$
3X-89G008B	31	0.34	0.31	**0.91**
19Y-89G064D	26	0.46	0.29	**0.62**
FL2_3	19	0.14	0.12	**0.86**
FL1_1	24	0.78	0.55	**0.70**
FL3_7	24	1.51	1.13	**0.75**
KY3_7	21	1.05	0.78	**0.74**
SE1_1	28	0.67	0.54	**0.81**

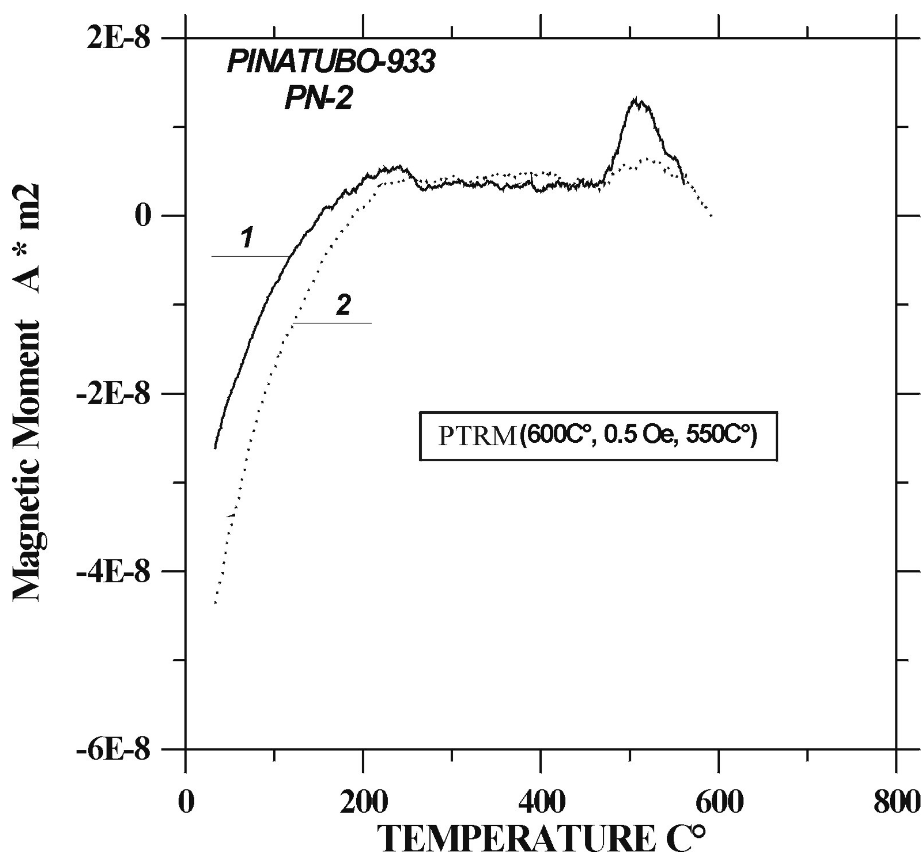


Fig. 5. Continuous thermal demagnetization of partial thermoremanence acquired in a field of 50 μT, cooling down from 600°C to 550°C before (curve 1) and after (curve 2) LTD treatment (see also text).

amount of pTRMs remaining undestroyed above 300°C was less than 15% of the total pTRM (Figure 3). This result probably indicates predominantly single-domain magnetic grains (Goguitchaichvili *et al.,* 2001). The seven other samples had to be heated to 600°C to completely demagnetize the pTRM (Figure 4). In those cases, a significant fraction of grains may have a multi-domain structure.

Low-temperature treatment removed a multidomain pTRM only partly (Figure 4, Table 1). We observed the reduction of pTRM tail from about 10 to 40%. This may be due to the fact that small deviations from stoichiometry of magnetite have an important effect on the Verwey transition, and the magnetic memory is controlled in part by the internal stresses developed during partial oxidation (Ozdemir *et al.,* 1993).

Figure 5 show the thermal demagnetization of pTRM (600°C, 50 μT, 550°C) carried by a sample containing: intermediary ilmeno-hematite and Ti-poor titano-magnetite before (curve 1) and after LTD treatment (curve 2). Both minerals have a multidomain domain structure as showed by Bitter technique observation (Bina *et al.,* 1999). The sample came from the 1991 eruption of Pinatubo volcano and showed total self-reversal due to the presence of ilmeno-hematites, and partial self-reversals observed from 500°C to 575°C due to titano-magnetite (Figure 5). This may be the first time that LTD was applied to self-reversed thermoremanence. In this case, the high-temperature peak was almost entirely suppressed by LTD.

CONCLUSION

Thirteen samples selected from previously studied volcanic units from Caucasus, Iceland and Philippines are tested. Samples present small viscosity indexes, Curie points compatible with Ti-poor titanomagnetites, stable remanence and reversible behaviour of susceptibility-temperature curves, and hysteresis parameters compatible with the pseudo-single-domain range (probably mixtures of single-domain and multidomain states).

The remanence carried by multidomain grains is removed only partly by low-temperature demagnetization. In general, 10 to 40% of ‘multidomain pTRM remanence’ were destroyed. This result may help to increase the success of paleointensity measurements.

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