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A rock- and paleomagnetic study of a Holocene lava flow in Central Mexico

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Abstract

Magnetic measurements of the Tres Cruces lava flow (ca. 8500 years BP, Central Mexico) show the presence of two remanence carriers, a Ti-rich titanomagnetite with a Curie temperature between 350 and 400°C and a Ti-poor magnetite with a Curie temperature close to 580°C. Magnetic changes after heating indicate that the titanomagnetite exsolves into magnetite and ilmenite when the sample is heated to 580°C. Paleointensity estimates with the Thellier and Thellier method [Thellier, E., Thellier, O., 1959. Sur l'intensité du champ magnétique terrestre dans le passé historique et géologique. Ann. Géophysique., 15, 285–376] were only successful up to temperatures of 350 to 400°C. This temperature corresponds with the Curie temperature of the titanomagnetite, which is probably pseudo-single or multi-domain. Therefore, the paleointensities should be interpreted with caution. The magnetic composition changes after 580°C heating may explain the large variations in previous paleointensity determinations for the Tres Cruces rocks [Gonzalez, S., Sherwood, G., Böhnel, H., Schnepp, E., 1997. Palaeosecular variation in Central Mexico over the last 30,000 years: the record from lavas. Geophys. J. Int., 130, 201–219] using the Shaw method [Shaw, J., 1974. A new method of determining the magnitude of the palaeomagnetic field: application to five historic lavas and five archaeological samples. Geophys. J. R. Astr. Soc., 39, 133–141]. © 2000 Elsevier Science B.V. All rights reserved.

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1. Introduction

The availability of accurate paleointensity records at several places on the earth would be a great help for geomagnetic dynamo modeling. However, this front in paleomagnetism has not yet reached the same level of reliability as the study of paleomagnetic directions and polarities of the earth's magnetic field, mainly because details of processes leading to acquisition of the natural remanent magnetization (NRM) are not sufficiently known.

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Sedimentary paleointensity records only reflect relative intensity changes of the magnetic field and these records are influenced by non-instantaneous NRM acquisition and environmental changes (e.g., Weeks et al., 1995; Kok and Tauxe, 1996; Mazaud, 1996; Worm, 1997). Lava flows may record absolute paleofield intensities but do not provide a continuous record of the earth magnetic field. It is generally assumed that lavas carry a thermoremanent magnetization (TRM). This TRM, acquired during the cooling of the lava flow, is proportional to the intensity of the earth magnetic field. Therefore, it is theoretically possible to determine the paleofield intensity by duplicating TRM acquisition in a known laboratory field. A commonly used method for paleointensity measurements has been developed by Thellier and Thellier (1959) and modified by Coe (1967). It compares the successive loss of the natural remanence (= NRM) with the acquisition of a laboratory thermoremanence in a controlled field (= TRM) between two temperature steps. Shaw (1974) developed an alternative method. This author compared (1) the decay in NRM and TRM, imparted at the Curie temperature of magnetite (580°C), between two alternating field (AF) demagnetization steps to calculate ''paleointensities'' and (2) the AF decay curves of an anhysteretic remanent magnetization (ARM) before and after heating to detect thermal alterations. Rolph and Shaw (1985) modified the Shaw (1974) ''paleointensity'' method by multiplying NRM/TRM ratios with the change in ARM after heating in the same AF coercivity interval. This modification is supposed to correct for thermal alteration.

Success rates of paleointensity measurements are often low (less than 50%) and even paleointensity estimates considered as reliable may differ within a single lava flow (e.g., Böhnel et al., 1997; Rolph, 1997). Reasons for these phenomena include (1) thermal resetting in nature, (2) magnetic alteration during laboratory heating, (3) magnetic alteration over geological time, (4) NRM of non-thermoremanent origin or (5) unstable NRM. If large multi-domain (MD) grains are detected, the measurements should be interpreted with caution, because one assumption for the Thellier method (Thellier and Thellier, 1959), the independence of partial TRMs, is not valid (Bol'shakov and Shcherbakova, 1979; Shcherbakov et al., 1993). Therefore, quality checks for absolute paleointensity measurements are desirable (Prévot et al., 1983, 1985; Haag et al., 1995; Kosterov and Prévot, 1998). Such checks may help to determine the reliability of thermomagnetic measurements in terms of geomagnetic field strength. They may also help to test the validity of methods which correct paleointensity determinations when the Thelliers measurements fail (McClelland and Briden, 1996; Valet et al., 1996). To contribute to this front of paleo and rock magnetism, we performed a magnetic study on a Holocene andesitic lava flow in Central Mexico.

The aim of this study is to present a simple method, which can be used to monitor major thermal alterations during PI determinations. Together with the quality of the latter, the results can be used to check the validity of the paleostrength determinations. The measurements can be divided into four groups: (1) The paleointensity measurements, using the Thellier method (Thellier and Thellier, 1959). (2) Room-temperature measurements of susceptibility, hysteresis loops and isothermal remanent magnetization (IRM) after several heating steps to monitor irreversible changes in the magnetic signal due to thermal alterations. (3) Alternating field (AF) and thermal demagnetization of IRM and ARM on unheated and heated specimens to determine magnetic composition before and after heating. (4) Thermomagnetic runs and low-temperature measurements to improve the interpretation. Samples from the Tres Cruces lava flow in Central Mexico were used for this study. Gonzalez et al. (1997) determined PI estimates of this flow using the Shaw method (Rolph and Shaw, 1985). Implications of our results on the validity of these estimates will also be pointed out.

2. Geological setting

The Tres Cruces volcano is situated in the Mexican volcanic belt, about 60 km west of Mexico City. This monogenetic volcano produced ash deposits (0.25 km^2) and an andesitic lava flow (Bloomfield, 1975). Datings of burned soils under this ash revealed ages of 8.39 ± 0.10 and 8.44 ± 0.07 ^{14}C ka, respectively (Bloomfield, 1975). The ages for the ash and lava flow are approximately the same, because monogenetic volcanoes are only active during a short period (generally less than 15 years). The Tres Cruces lava flow has a maximum length of 5 km and a maximum height of 40 m (Bloomfield, 1975) and is covered by vegetation. We sampled the Tres Cruces flow a few kilometers southeast of San Pedro Techuchulco. The geographical coordinates of this site (19°06.17′N, 99°30.11′W) were determined with GPS (Geographical Positioning System). Two sites (I and II), about 80 m apart, were sampled. These sites, two big outcrops of fresh rocks, are situated in the center of the flow. Samples in these sites were randomly taken with a minimum distance of 25 cm between the individual samples. Unless otherwise stated, no relationships between variations in rock and paleomagnetic results and the sampling location exist.

In the laboratory, the paleomagnetic cores were cut into 10.5-cm^3 volume specimens. Only cores containing three (or more) specimens were considered further. The specimens in the deepest part of the rock were used for paleomagnetic analysis. Specimens situated in the core center were used for IRM and low-field susceptibility measurements. Specimens in the upper part of the core, which is closest to the rock surface, were used for other rock magnetic experiments (mainly hysteresis measurements). Following this procedure, this paleo- and rock magnetic study has been performed on 12 paleomagnetic cores. Four of these cores are from site I.

3. Paleomagnetic measurements

Paleointensity (PI) estimates were made with the help of the Thellier–Thellier method (Thellier and Thellier, 1959) as modified by Coe (1967). Samples were first heated to a particular temperature and cooled in zero field (partial thermal demagnetization of the NRM). Then the samples were heated to the same temperature in a 50-μT field and cooled in the field to produce a partial thermoremanent magnetization (pTRM). After the 300, 400, 500, and 580°C heating steps, so-called pTRM checks were carried out: pTRM acquisition was repeated for previous lower temperature steps to check for thermal alteration. Results are plotted in NRM versus TRM diagrams. Specimens were heated in a Magnetic Measurements Thermal Demagnetizer (MMTD1) furnace and the remanence was measured with a JR-5 spinner.

Before discussing the paleointensity estimates, we present the NRM results. NRM intensities before heating range from 1.82 up to 5.77 A/m. Thermal demagnetization diagrams show that nine specimens have a small overprint, removed with temperature up to 200–250°C, and a stable magnetization component removed at 580°C. (Fig. 1a,b). Directional variations of the latter, determined by fitting a least square line through the demagnetization steps (Kirschvink, 1980), are limited (Table 1). Three specimens have multiple magnetization components; the direction of the low-temperature component is scattered and the dispersion in the high-temperature component is larger than for samples with one NRM component (Table 1). The mean NRM direction, derived from the specimens with one magnetization component, corresponds with the mean of Gonzalez et al. (1997) (Table 2). Urrutia-Fucugauchi and Martinez-Serrano (in press) also performed a paleomagnetic study on this flow, but their directions show a large dispersion ($D = 330.9°$, $I = 26.5°$, $N = 6$, $\alpha_{95} = 49.3$, $k = 3$). Gonzalez et al. (1997) and Urrutia-Fucugauchi and Martinez-Serrano (in press) did not discuss the magnetic properties of the Tres Cruces lava flow.

Due to spurious NRM-TRM diagrams and/or a NRM with multiple magnetization components PI determinations failed for five specimens. The remaining seven specimens show good NRM–TRM relationships up to either 350 or 400°C and successful pTRM tests (Fig. 1d,e,f,). Above these temperatures NRM intensities decrease, but TRM intensities hardly increase (Fig. 1). Hence, PI estimates could only be made up to maximum 400°C. These estimates (Table 2) are more consistent than the PI estimates from Gonzalez et al. (1997), which were determined with the Shaw (1974) method and the Rolph and Shaw (1985) correction.

4. Rock magnetism

Rock magnetic measurements were carried out to determine magnetic mineralogy and changes in mag-

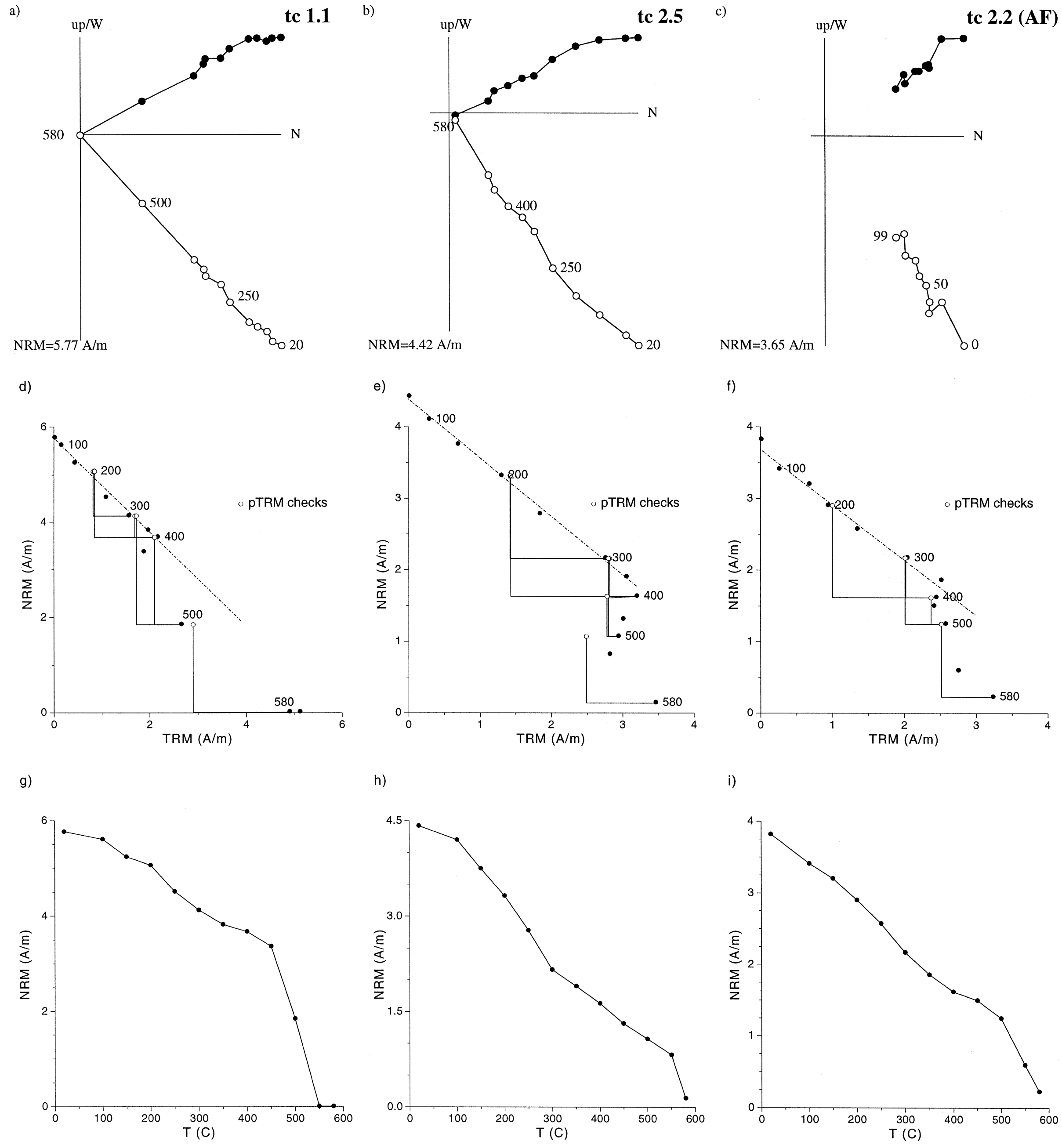

Fig. 1. (a) NRM thermal demagnetization diagram (Zijderveld, 1967) for samples 1.1 and 2.5. (c) AF demagnetization diagram for sample 2.2; another specimen from this paleomagnetic core was used for Thellier–Thellier paleointensity experiments. (d,e,f) NRM versus TRM intensity and (g,h,i) NRM thermal decay curves for samples 1.1, 2.5 and 2.2.

netic properties during heating. To link these changes with the paleointensity measurements, specimens for the rock magnetic measurements were heated and cooled under the same conditions as the specimens

Table 1
Directions and paleointensities for the measured samples of the Tres Cruces lava. Average values for both sites are marked in bold letters. For the mean NRM directions only samples with one component (marked with a star) are considered. Paleointensity estimates for these samples are determined between 100 and 350 or 400°C. The quality of these estimates is calculated after York (1966) and Coe (1978)

Specimen	*D*	*I*	α_{95}	*k*	PI (μT)	*s* (μT)	Δ*T* (°C)	*f*	*g*	*q*	σ_e (μT)
tc 1.1	345.0	46.3★			49.3	3.6	100–400	0.34	0.80	3.9	
tc 1.2	330.3	44.0★			49.4	4.6	100–400	0.45	0.79	3.9	
tc 1.3	4.7	60.4			–	–	–	–	–	–	–
tc 1.4	306.25	52.1			–	–	–	–	–	–	–
Mean site 1	**336.9**	**46.3**			**49.4**						**0.1**
tc 2.1	52.8	53.7			–		–	–	–	–	
tc 2.2	339.3	41.0★			40.2	3.5	100–400	0.49	0.83	6.0	
tc 2.3	340.1	50.1★			37.9	2.4	100– 350	0.45	0.82	6.1	
tc 2.4	344.4	53.0★			–		–	–	–	–	
tc 2.5	333.5	52.7★			42.4	1.4	100– 400	0.59	0.82	15.1	
tc 2.6	345.1	53.2★			36.9	4.1	150– 400	0.55	0.83	4.1	
tc 2.7	337.0	52.4★			–		–	–	–	–	
tc 2.8	334.75	55.0★			43.0	5.3	150– 350	0.53	0.80	3.4	
Mean site 2	**339.2**	**51.1**	**4.0**	**223.9**	**40.0**						**4.2**
Site 1 + 2	**338.5**	**50.3**	**3.7**	**195.6**	**42.7**						**10.0**

used for the paleointensity determinations. IRM intensity, low-field susceptibility and hysteresis parameters were measured on samples from all 12 paleomagnetic cores. Other analyses were performed on less samples.

4.1. Susceptibility, isothermal and anhysteretic remanent magnetization

Low-field susceptibilities (K) were measured with a KLY-2 Kappa-Bridge. K was measured on all samples before the other magnetic measurements. During the thermal experiments, K was also measured at room temperature after each heating step. Fig. 2a shows that K increases up to 500°C and decreases after heating to higher temperatures.

Table 2
Paleomagnetic directions and intensities from Gonzalez et al. (1997)

Mean NRM direction				
D	*I*	*N*	α_{95}	*k*
337.4	55.9	6	3.1	462.7

Paleointensity estimates (Shaw method)

Sample	PI (μT)
S-6Bb	26.75 ± 0.62
S-6Cb	56.13 ± 0.61
S-6Db	no results
S-6Eb	62.01 ± 1.67
S-6Gb	33.01 ± 0.91

IRM and ARM intensities were measured with a JR-5 spinner. IRM was imparted with a pulse magnetizer using a 1-T field. Unfortunately, the maximum AF field of the Schonstedt demagnetizer (99.9 mT) was insufficient to saturate ARM (Fig. 3a). Gonzalez et al. (1997) imparted an ARM with a 150-μT DC field and a 200-mT AC field. These measurements confirm the high AF coercivities of ARM. They also show that after 580°C heating the AF demagnetization is less effective at low fields (<30 mT), but relatively more ARM is removed with high alternating fields (Fig. 3b). NRM and TRM also have high AF unblocking spectra (Figs. 1c and 3c).

Imparted IRM intensities gradually decrease between 200 and 450°C heating, but are considerably higher after 580°C. Trends in IRM are inverse to those in susceptibility (Fig. 2). A 99-mT AF is insufficient to remove the IRM (Fig. 4a). After heating the specimens at 580°C, AF demagnetization is more effective for high fields (Fig. 4a). Then the IRM, imparted in an unheated specimen and a specimen heated to 580°C, was thermally demagnetized (both specimens are from the same paleomagnetic core). The IRM in both specimens is removed at 580°C, typical for magnetite (Fig. 4b). However, in

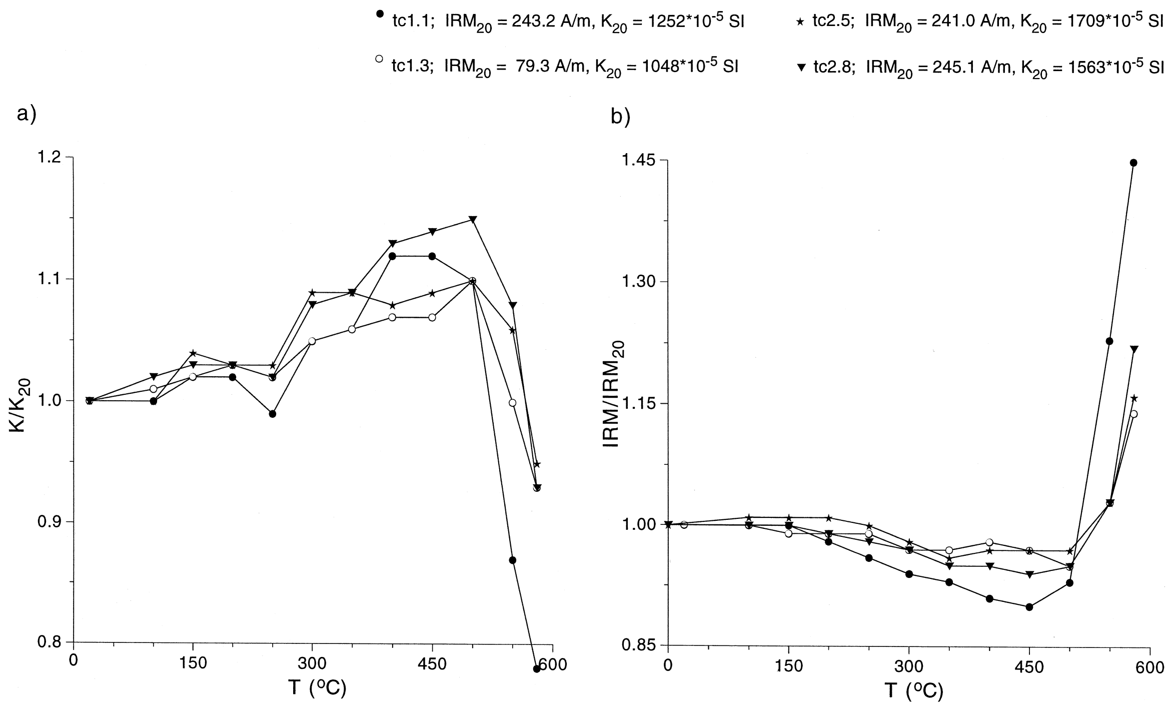

Fig. 2. (a) K and (b) IRM after heating. Both parameters are measured at room temperature and normalized on K and IRM values before heating. K and IRM values before heating are shown in the right column.

contrast to the 'heated' specimens, the 'unheated' specimens also show the presence of a second low-temperature (LT) magnetic phase. The unblocking temperature of this LT component varies somewhat between the specimens, but is generally in the temperature range 350–400°C (Fig. 4b).

4.2. Hysteresis parameters

Hysteresis loops were measured at room temperature with an Alternating Gradient Magnetometer of Princeton Meas. (MicroMag), using a maximum field of 1 T. After correction for the small paramagnetic high field slope, the saturation (M_s), remanent magnetization (M_r) and coercive force (B_c) were determined. The remanent coercive force (B_{cr}) was measured by a backfield demagnetization of a remanence imparted with a 1-T field. As only small chips (20–30 mg) can be measured with the MicroMag, four chips per paleomagnetic core were measured. To monitor thermal alterations, chips were broken from a larger specimen after heating and measured at room temperature. As the hysteresis parameters of four chips were measured after each heating step, changes in their average are related to magnetic alteration.

The remanence ratio (M_r/M_s) ranges between 0.1 and 0.2 and the coercivity ratio B_{cr}/B_c (3.7–5.0) is somewhat enhanced (Fig. 5a). B_c and B_{cr} range between 7–10.5 mT and 30–50 mT, respectively (Fig. 5b). Hysteresis parameters do not significantly differ between sites I and II and there is no relationship between determined 'paleointensity' values and hysteresis parameters. However, it is worth noticing that the two specimens with the lowest B_{cr} and the smallest enhancement in B_{cr}/B_c have a two-component NRM and thus a failed paleointensity experiment (Fig. 5, Table 1).

M_s tends to increase after heating, but large error bars in average values (Fig. 6) hamper the interpretation. For three of the four measured specimens the remanence ratio (M_r/M_s) increases after the 580°C step. B_{cr} slightly decreases up to 450°C, but increases after heating at higher temperatures. B_c hardly changes up to 450°C, but considerably in-

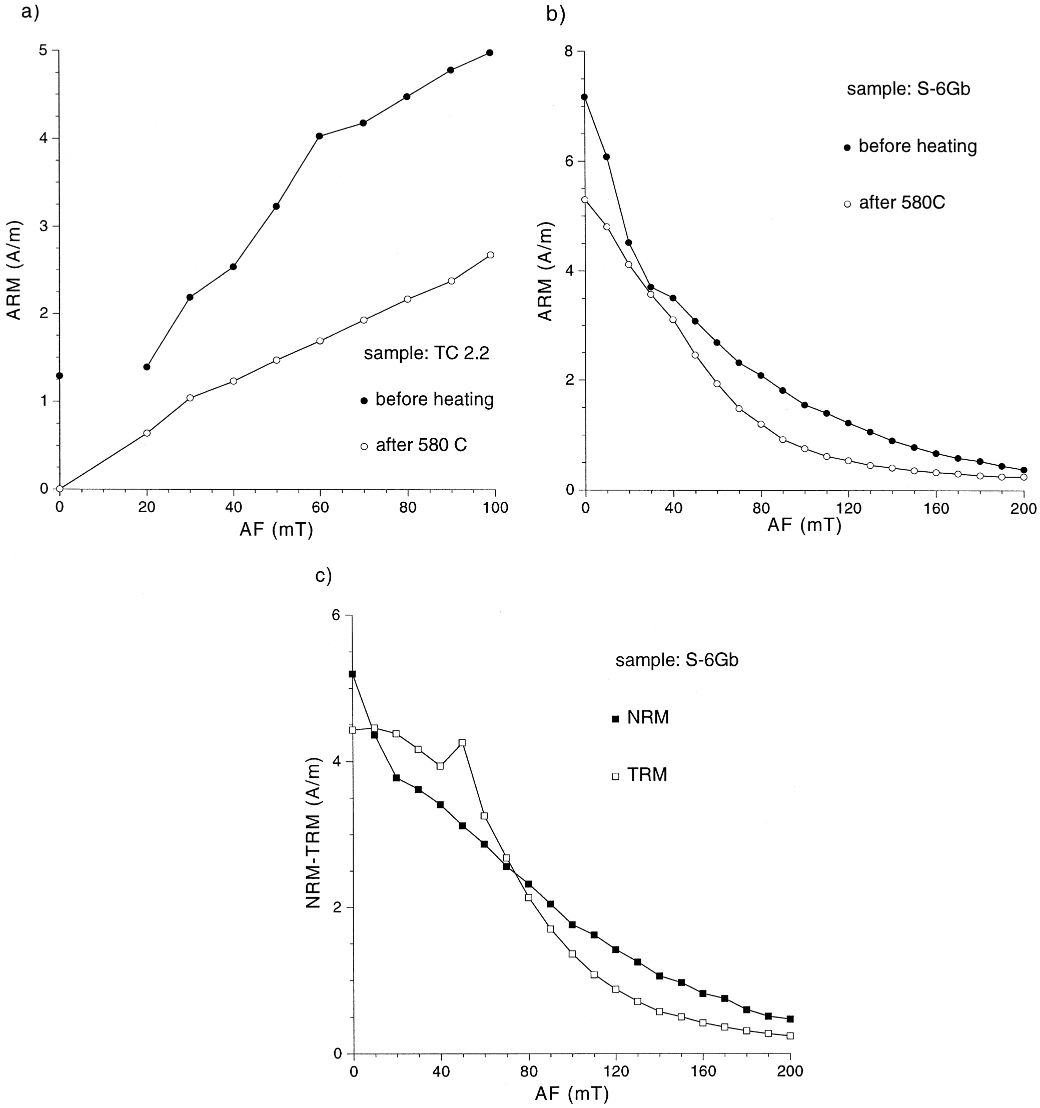

Fig. 3. (a) ARM acquisition curves. (b) ARM (closed circles before heating; open circles after heating) and (c) NRM (closed squares), TRM after 580°C heating (open squares) versus AF demagnetization. Results shown in (b) and (c) are from the paleointensity determinations on sample S-6Gb by Gonzalez et al. (1997).

creases after 580°C heating. B_{cr}/B_c decreases after heating at 580°C.

4.3. Thermomagnetic runs

A horizontal Curie balance of Utrecht University (Mullender et al., 1993) was used to measure saturation magnetization versus temperature (M_s–T) curves. Successive runs in air up to 350, 450, 500, 580 and 700°C were measured on two specimens. The results show reversible thermomagnetic runs up to 450°C, but runs with a higher maximum temperature show slightly higher magnetizations during cooling (Fig. 7). Furthermore, runs up to 450°C indicate

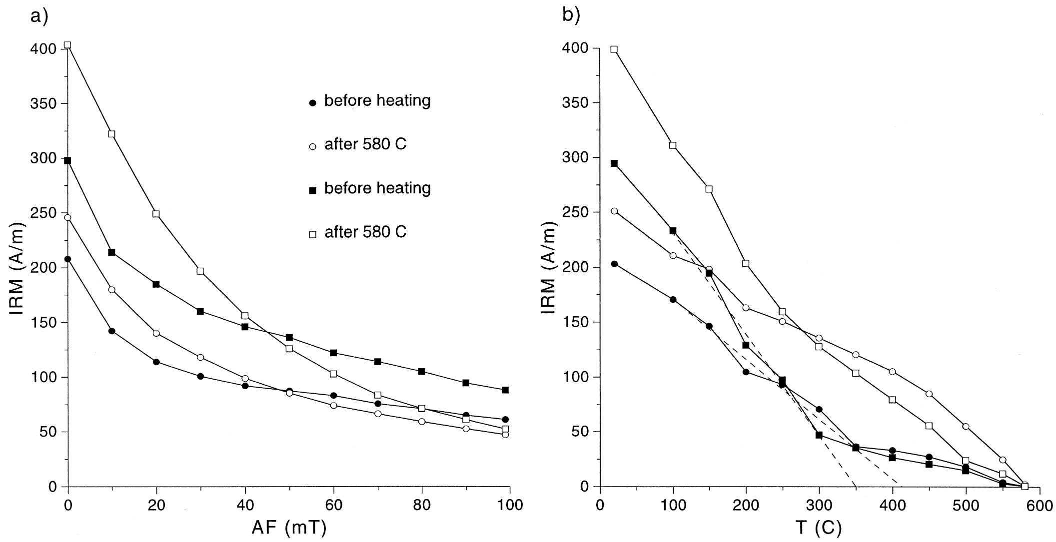

Fig. 4. IRM (a) AF and (b) thermal decay curves before (closed symbols) and after heating (open symbols) to 580°C. The dotted line shows the unblocking temperatures of the LT component.

a low-temperature magnetic phase, which has a Curie temperature around 350°C. This phase is probably associated with the 350–400°C IRM unblocking temperature for the ‘unheated’ specimens (Fig. 7).

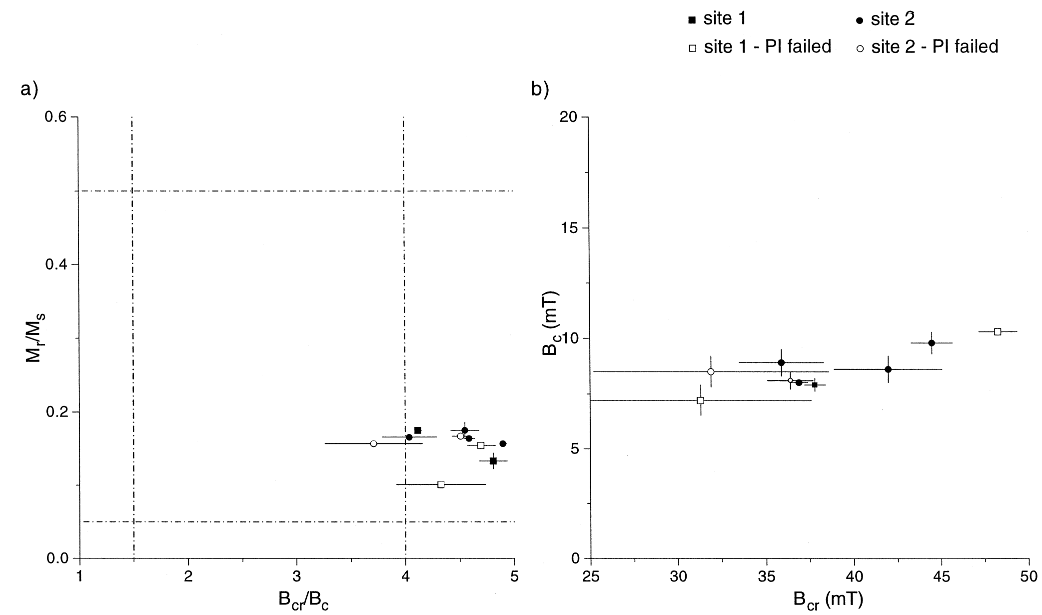

Fig. 5. Hysteresis parameters [(a) M_r/M_s, versus B_{cr}/B_c (b) B_c versus B_{cr}] at room temperature. Open (closed) symbols denote samples, which Thellier–Thellier paleointensity experiments failed (passed).

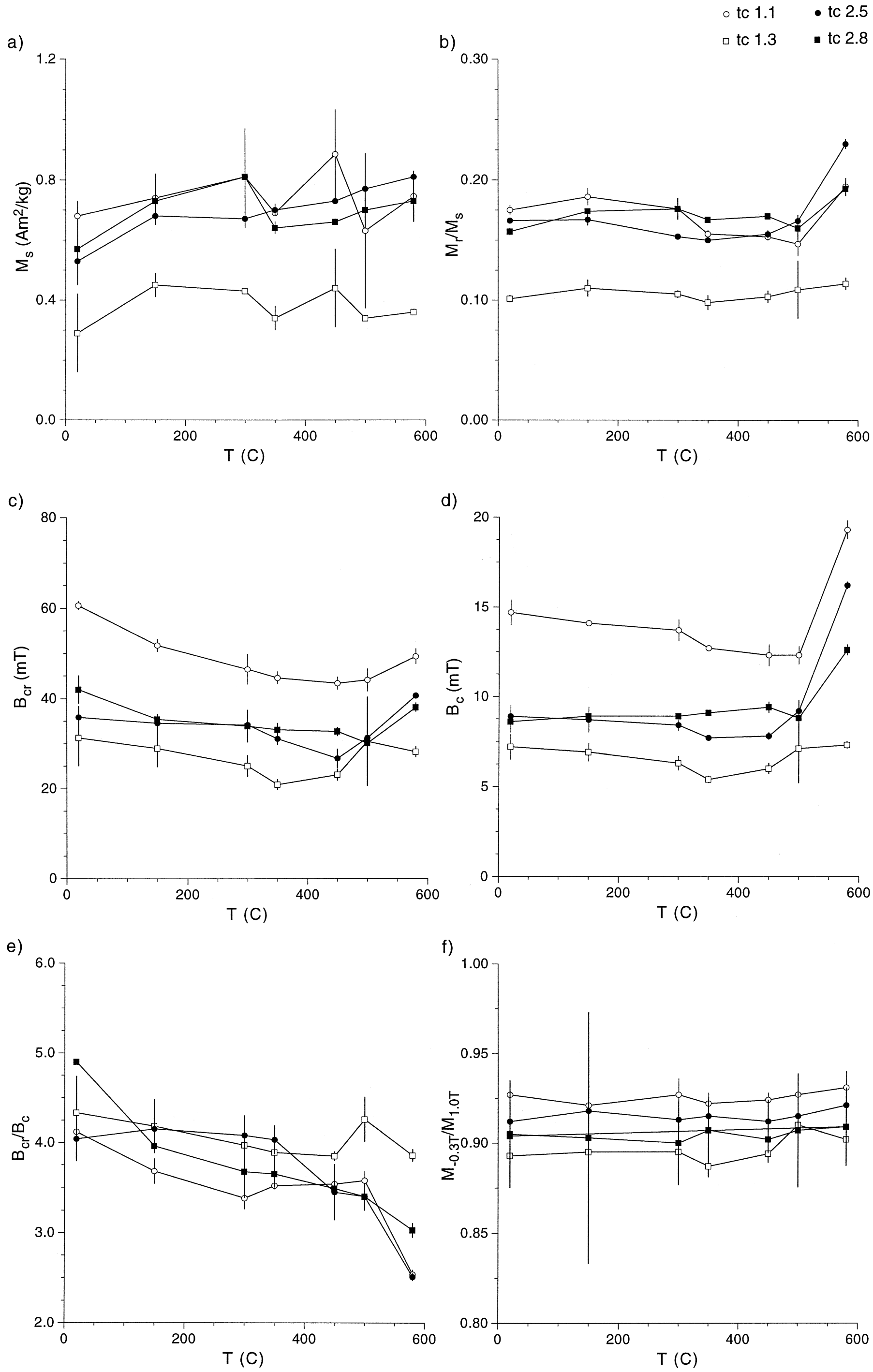

Fig. 6. Changes in (a) M_s, (b) M_r/M_s, (c) B_{cr}, (d) B_c, (e) B_{cr}/B_c and (f) $S\ (=M_{-0.3}T/M_{1.0}T)$ ratios after heating. Sample TC 1.3 shows the weakest variations in hysteresis parameters during heating. This sample also has the lowest coercivities and remanence ratios and smallest variations in K and IRM (Fig. 2).

This Curie temperature increases and disappears in thermomagnetic runs with higher maximum temperatures. This observation agrees with the IRM thermal decay curves, which also showed the disappearance of a low-T_c phase component after 580°C heating (Fig. 4b).

4.4. Low temperature measurements

Low-temperature IRM versus T measurements were made with a Quantum Design Superconducting Susceptometer (MPMS). An IRM was imparted with a 2.5-T field at room temperature. Then, the remanence was measured as the specimens (0.29–0.33 g) were cooled to 20 K in zero field. In the next step, the specimen was heated to 580°C, before carrying out the same low-temperature experiment. The first run shows an increase in magnetization on cooling between 300 and 200 K (Fig. 8). Dankers (1978) observed a similar intensity increase for a crushed natural titanomagnetite ($Fe_{2.4}Ti_{0.6}O_4$) and related this phenomenon to a higher spontaneous magnetization below room temperature for titanomagnetites (Creer and Like, 1967). After heating the specimen at 580°C, the low-temperature measurements show a much smaller intensity increase between 300 and 200 K and a larger intensity drop at 116 K (Fig. 8), reflecting the crystallographic Verwey transition for magnetite (Verwey, 1939).

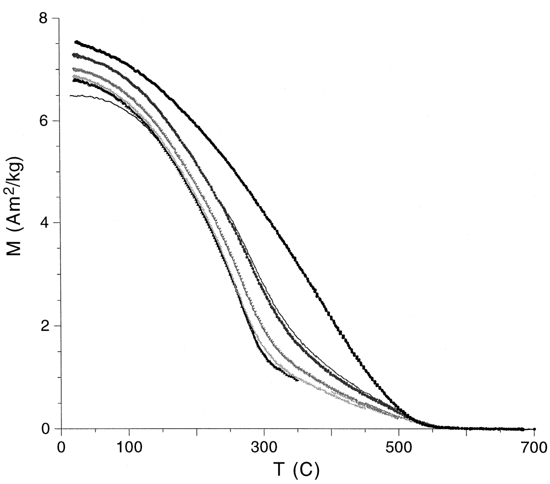

Fig. 7. A typical example of successive thermomagnetic M_s-T runs up to 350, 450, 500, 580 and 700°C. Lines (dotted lines) denote heating (cooling) curves.

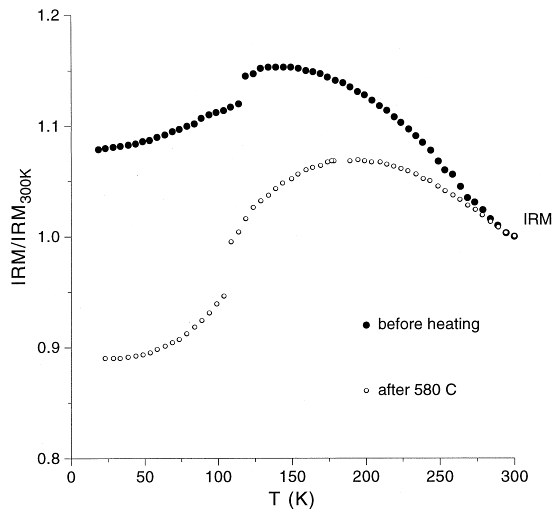

Fig. 8. Low-temperature measurements. The IRM was imparted with a 2.5-T field at 300 K (27°C), before cooling the specimen to 20 K (−253°C) in a zero field.

5. Discussion

The mean ‘paleointensity’ for the Tres Cruces lava flow, determined with the Thellier–Thellier method, does not significantly differ from the present geomagnetic field intensity in Central Mexico (45 μT) (Urrutia-Fucugauchi and Campos-Enriquez, 1993). However, NRM directions do significantly differ from the orientation of the present day field (Dec = 9.2° and Inc = 47.4°; Urrutia-Fucugauchi and Campos-Enriquez, 1993), indicating that the NRM is not a recent overprint and may be primary. This does not, however, ensure that the determined ‘paleointensities’ reflect the paleofield intensity during the deposition of this ca. 8500-year old lava flow. A critical comparison between the rock magnetic data, thermal alterations and paleomagnetic signal may help to conclude whether the latter is likely or not.

5.1. Magnetic alterations above 500°c

Above 500°C, changes in IRM, χ (Fig. 2), hysteresis parameters (Fig. 6) and thermomagnetic runs

(Fig. 7) indicate the occurrence of thermal alterations. Kono (1985; 1987) studied thermal alteration of a titanomagnetite ($Fe_{2.6}Ti_{0.4}O_3$) in a historic Japanese basalt. This author heated the basalt in air at 630°C using different heating times and found considerably higher B_{cr} and B_c values, slightly higher M_r/M_s and M_r values, and lower B_{cr}/B_c values after heating. Together with the results of the NRM and TRM thermal decay curves and thermomagnetic runs, these changes were attributed to exsolution of the original multi-domain (MD) and pseudo-single domain (PSD) titanomagnetite fraction into a magnetite–ilmenite intergrowth with single domain (SD) properties (Kono, 1985). A magnetite–ilmenite intergrowth was already present in the samples and probably formed during initial cooling of the lava. Higher B_{cr}, B_c, M_r/M_s, IRM and lower B_{cr}/B_c values are also observed for the Tres Cruces samples after heating. Furthermore, the LT component in the IRM thermal decay curves and thermomagnetic runs disappears when samples are heated at 580°C (Figs. 4 and 9). Although the Tres Cruces samples were only heated at 580°C for 35 min, all changes agree with those of Kono (1987). Therefore, we relate the magnetic change after 580°C to the formation of more pure magnetite, intergrown with ilmenite, from a probable MD-PSD titanomagnetite. Formation of magnetite after 580°C heating is also indicated by the clearly visible low-temperature Verwey transition for the heated specimens (Fig. 8). If our interpretation is correct, the low M_r/M_s ratios (< 0.2) may reflect the multi-domain Ti-rich titanomagnetite (Day et al., 1997). High B_{cr}/B_c (> 3.7) ratios may also be influenced by these grains, but the slight enhancement in B_{cr}/B_c is more likely due to the presence of two magnetization carriers. According to this interpretation, the LT component in the IRM thermal decay curves and thermomagnetic runs reflects the MD-PSD titanomagnetite. The removal of this component around 400°C is interpreted as a Curie temperature, because the thermomagnetic runs are reversible up to temperatures lower than 500°C.

Strangway et al. (1968) and Senanayake and McElhinny (1981) reported that titanomagnetite grains with exsolution lamellae of ilmenite possess high stability of magnetization. This is because ilmenite lamellae subdivide titanomagnetite grains into a number of magnetically independent small regions, which are elongated enough to behave as single domains. Hence, this interpretation explains the high AF coercivities by magnetite–ilmenite intergrowth. After heating to 580°C, IRM and ARM are easier removed with high AFs, but the relative amount of ARM and IRM demagnetized up to 20 mT AF decreases (Figs. 3b and 4a). Hence, changes in both high and low AF coercivity magnetic minerals occur, implying that AF decay curves do not exclude transformation of Ti-rich titanomagnetite into magnetite after 580°C heating.

5.2 Limitations comparison rock magnetic results paleointensity measurements

A critical point is to what extent rock magnetic results based on IRM and high-field measurements can be related to NRM and TRM behavior. This relationship is not straightforward, because (1) NRM unblocks at generally higher temperatures than IRM (Fig. 2d,e) and does not have an unblocking temperature between 350 and 430°C. Furthermore, (2) IRM intensities increase (Fig. 2b), but ARM intensities of Gonzalez et al. (1997) decrease after heating the sample in 580°C (Fig. 3b,c). On the other hand, (1) all remanences, including IRM, are difficult to demagnetize with an AF and (2) trends in IRM, induced with a strong magnetic field, and in χ, measured in a weak magnetic field, versus heating temperature are opposite, indicating a relationship. The failure of Thellier PI determinations around 350–400°C corresponds with the unblocking temperature of the LT component in the IRM thermal decay curves and thermomagnetic runs. This does not necessarily explain the failure of the Thellier experiments. However, although NRM does not show a LT component, this observation suggests that up to 350–400°C TRM (and NRM?) are for a considerable part carried by the LT component, which is probably PSD-MD titanomagnetite. Large multi-domain grains do not carry a stable magnetization and have concave instead of linear NRM-TRM relationships (Levi, 1977). One assumption for the Thellier method, the independence of partial TRMs is not valid for MD grains (Bol'shakov and Shcherbakova, 1979). Therefore, it can be ruled out that the NRM-TRM ratios up to 350–400°C, assuming that MD-PSD grains carry them, accurately record paleofield intensity. The fact that two samples with the lowest M_r/M_s, B_{cr}, and B_c values and weakest enhancement in B_{cr}/B_c have multiple NRM components reinforces the suggestion that an unstable NRM is related to large PSD-MD grains. These multiple component NRMs and unsuccessful paleointensity experiments are probably not due to thermal alteration, because these samples show the smallest change in magnetic parameters after heating (see sample TC 1.3, Figs. 2 and 7).

The failure of the Thellier measurements around 350–400°C is not related to a sudden change in room-temperature IRM, χ and hysteresis parameters (Figs. 2, 3, 7 and 8). TRM intensities do not increase between 400 and 550°C and may even decrease between the individual heating steps (Fig. 1e,f). The latter suggests thermal alterations. Hence, the failure of Thellier experiments around 350–400°C corresponds with the unblocking temperature of the LT component, but cannot be explained by this unblocking alone.

The slight increase in χ and decrease in IRM, B_{cr} and B_c suggest the occurrence of gradual irreversible magnetic changes between 200 and 450°C. These might contribute to the failure of the Thellier experiments. Taking into account that thermomagnetic runs are reversible and changes in magnetic parameters are limited below 500°C, it is unlikely that these changes reflect large chemical alterations. The slight increase in χ and decrease in IRM and coercivities may be due to domain rearrangements, caused by reduction in microstress after heating (Smith and Merrill, 1984; Enkin and Dunlop, 1987). Kosterov and Prévot (1998) related similar observations (Thellier diagrams with decreasing NRM intensities and no increasing TRM intensities together with decreasing coercivities) in Jurassic Lesotho basalts by a rearrangement of magnetic domains. However, with our measurements it is not straightforward to explain the failure of the Thellier experiments from 400°C. Nevertheless, the results can be used for their initial purpose: checking the validity of the PI determinations in terms of paleofield intensity.

5.3. Validity of paleointensity measurements

This study shows the presence of two remanence carriers in the Tres Cruces lava flow. When heated to 580°C, the low-temperature titanomagnetite component transforms further into a purer magnetite. Although this alteration is hardly visible in AF decay curves (Fig. 3), it leads to some doubts whether comparing NRM with a TRM imparted when heating the sample at 580°C provides accurate PI estimates. This is because the latter remanence might not be thermoremanent but partly carried by a chemical remanent magnetization (CRM). The Rolph and Shaw (1985) modification may not correct for this alteration because ARM AF decay curves are not diagnostic for transformation of Ti-rich titanomagnetite into a magnetite–ilmenite intergrowth. As the effect of this alteration varies per sample (Figs. 2 and 7), the variations in the paleointensity estimates determined with the Shaw-method (Gonzalez et al., 1997) may be related to this transformation of Ti-rich titanomagnetite into magnetite.

PI values determined with the Thellier and Thellier (1959) method are more consistent, but the rock magnetic results suggest that large multi-domain grains may carry them. This finding and the observation that the NRM demagnetization diagrams show an overprint up to 200°C lead to serious doubts about their reliability in terms of paleofield recorders. Therefore, our multi-proxy magnetic approach may serve for future studies. Firstly, it may separate high-quality paleointensity data from other paleofield estimates. Secondly, it shows the difficulty in obtaining good paleofield intensity records from basalts, which contain both titanomagnetite and magnetite.

6. Conclusions

Magnetic measurements show the presence of a Ti-rich titanomagnetite and a pure magnetite in the Tres Cruces lava flow (Central Mexico). χ, IRM and hysteresis parameters change when the samples are heated above 500°C. High- and low-temperature measurements indicate that these changes reflect alteration of the Ti-rich titanomagnetite into magnetite. The exact relationship between NRM properties and these rock magnetic results is a matter of debate. Nevertheless, the latter indicates that the large dispersion in paleointensity estimates obtained with the Shaw (1974) method may be related to the transformation of titanomagnetite into magnetite. Secondly, the failure of the Thellier and Thellier (1959) method around 350–400°C and the unblocking temperature of the Ti-rich titanomagnetites in the same temperature range indicate that the latter ''paleointensity'' estimates might be carried by the titanomagnetite. As the low temperature titanomagnetite component may have multi-domain properties, the geomagnetic significance of these paleointensity estimates needs to be verified by performing a paleo- and rock magnetic study on a nearby lava flow with a similar age.

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