A cooling rate bias in paleointensity determination from volcanic glass: An experimental demonstration

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[1] The suitability of volcanic glass for paleointensity determinations is the basis of many studies. The dominant single domain (SD) magnetic remanence carriers, the pristine character of volcanic glass, and the possibility to correct paleointensity data for cooling rate dependence using relaxation geospeedometry are all arguments that have been made in favor of this technique. In the present study the validity of cooling rate correction is tested using remelted volcanic glass. To obtain a stable multicomponent glass, with ideal magnetic properties, a natural phonolitic glass from Tenerife was remelted in air to avoid heterogeneity and degassing in later experiments. Further, it was tempered for altogether 10 hours at 900°C to yield a sufficient concentration of magnetic remanence carriers. To exclude nucleation or crystallization, six samples were then heated to about 60°C above the calorimetric glass transition temperature (≈660°C) and quenched at different rates from 0.1 to 15 K/min. Rock magnetic measurements show that low titanium titanomagnetite in the SD range is the main remanence carrier. After performing paleointensity experiments using a modified Thellier method, the dependence of the thermoremanence on cooling rate was investigated. Using the synthesis cooling rates and the experimentally determined magnetic cooling rate dependencies we were able to correct the data and obtained a mean paleointensity of 46.9 ± 1.3μT, which reflects the ambient field of 48μT within error. The uncorrected mean paleointensity corresponds to a 18% larger value of 56.5 ± 0.9μT. Therefore, application of a cooling rate correction is essential to obtain the correct ancient magnetic field intensity from SD assemblages in volcanic glass.

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1. Introduction

[2] Obtaining accurate values of absolute paleomagnetic field strength is of central interest for a variety of geoscientific problems, ranging from the Earth's deep interior to the magnetosphere. Based on paleointensity information two preferred states of the geodynamo were postulated, a low-field and a high-field state [*Shcherbakov et al.*, 2002; *Heller et al.*, 2003], suggesting significantly distinct field generation processes within the Earth's core. The predominant field intensity state during long lasting magnetic quiet zones, like the Cretaceous normal superchron, has been studied deeply [*Prévot et al.*, 1990; *Pick and Tauxe*, 1993; *Cottrell and Tarduno*, 2000]. The evolution of the Archaean magnetic field strength is investigated for the evolution of the magnetosphere, shielding the early Earth's atmosphere from solar-wind erosion [*Tarduno et al.*, 2007]. Even dating of burned archeological artifacts can be achieved by comparing archeointensity determinations with known historic intensity evolution curves [*Pavón-Carrasco et al.*, 2008].

[3] Unfortunately reliable paleointensity values are difficult to obtain because many factors can bias the results. One of these biasing mechanisms, which is only seldom considered, is related to a difference between cooling rates in nature and during the laboratory paleointensity experiment. It has been shown theoretically and experimentally that single domain (SD) particles, which are usually thought to provide the best paleointensity record according to other magnetic-domain-state-related biasing factors, are most strongly affected by cooling rate differences. Often, significant overestimates of the geomagnetic field are observed [*Halgedahl et al.*, 1980; *Fox and Aitken*, 1980; *McClelland-Brown*, 1984; *Chauvin et al.*, 2000; *Leonhardt et al.*, 2006]. Therefore, a technique to enable correcting the cooling rate effect, is essential to obtain accurate paleointensity data.

[4] For archeological materials, mostly burned artifacts like potsherds, such corrections are sometimes conducted

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using heating/cooling cycles, which are thought to resemble the original ancient burning condition [*Fox and Aitken*, 1980; *Chauvin et al.*, 2000; *Genevey and Gallet*, 2002]. For volcanic rocks, however, cooling rates are rarely considered for [*Bowles et al.*, 2005]. Based on historical dated obsidians from Lipari, Italy, *Leonhardt et al.* [2006] proposed a cooling rate correction technique, which can be applied to volcanic glasses. The physical state of glass contains a thermal history which is locked in at the glass transition [*Dingwell and Webb*, 1990; *Dingwell*, 1995]. Thus, natural glasses contain a record of their natural cooling rates. By using relaxation geospeedometry [*Wilding et al.*, 1995; *Gottsmann and Dingwell*, 2001; *Potuzak et al.*, 2008; *Nichols et al.*, 2009] natural cooling rates of glasses can be reconstructed. Laboratory cooling rates are known and the magnetic cooling rate dependence of the thermoremanent magnetization (TRM) is determined experimentally. Using a linear extrapolation function, the absolute paleointensity values of the pristine, SD dominated glass can then be determined.

[5] The validity of the cooling rate correction technique and its inherent assumptions are tested in this study. Six samples of remelted volcanic glass are investigated, which were quenched under increasingly faster cooling conditions and acquired their remanent magnetization in a known magnetic field. Thus, an analysis of the influence of different cooling histories on the determined paleointensity is possible and the extent of any field overestimate can be quantified.

2. Sample Preparation

[6] Samples were prepared at the Department of Earth and Environmental Sciences of the University of Munich. To obtain a stable multicomponent glass with ideal magnetic properties, circa 500g of a natural phonolitic glass from Lavas Negras on the north side of Teide, Tenerife, was remelted in a thin-walled platinum crucible (Figure 1a) using a Nabertherm HT14/07 furnace operating at 1600°C in air. The sample was held at these conditions for circa 12 hours to ensure homogeneity, volatile escape and fining of bubbles. Rock magnetic experiments for the glassy products of this fusion showed only paramagnetic contributions. Therefore, the sample was tempered at 900°C in air to yield a sufficient concentration of magnetic remanence carriers. In preliminary qualitative DTA (Differential Thermal Analysis) measurements an exothermal peak at 835°C was interpreted as the beginning of crystallization. Thus, in order to ensure limited growth yet significant nucleation of crystals the annealing temperature was chosen to lie slightly above these first signs of crystallization. The sample was heated with ≈7.5 K/min up to 900°C and held there for 2 hours, then subsequently cooled down by switching off the electrical power of the oven and opening its door. This procedure was repeated twice with annealing of 3 hours and 5 hours, respectively. After each annealing step, rock magnetic experiments showed an increasing abundance of magnetic minerals and a sufficient concentration was found after the 5 h step. Next, the glass transition temperature T_g was determined by relaxation geospeedometry. For this purpose, the heat capacity c_p of the glass was measured in a Differential Scanning Calorimeter (DSC) and the peak in c_p was taken as T_g. Detailed descriptions of the experimental procedures are given by *Wilding et al.* [1995], *Gottsmann and Dingwell* [2001], *Potuzak et al.* [2008] and *Nichols et al.* [2009]. For one sample, that had previously been cooled with 5 K/min, T_g = 663°C was determined. To exclude further nucleation or crystallization six miniature cores of 5 mm diameter were then heated in air in a Netzsch 402C dilatometer with 10 K/min to circa 60°C above T_g at around 720°C, kept at this temperature for five minutes and each quenched at one of the following cooling rates: 0.1, 0.5, 1, 5, 10 and 15 K/min. Measurement of the magnetic field intensity inside the bifilar wound Netzsch dilatometer using a fluxgate sensor showed that it is identical to the ambient magnetic field at Munich, i.e., 48 μT. Sample names (e.g., LNN3-600-0.1) indicate the natural sample from which the remelted glass was derived (LNN3: Lavas Negras North, sample 3), holding time at 900°C (600 min) and quench rate (e.g., 0.1 K/min).

3. Magnetic Mineralogy and Domain State

[7] Rock magnetic measurements to identify the magnetic mineralogy and its domain state were done at a Variable Field Translation Balance and analyzed using the RockmagAnalyzer software [*Leonhardt*, 2006]. Isothermal remanent magnetization (IRM) acquisition, isothermal backfield curves (Figure 1c) and hysteresis loops at room temperature as well as thermomagnetic curves (applied field: about 400 mT, Figure 1d) were measured in that order on specimens belonging to the same miniature cores as those used for paleointensity experiments. In addition, sample LNN3-600-0.1 was stepwise heated to 420°C, 480°C and 530°C and after each of these thermomagnetic measurements, the backfield and hysteresis measurements were repeated at room temperature to test for thermal stability.

[8] Curie temperatures, hysteresis and backfield parameters as well as tail and quality parameters of the later paleointensity experiments can be found in the auxiliary material.[1] Heating and cooling curves of the thermomagnetic measurements are reversible, indicating an absence of alteration (Figure 1d). This is further supported by the almost identical hysteresis and backfield parameters of sample LNN3-600-0.1 after the different heating steps. Determinations of second derivatives for the thermomagnetic curves of all samples indicate a Curie temperature T_C between 410 and 460°C, corresponding to titanomagnetite ($Fe_{3-x}Ti_xO_4$) with titanium contribution of $x \approx 0.22$. Between 70 and 90°C a very small kink is found in the thermomagnetic curves of all samples. *Buddington and Lindsley* [1964] have shown that in silicic melts low-titanium titanomagnetite can coexist only with high-titanium hemoilmenite, but not with high-titanium titanomagnetite. Thus, the low T_C of about 80°C would relate to titanium contribution $y \approx 0.6$ in hemoilmenites ($Fe_{2-y}Ti_yO_3$). This hemoilmenite fraction, however, has a negligible contribution to the whole magnetization.

[9] T_C and therefore also the blocking temperatures T_b lie well below T_g of ≈660°C, excluding any bias to paleointensity determination related to the glass transition

[1] Auxiliary materials are available in the HTML. doi:10.1029/2009JB006964.

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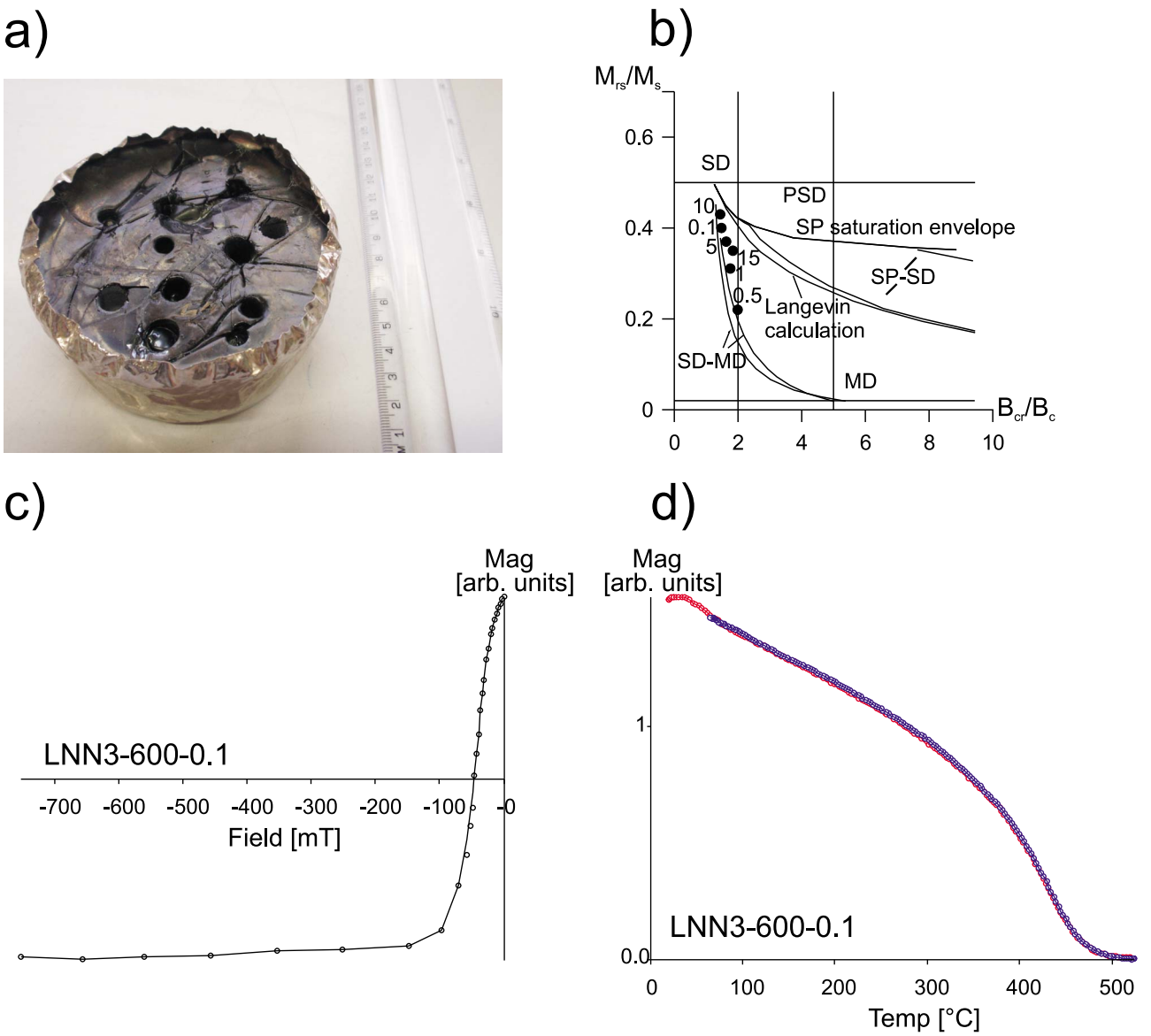

Figure 1. (a) Picture of the remelted glass in the platinum crucible; (b) hysteresis parameters of the different samples (indicated by the respective cooling rates), shown in a Day plot [*Day et al.*, 1977] with domain state related boundaries and mixing lines by *Dunlop* [2002]; and (c and d) representative backfield and thermomagnetic (reduced for paramagnetism) curve (sample LNN3-600-0.1).

[*Smirnov and Tarduno*, 2003]. Fast saturating IRM curves as well as Bloemendal S_{300} values [*Bloemendal et al.*, 1992] close to 1 are indicative for magnetically soft material, as expected for titanomagnetite as main remanence carrier. Hysteresis parameters analyzed according to *Dunlop* [2002] show close to SD behavior, although minor variations along the SD-MD mixing line are observed (Figure 1b). IRM and backfield data plotted as suggested by *Henkel* [1964] lie close to the line for ideal Stoner-Wohlfarth particles, further supporting a predominant SD character of the remanence carrying particles (see auxiliary material). This conclusion is underlined by repeated thermal demagnetizations during the Thellier-Thellier experiments. Hereby the absence of any magnetization tails (Figure 2) confirms SD behavior of the remanence carrying fraction.

[10] There is no obvious trend in hysteresis, backfield and/or T_C data that can be related to the varying cooling rates. All measurement parameters are very similar. This supports our hypothesis that by heating only to about 60°C above the glass transition temperature before quenching under different cooling conditions, new nucleation or growth of crystals can be neglected. Thus, in this manner, rock magnetically very similar samples containing SD titanomagnetites have been obtained, which differ essentially only in their cooling histories.

4. Paleointensity Determination

[11] All paleointensity determinations were conducted in a MMTD20 thermal demagnetizer at the paleomagnetic laboratory of LMU Munich in Niederlippach. Laboratory

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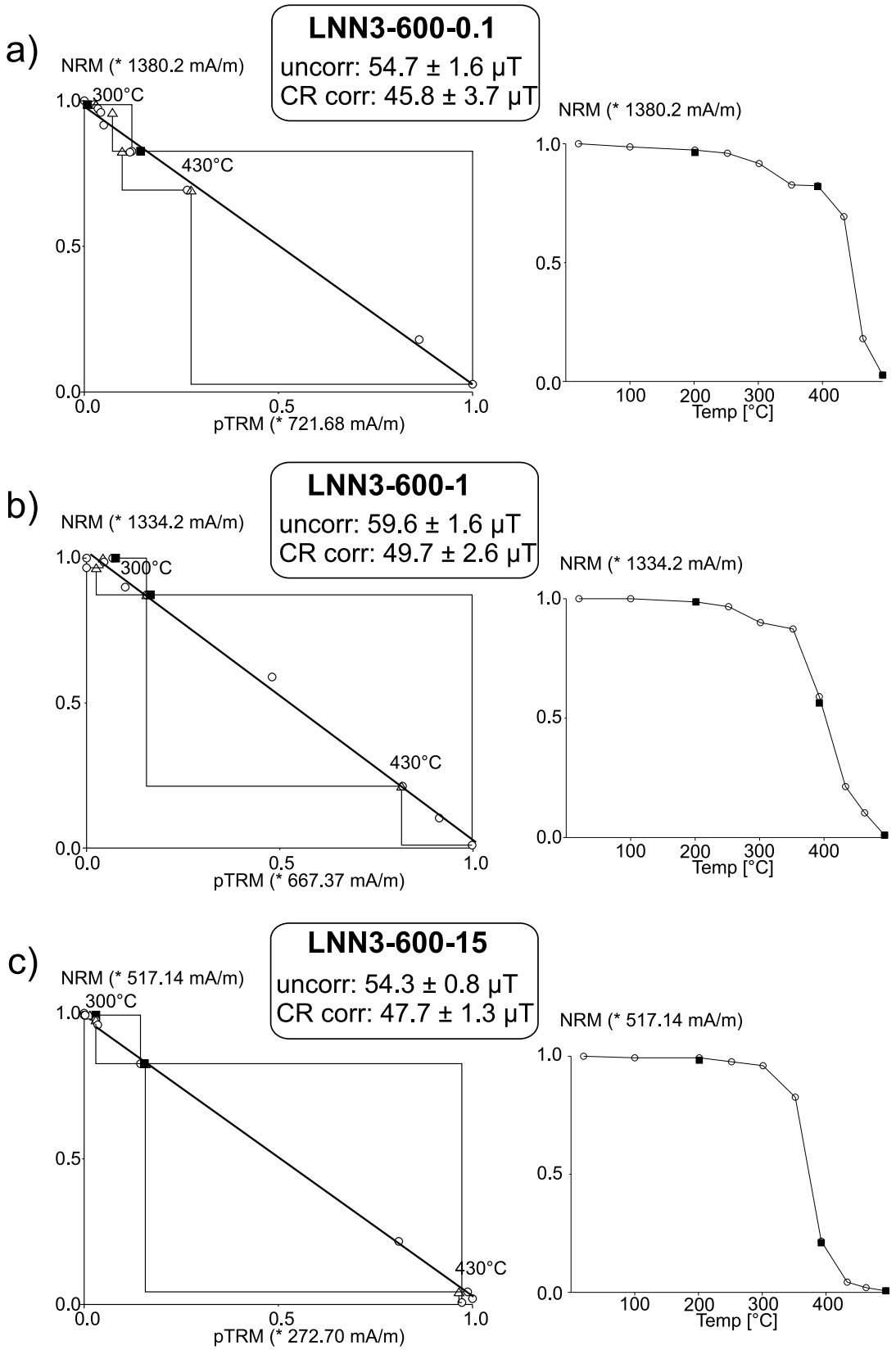

Figure 2. NRM/pTRM diagrams (triangles: alteration checks, squares: additivity checks) and respective decay plots of demagnetization steps (squares: tail checks) for three of the remelted samples with cooling rates of (a) 0.1, (b) 1, and (c) 15 K/min. Temperatures indicate temperatures on the display of the Shaw oven; especially at higher steps, sample temperatures are significantly lower. Intensity results are given for uncorrected and cooling rate (CR) corrected analysis.

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Table 1. Paleointensity Results and Correction[a]

Sample	q	$H_{UC} \pm \sigma_{UC}$ (μT)	f_{CR}	$H_{CR} \pm \sigma_{CR}$ (μT)
LNN3-600-0.1	20.7	54.7 ± 1.6	1.194 ± 0.038	45.8 ± 3.7
LNN3-600-0.5	20.0	57.1 ± 1.3	1.396 ± 0.028	40.9 ± 2.9
LNN3-600-1	26.5	59.6 ± 1.6	1.199 ± 0.016	49.7 ± 2.6
LNN3-600-5	25.4	53.4 ± 1.0	1.260 ± 0.029	42.4 ± 2.5
LNN3-600-10	88.5	57.6 ± 0.3	1.181 ± 0.008	48.8 ± 0.7
LNN3-600-15	33.0	54.3 ± 0.8	1.138 ± 0.009	47.7 ± 1.3
Weighted average		56.5 ± 0.9		46.9 ± 1.3

[a]Sample names contain the original sample reference of the remelted glass (LNN3: Lavas Negras North, sample 3), the tempering time at 900°C (600 min) and the quench rate in K/min (e.g., 0.1 K/min). H_{UC} and H_{CR} are the paleointensity values of the different samples with associated errors for the uncorrected and cooling rate corrected determinations, respectively. Uncertainties are determined by error propagation and include the scatter about the straight line segment and the uncertainty related to the cooling rate correction factor f_{CR}. Also shown are weighted averages of the intensity values and associated uncertainties. The quality factor q was chosen as weighting parameter for H_{UC} and $1/\sigma_{CR}$ for H_{CR}.

fields of 30 ± 0.1 μT were used for all measurements and applied during heating and cooling. Intensity measurements were done using the modified Thellier-technique MT4 [*Leonhardt et al.*, 2004a], which is a zero-field first method that includes partial TRM (pTRM) checks (in-field), additivity checks (zero-field) [*Krása et al.*, 2003], and pTRM-tail checks (zero-field) [*Riisager and Riisager*, 2001]. Directional differences between the applied field and the natural remanent magnetization (NRM) of the pTRM-tail check are taken into account according to *Leonhardt et al.* [2004b]. All determinations were analyzed using the ThellierTool4.21 software and its default criteria [*Leonhardt et al.*, 2004a]. The samples do not show any magnetic anisotropy as was indicated by anisotropy of magnetic susceptibility (AMS) being well below the signal of the sample holder and B_{cr} values of about 40 mT. An isotropic behavior of the samples is reasonable as during the production of the remelted glass no anisotropy "source", such as the flow direction of a natural obsidian deposit, was present, i.e., no differential stresses or resulting strains obtained during the synthesis.

[12] The quality of individual paleointensity determinations is very good. Linear trends covering a fraction of the NRM $f \geq 93\%$ were analyzed for all samples. Quality factors q exceed 20, no alteration is present (difference ratio DRAT < 2.4%) and, as mentioned before, magnetization tails are small. Uncorrected paleointensity results range from 53.4 ± 1.0 to 59.6 ± 1.6 μT, giving a mean weighted paleointensity of 56.5 ± 0.9 μT (Table 1; weighting factor q), which exceeds the ambient field during experimental cooling by about 18%. NRM/TRM plots and decay plots of three samples with cooling rates of 0.1, 1 and 15 K/min are shown in Figure 2.

5. Cooling Rate Dependency

[13] Although a dominating SD behavior is found, hysteresis and pTRM tail measurements suggest slightly varying domain states of the different samples. Due to domain state variations, also a varying cooling rate dependency of the TRM is to be expected [*McClelland-Brown*, 1984] and a direct correlation between paleointensity and cooling is hampered. Following *Leonhardt et al.* [2006], the magnetic cooling rate dependency was measured. A field of 30 ± 0.1 μT was applied. For fast cooling the cooling fan of the MMTD20 furnace was used just like during the paleointensity experiments, while slow cooling was obtained through cooling without fan operation. Laboratory cooling rates were determined from basaltic samples that have the same size and volume as the studied miniature cores between 700°C and 600°C. This temperature range was chosen, because it includes the glass transition at about 660°C and determination of natural cooling rates is only possible at the glass transition. Although the change in cooling rates down from the glass transtion to blocking temperatures may not be exactly the same in nature and in the laboratory, this is so far the closest one can get to a comparison between natural and laboratory cooling. To get the mean cooling rates of our furnace in this temperature range the temperature decrease, measured by a thermocouple inside one of the basaltic samples, was monitored versus time. An initial fast heating/cooling cycle with a cooling rate of ≈410 K/min was used to imply a TRM ($TRM_{f,1}$). Then a 74-fold slower heating/cooling cycle with a cooling rate of ≈5.5 K/min ($TRM_{s,1}$) and again a higher one ($TRM_{f,2}$) in order to check for alterations during the experiment, were performed. $TRM_{f,1}$ and $TRM_{f,2}$ differ by ≤2%, confirming that alteration is absent. These differences are also used as error estimate for inaccuracy in determination of both $TRM_{s,1}$ and $TRM_{f,1}$. A conservative estimate of the uncertainty in cooling rate determination is 10% for fast cooling and 5% for slow cooling.

[14] For slow cooling experiments the TRM intensity is 11% to 26% larger than for fast cooling, as would be expected for a non-interacting SD assemblage [*Halgedahl et al.*, 1980; *Dodson and McClelland-Brown*, 1980]. The magnetic cooling rate dependency is extra-/interpolated to the original cooling rates as used during the synthesis of the remelted glasses. The laboratory measured $TRM_{f,1}$ and $TRM_{s,1}$, both normalized to $TRM_{f,1}$, are plotted versus $\ln(\dot{T}_{f,1}/\dot{T})$ (Figure 3). A linear extra-/interpolation is valid according to *Halgedahl et al.* [1980], if non-interacting SD particles, dominantly blocking close to the respective blocking temperature, are the remanence carriers. As our samples are at least close to SD and unblock sharply within about 50°C, these conditions are fulfilled. The previous error estimates for inaccuracy of laboratory cooling rate and magnetization determination allow a minimum/maximum error propagation towards the natural cooling rates (Figure 3). The obtained cooling rate correction factor f_{CR} = TRM/$TRM_{f,1}$ is then used to correct the paleointensity values H_{UC} (Table 1) by

$$H_{CR} = \frac{H_{UC}}{f_{CR}}. \quad (1)$$

[15] Error propagation, including the uncertainties of the paleointensity experiments and of the cooling rate correction factor determination, gives the total uncertainty σ_{CR} of the individual cooling rate corrected paleointensity values. Application of cooling rate correction significantly reduces the paleointensities to a mean value of 46.9 ± 1.3 μT (weighting factor $1/\sigma_{CR}$, Table 1). This reflects the ambient

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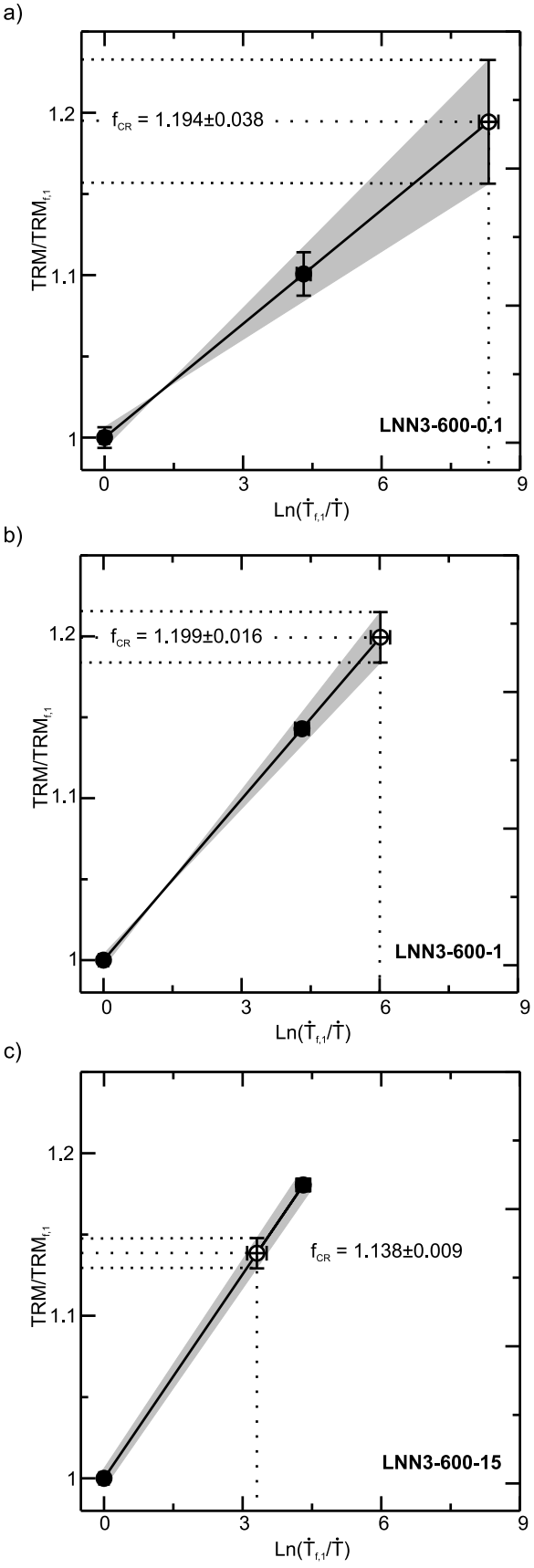

Figure 3. Cooling rate correction using the laboratory measured cooling rate dependency (solid symbols) and related uncertainties, as well as the linear extra-/interpolation of the TRM dependency to the synthesis cooling rates (open symbols). Same samples as in Figure 2, i.e., samples cooled with (a) 0.1, (b) 1, and (c) 15 K/min.

field value of 48 μT very good and verifies the need for coolingrate correction in SD dominated materials.

6. Discussion and Conclusion

[16] Rapid laboratory cooling during paleointensity determination, opposed by slow cooling during ancient NRM acquisition, leads to paleointensity overestimates and hence a strong bias towards erroneously higher field values in SD-dominated paleointensity recorders. In this study, laboratory experiments were conducted on remelted volcanic glass with known cooling histories during TRM acquisition, whose SD character is confirmed by rock magnetic measurements and small magnetization tails. A 75-fold lower cooling rate results in 11% to 26% higher TRM acquisition values, which exceeds the theoretically predicted magnetic cooling rate dependencies of SD magnetite which would be in the order of 10% [*Halgedahl et al.*, 1980; *Dodson and McClelland-Brown*, 1980]. TRM overestimates exceeding the theoretically predicted values were also observed in other experimental studies on archeomagnetic materials [e.g., *Genevey and Gallet*, 2002]. A possible reason for the here observed overestimates of 18% on average, could be related to titanomagnetites (TM20) as remanence carriers and not SD magnetite or hematite as used in the theoretical studies. Titanomagnetites are characterized by different magnetic parameters, of which in particular the blocking temperature relationship, relaxation times and anisotropy are relevant for cooling rate dependencies. It should also be mentioned that overcorrections of the expected paleointensity and largest TRM overestimates are found particularly for two samples LNN3-600-0.5 and LNN3-600-5. These two overcorrections give rise to slightly larger uncertainties of the average cooling rate corrected field value compared to the uncorrected paleointensity estimate. Nevertheless the originally applied field is then correctly reproduced (Table 1). The reason for overcorrection in the two specimens remains elusive, because all determined rock magnetic parameters are similar to the other specimens. Only indications for slightly larger MD contributions are found for both samples (see Day plot of Figure 1b and tails in Table S1 in the auxiliary material) although both samples are dominated by SD remanence. MD contribution, however, would be expected to reduce the TRM ratio [*McClelland-Brown*, 1984; *Fabian and Leonhardt*, 2009].

[17] Cooling rates of 290 K/min during the Thellier experiment are larger than the cooling rates of 0.1 to 15 K/min used for initial TRM acquisition. These initial cooling rates correspond well to the middle range of naturally observed cooling rates in volcanic glasses (140 K/min to 0.001 K/min [*Gottsmann et al.*, 2004; *Gottsmann and Dingwell*, 2002]), yet they are already sufficient to generate a maximum field overestimate about 18%. Similar overestimates of 22% are reported from natural volcanic glasses [*Leonhardt et al.*,

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2006]. These large overestimates clearly underline that cooling rate correction is necessary to obtain correct past geomagnetic field values in SD assemblages. Such correction involves two basic requirements: (1) knowledge of the natural cooling rate during NRM acquisition; (2) a known dependency between TRM and cooling rate if extra- or interpolation is necessary.

[18] The first requirement can be achieved for volcanic glass, where natural cooling rates can be determined by relaxation geospeedometry [*Wilding et al.*, 1995]. This technique also yields the glass transition temperature (T_g), which marks the transition between a supercooled liquid and a solid glass. T_C of our samples (≤440°C) is well below T_g of 660°C. Hence, the samples are suitable for paleointensity determination as TRM acquisition occurs in a solid-like state. Further, for archeomagnetic investigations on burned artifacts, results from experimental archeology can be consulted to determine the natural cooling rate [e.g., *Genevey and Gallet*, 2002]. However, for the most widely used material in absolute paleointensity studies, basaltic rocks, an accurate determination of cooling history remains elusive. The second requirement, extra-/interpolation towards the natural cooling rate, requires a known magnetic cooling rate dependency. It has been shown theoretically [*Halgedahl et al.*, 1980] that a linear extra-/interpolation as used in Figure 3 is applicable for a narrow unblocking spectra as observed in our samples. Magnetic cooling rate dependencies of wide unblocking spectra and PSD to MD dominated materials, which are usually considered as less reliable for paleointensity studies, will require further investigation.

[19] The treatment of uncertainties of extra-/interpolation follows the outline of *Leonhardt et al.* [2006]. The errors for the laboratory cooling rates and uncertainties in the TRM acquisition experiment are conservative estimates. Thus, extra-/interpolation of these uncertainties towards the natural cooling rates (Figure 3) gives a realistic upper limit for the uncertainties associated with cooling rate correction. The final accuracy of individual cooling rate corrected paleointensity determinations (σ_{CR}) is related to the sum of uncertainties caused by deviations from the straight line segment and the error of the correction factor f_{CR}. Therefore, σ_{CR} comprises the quality of all successive experiments for each sample. The weighted average paleointensity of the remelted glass samples is then determined using $1/\sigma_{CR}$ as weighting parameter.

[20] After cooling rate correction our remelted glass samples give a paleointensity of 46.9 ± 1.3 μT, which reflects the originally applied field value of 48 μT within its margins of error. Without the two overcorrected values, the applied field of 48 μT would be exactly obtained after correction (48.3 ± 0.6 μT). The uncorrected average value of 56.5 ± 0.9 μT exceeds the original field by 18%. Therefore, application of cooling rate correction is essential to retrieve the correct ancient field value in case of SD dominated material.

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