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The ionic reordering in Fe-Ti ferrimagnetics as a dominant source of the reversed RM in basaltic rocks

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Abstract: The basalts of the Pleistocene to Quaternary age from southern part of Slovakia were investigated. These are the basalts containing predominantly the titanomaghemites (Ti-Mgh-es) which carry the reversed remanent magnetization (RM). The partial thermoremanent magnetization (PTRM) in original and artificially prepared samples was induced in the geomagnetic field of normal polarity and intensity $H \approx 48\mu$T. The PTRM reversed by the self-reversed mechanism was induced in all the above-mentioned types of basalts, in the interval 300 to 475° C. In this temperature interval an increase of magnetic susceptibility with a peak about 450° C was detected. The acquired reversed RM and PTRM of basalts have been explained on the basis of the reordering of magnetic ions in the A and B sub-lattices of the oxidized Fe-Ti ferrimagnetics in basalts.

Key words: the Ti-Mgh phase, ionic reordering in ferrimagnetic – the source of the self-reversed remanent magnetization

1. Introduction

In paleomagnetism and the magnetostratigraphy the normal and the reversed polarities of the remanent magnetization (RM) of rocks (a field-reversal hypothesis) are commonly applied for an interpretation. A time dependent change of the polarity of the geomagnetic field was derived by comparing the polarity of the RM with geological, radiometric or paleontological ages of the respective rock. Because the carriers of magnetic properties in rocks are mostly the Fe-Ti compounds with a very intricate properties, one could prefer an idea that the reversed RM of rocks is not a reflection of some reversally oriented geomagnetic field but that they have a self-reversal origin. Previously I presented the results of the reproducible self-reversed laboratory induced partial thermoremanent magnetization (PTRM) for many types of the intermediate volcanic rocks (*Orlický, 2001, 2002, 2002a, 2002b, 2002c, 2003, 2003a).*

My first paleomagnetic studies of basalts were focused to an assessment of the succession, using normal or reversed RM and the radiometric ages of rocks *Orlický et al. (1982).* The paleomagnetic and radiometric data were successively enlarged and more precise interpretation of data was made (*Orlický et al., 1996; Orlický, 2004*). Because the interpretation of the paleomagnetic data and the correlations with the radiometric ages appeared to be gradually not consistent, I refused to continue in correlating polarities of RM of basalts with the paleomagnetic polarity time scales. The phenomena such as the magnetostatic interactions and the self-reversal RM appeared in those results which dominantly influenced the magnetic polarity of RM and the magnetic behaviour of rocks. I started with a study of magnetic minerals by own method and with the apparatus which I completed in 1985 (*Orlický, 1990*), to obtain the Curie temperatures and the magnetic behaviour of Fe-Ti magnetic minerals in the rocks. These new results have also reflected the changes in magnetic phases of magnetic minerals of the rocks, except for their Curie temperatures. In *Orlický et al. (1992)* and *Orlický (2004)*, and in all further works a detailed study of the magnetism carriers, magnetic properties of the magnetic minerals and their extraordinariness were completed (*Orlický et al., 2005; Orlický, 2006, 2006a, 2006b, 2006c, 2007*). A realistic interpretation of magnetic and paleomagnetic data of basalts were presented in *Orlický (2006d).* In these works I outlined a conception of the so-called superparamagnetic behaviour of Fe-Ti magnetic minerals and an origin of the self-reversed RM of basalts was considered. Successively I enlarged the magnetic data to the study the hysteresis properties and the domain structure of Fe-Ti minerals of basalts (*Orlický, 2008, 2008a*). All results in the cited works, especially the magnetic behaviour of Fe-Ti and Fe minerals, have persuaded myself that the reversed RM has not been generated by reversed geomagnetic field during origin of the rocks, but by the self-reversal mechanism. Despite some theory about a possibility of self-reversal arrangement of the reversed magnetization (*Néel, 1955*) not enough experimental work has been done to clarify the nature of the reversed RM of the rocks.

In this paper I present the results of the laboratory induced PTRM of natural compact basalts and the artificially prepared grinded basalt samples fixed in gypsum.

Some knowledge about the titanomagnetites (Ti-Mt-es), such as the basic carriers of magnetic properties in the basalts, their mineralogy, the alterations and their magnetic domain structures have been described. This topic is important with respect to a reality, that the continental and sub-marine basalts within large blocks, in many cases also in a very large regional areas are very frequently magnetized reversally. The goal of this study is to reveal the possible sources of the reversed RM of the rocks.

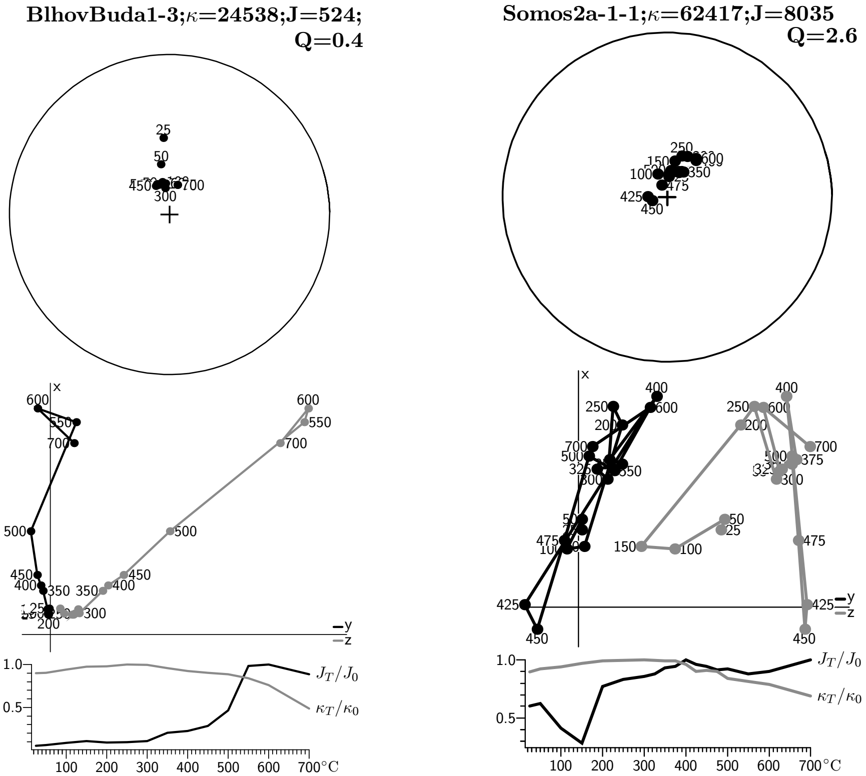
2. Experimental data

In the previous works there were distinguished the basalts with the Ti-Mt and Ti-Mgh bearing minerals from those with the Hem-Ilm bearing magnetic oxides, both types with the reversed RM (*Orlický, 2006d; Orlický and Funaki, 2008*). This article will deal with the former types of basalts.

An acquisition of partial thermoremanent magnetization (PTRM) of the samples in the today's geomagnetic field of the intensity $H \approx 48\,\mu\mathbf{T}$.
Previously, in paleomagnetic practice the standard laboratory procedures have been commonly applied to evidence the most stable component of the RM of the rock. The alternating field (AF) or thermal demagnetization of the samples within a fully compensated external magnetic field were commonly used. The results of these methods can reveal the most stable component of the RM, either of normal or reversed orientation, but they could say nothing about the mechanism and an origin of the reversed RM of rocks.

The magnetization of the compact and artificially prepared samples of volcanic rocks in the geomagnetic field of intensity $H \approx 48\mu\,\mathrm{T}$ was applied. An inducing of the PTRM at the following temperature steps was applied: 50, 100, 150, 200, 300, 350, 400, 450, 500, 600, 700° C. The samples were placed on the holder of the non-magnetic furnace. They were oriented in the positive direction of the field. A speed of heating of the samples was about 3°/min. During about 20 min., the magnetization at the specific temperature ($\mathrm{T}_{concr.}$) was realized. The samples were then cooled by speed

An inducing of PTRM of the basalt samples with normal RM



BlhovBuda1-3;κ=24538;J=524; Q=0.4

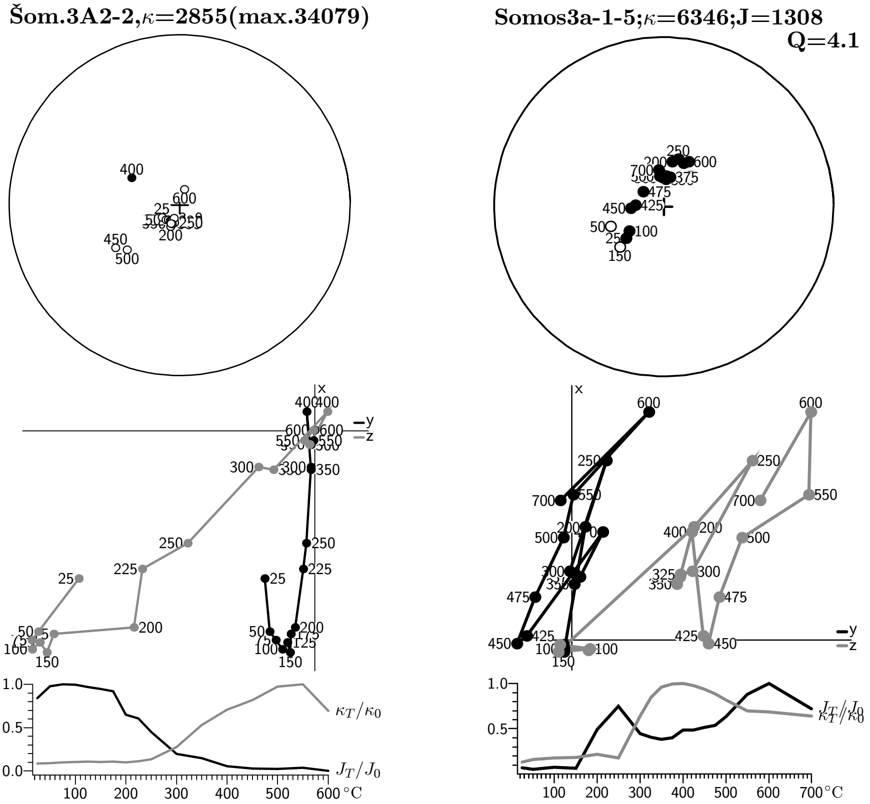
Somos2a-1-1;κ=62417;J=8035 Q=2.6

Fig. 1. Thermal inducing of natural basalt samples BlhovBuda1-3 and Somos2a-1-1, both with normal RM. Zijderveld diagrams and stereographic projections; • (○)-positive (negative) polarity of RM; κ – magnetic susceptibility (above the pictures: the κ is $\times 10^{-6}$ SI Units, and remanent magnetization J is in nano Tesla (nT)); κ_T at T, κ_0 – at 25° C; J_T (J_0) – remanent magnetization at T and at 25° C, respectively; Q – Koenigsberger ratio.

of about 3° C/min. to laboratory temperature. The RM of the samples was measured by the spinner magnetometer JR-5 at room temperature. The change of magnetic susceptibility (κ) was measured at room temperature by the susceptibility meter KLY-2, after each laboratory treatment of the samples (Figs. 1–3 and Figs. 5, 6).

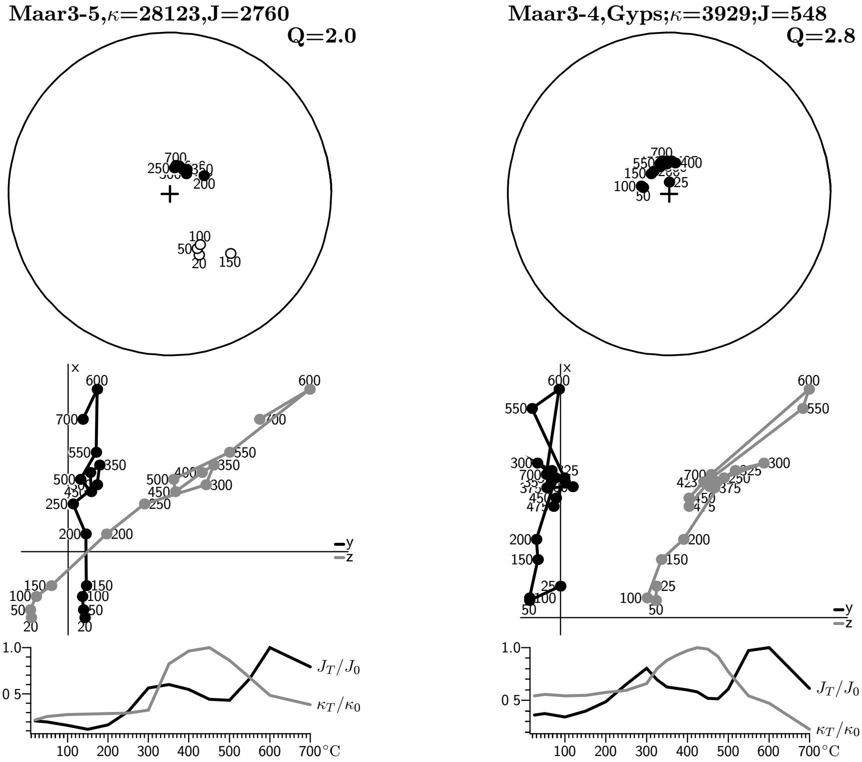
In Fig. 6, there is an example of inducing of the self-reversal PTRM of the artificially prepared samples (the grinded basaltic grains were fixed in non-magnetic gypsum). In these samples the PTRM was induced only at concrete temperature. During heating and cooling of the sample the field was fully compensated. During this process the sample Maar3-4kG received the reversed PTRM = – 36 nT, and the sample Velkop2-4kG received the

An inducing of PTRM of the basalt samples with reversed RM


Šom.3A2-2,κ=2855(max.34079)

Somos3a-1-5;κ=6346;J=1308 Q=4.1

Fig. 2. Thermal demagnetization (left) of the intensely altered basalt sample from the locality Šomoška (loc. 45; age = 4.06 Ma); Thermal inducing of natural basalt sample Somos3a-1-5 with reversed RM (right). Zijderveld diagrams and stereographic projections; • (∘) – positive (negative) polarity of RM; κ – magnetic susceptibility (above the pictures: the κ is $\times 10^{-6}$ SI Units, and remanent magnetization J is in nano Tesla (nT)); κ_T at T, κ_0 – at 25° C; J_T (J_0) – remanent magnetization at T and at 25° C, respectively; Q – Koenigsberger ratio.


Maar3-5,κ=28123,J=2760
Q=2.0

Maar3-4,Gyps;κ=3929;J=548
Q=2.8

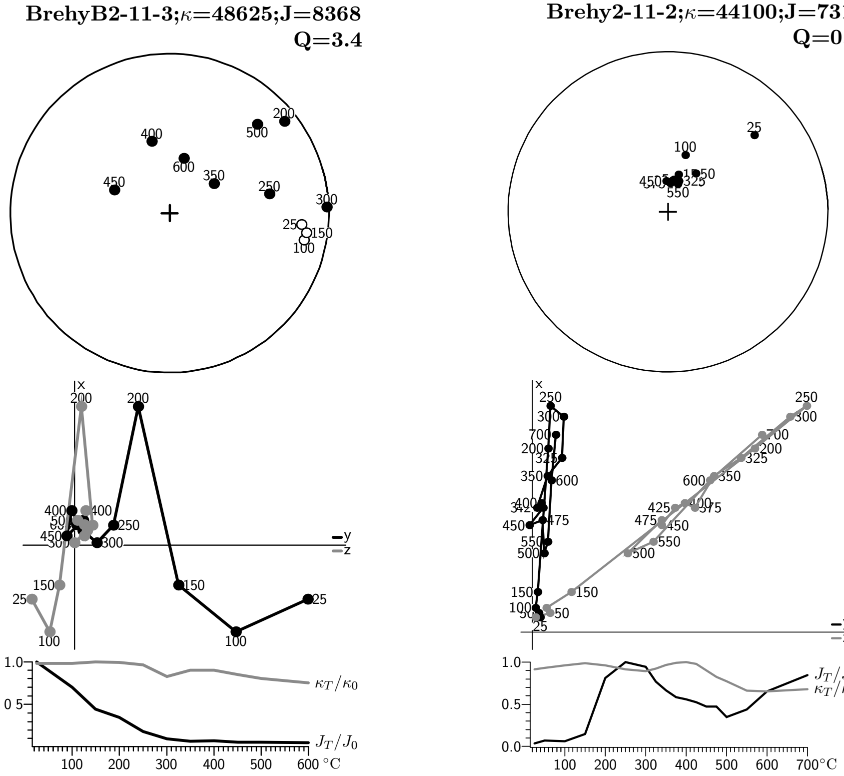
Fig. 3. Thermal inducing of natural basalt sample Maar 3-5 with reversed RM and the artificial grinded basaltic sample fixed in gypsum Maar 3-4 g. For other descriptions and explanations see Fig. 2.

reversed PTRM = – 100 nT. The samples from further three localities featured a strong tendency towards the acquisition of the reversed PTRM at 500° C.

Basic results

The results have shown that the behaviour of the PTRM and magnetic κ of the samples with normal RM differ significantly from those with the reversed RM during thermal treatment. In Fig. 1 we depict the results of PTRM acquisition of the samples of basalts with normal RM. The carrier of magnetism in the sample Blhov Buda is the magnetite with the Curie

Thermal demagnetization and an inducing of PTRM of the natural nepheline basanite from Brehy locality



BrehyB2-11-3;κ=48625;J=8368 Q=3.4

Brehy2-11-2;κ=44100;J=731; Q=0.3

Fig. 4. Thermal demagnetization of nepheline basanite sample Brehy B2-11-3 and thermal inducing (PTRM) of sample Brehy B2-11-2 in normally oriented magnetic field of intensity $H = 48\mu$ T. For other descriptions and explanations see Fig. 2.

temperature $T_C \approx 580°$ C and Verwey temperature $T_V \approx -150°$ C. No self-reversed PTRM was acquired in such types of basalts during the laboratory magnetization. A dominant carrier of magnetism in the sample Šomos2a1-1 is Ti-rich Ti-Mt with $T_C \approx 130°$ C. There is also a second Ti-Mt phase with $T_C \approx 230°$ C. We see from Fig. 1 that there is a change of PTRM as follows: an expressive decrease of PTRM (in nT) in the interval $25-150°$ C (-4528), rapid increase of PTRM in 150 to $\approx 200°$ ($+4528$), gradual increase in 200

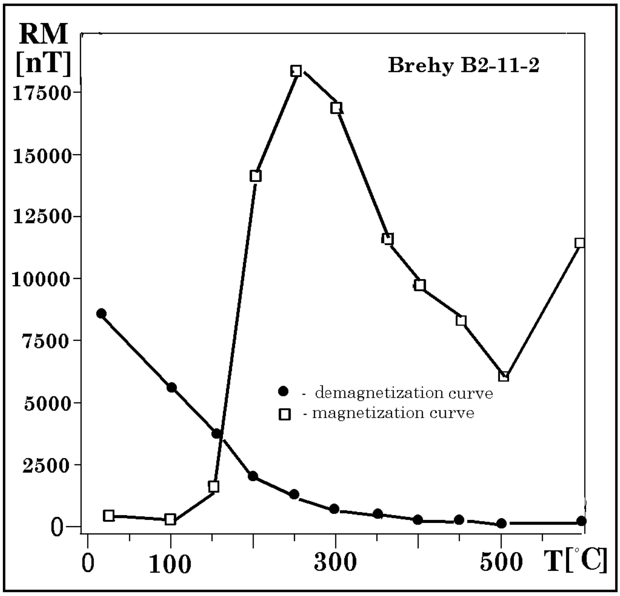
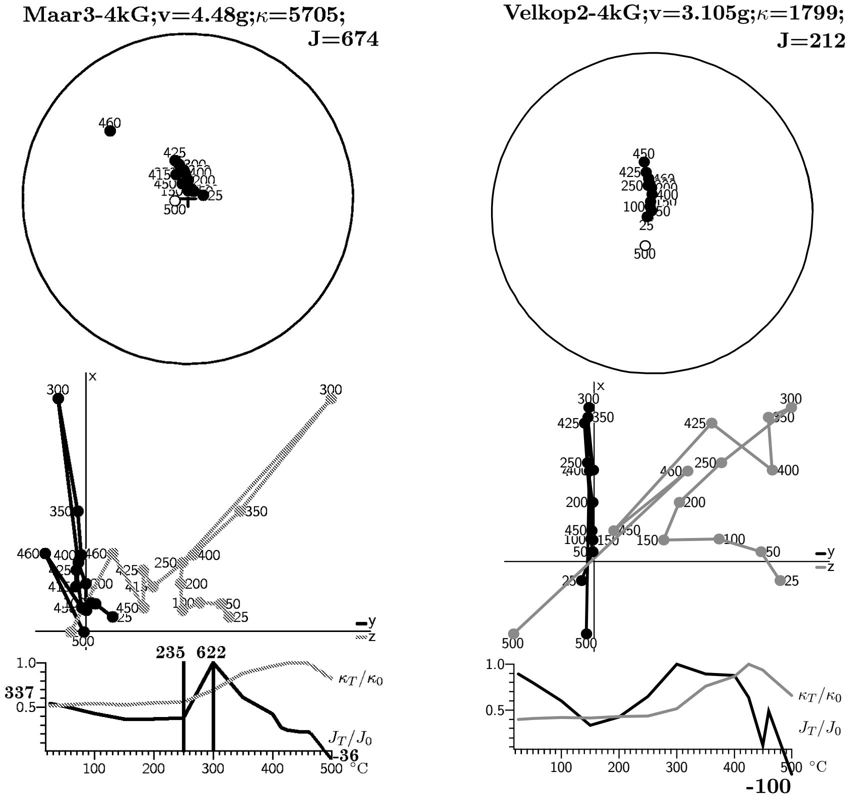


Fig. 5. The results of the Thellier method of nepheline basanite sample Brehy B2-11-2. Explanations see inside of the picture.

to ≈ 400° C (+6508), decrease in 400 to 550° C (−1798) and increase of PTRM in 550 – 700° C interval (1802 nT). The decrease of PTRM in the interval 400 – 550° C is evidenced also by the projection of end vector of RM into both xy and xz planes (Fig. 1). In the Ti-Mt bearing compact basalts and the artificial samples, there is gradual decrease of PTRM in the interval 25 to about 150 – 200° C. This decrease corresponds to demagnetization of residual RM due to destroying of the domains, of the first magnetic phase of the samples (the phase is documented with the Curie temperature measurement of the T-rich Ti-Mt samples). In the interval from 150 (200) mostly to about 300° C a conspicuous increase of PTRM (in some cases more than 10 times an initial value of RM) of the samples was acquired.

The basalts with the reversed RM have completely other type of temperature dependence of the PTRM and κ with regard to those with normal RM. The examples of such behaviour are shown in Figs. 2, 3. In the samples exampled, in those from other 13 localities, also in artificial samples, there was detected very intense decrease of PTRM in the interval from 300

An inducing of PTRM of artificial basalt samples, originally with reversed RM


Maar3-4kG;v=4.48g;κ=5705; J=674

Velkop2-4kG;v=3.105g;κ=1799; J=212

Fig. 6. Thermal inducing of artificially prepared samples Maar3-4k and Velký kopec2-4k; artificial samples – grinded basaltic sample fixed in gypsum; These samples were induced only at concrete temperature; heating and cooling of the sample in other temperature intervals was realized in fully compensated field (k). Other descriptions and explanations see in Fig. 2.

to about 475° C (500° C), with a minimum value mostly at about 450° C. An increase of κ is evident over the temperature of 300° C. The κ reaches the maximum value mostly at about 450° C and after that temperature it decreases. A peak of κ at about 450° C coincided mostly with the minimum value of PTRM of the samples. The decrease of PTRM results the generation of the reversed component of PTRM. The value of the self-reversed PTRM component of the compact samples from 13 localities is in the range −1346 to −7989 nT. The reversed PTRM of the rocks is carried by a secondary, low temperature oxidized Fe-Ti phase.

Except for the samples in Figs. 2, 3, the additional about 125 basalt samples from central and southern Slovakia and many samples from abroad have shown the above described κ and PTRM behaviour. E.g. some samples from three localities of the Eocene to Miocene age basalts from Bohemian Massif (*Orlický, 2002*), some basalts of the Neogene age and the Cretaceous to Jurassic nepheline basanites from Nigeria and some Cretaceous basalts from Syrian Arab Republic (*Orlický, 2003*).

A very important is a case of very young (cca 130 to 220 ky age) nepheline basanites from Brehy locality in central Slovakia. In this locality (very large basaltic quarry) there were sampled larger pieces of oriented rocks from 22 individual places. Several samples showed a tendency to have quite unstable reversed RM. The results of thermal demagnetization and those of inducing the PTRM of one sample are in Fig. 4. We see that the reversed RM of the sample survived up to about 150° C by its thermal demagnetization. During thermal magnetic inducing of the sample an expressive decrease of the PTRM is evidenced in the interval cca 300 to 500° C. Increase of κ is present in the range from 300° with a peak at about 420° C. The decrease of PTRM in the interval 300–500° C is evidenced also by the projection of end vector of RM into both xy and xz planes (in the Zijderveld diagram), which have an opposite direction in this interval (Fig. 4). This decrease of PTRM is as a result of generation of the reversed component of PTRM with the value of about −16 833 nT. The decrease of PTRM of a parallel sample was confirmed also at the so called magnetization curve during the Thellier method experiment in Fig. 5. The whole volcanic body of the Brehy locality originated surely in the era of normal orientation of the geomagnetic field. In this nepheline basanite a dominant portion of the samples of the whole body is characterized by normal RM. So, if we find there the samples of basalts with the reversed RM and the behaviour of the PTRM similar to those of the self-reversed PTRM tendencies, they must have been magnetized self-reversally.

I deduce that the Ti-rich Ti-Mt bearing basalts from the world-wide localities have easily acquired the self-reversed RM during their alterations in favour of the titanomaghemites.

The self-reversal was studied also by other authors. *Doubrovine and Tarduno (2004)* reported the results of some oceanic basalts from ODP Site 883/1204. They explained the partial and complete self-reversal due to N-type behaviour. They suggested that the magnetization is carried by the titanomaghemite (Ti-Mgh) Fe-Ti associations. They showed that the acquisition of normal PTRM until 250–275° C, followed by the acquisition of a component antiparallel to the applied field (self-reversal) between 250–350° C. At higher temperatures they again observed the acquisition of a PTRM oriented parallel to the applied field. For the samples which showed a pronounced reversed component of NRM, the acquisition of reversed PTRM produced a true self-reversal of total magnetization. No self-reversal was observed in repeated runs, suggesting that the magnetic mineralogical phase responsible for the acquisition of self-reversed PTRM underwent irreversible alteration at temperature $\leq 400°$ C. Hemoilmenite, if present, would retain its self-reversing properties after such thermal treatment.

Krása et al. (2005) studied self-reversal in samples of basalts from Olby (Quaternary Chaine de Puys, France) and from Vogelsberg locality (Miocene to Pliocene volcanic complex of olivine thoelitic and alkaline olivine basalts, Germany). They explained the self-reversal of RM of basalts by the existence of two magnetic phases with different blocking temperatures (T_B) which are magnetically coupled. The lower T_B corresponds to the primary Ti-Mt (mother phase) crystalizing from the basaltic magma. The remanence with higher T_B is carried by Ti-Mgh or its inversion product (daughter phase) evolving from the primary Ti-Mt by partial low-temperature oxidation. Analytical and numerical calculations of thermoremanence acquisition demonstrate that two-phase particles with the experimentally observed geometry and magnetic properties are able to acquire the self-reversed remanent magnetization due to the magnetic screening plus blocking phenomena.

A partial self-reversal behaviour of the Ti-rich Ti-Mt-es was proven also by *Pan et al. (2006)*, who studied the Thellier-Coe paleointensity of Al-substituted Ti-Mt-es ($Fe_{2.24}Ti_{0.64}Al_{0.12}O_4$) of the Neogene Hannuoba basalts from northern China. The pseudo-single-domain (PSD) grains are the remanence carriers of the Ti-Mt bearing basalts. The laboratory experiments were performed within the argon gas to prevent an oxidation of Fe-Ti oxides. The authors revealed, that between 300 and 460° C, specimens acquire thermoremanence with the antiparallel direction to the external field, leading to intensity decreases. They explained a decrease of PTRM as being due to a partially self-reversed thermoremanence carried by Al-substituted Ti-Mt and newly formed magnetite. They ruled out the maghemitization of Al-Ti-Mt, as a source of decrease of PTRM.

Magnetic mineralogy of the Ti-Mt – Ti-Mgh bearing rocks

The basalts have commonly very heterogeneous magnetic properties. While non oxidized, or only very softly oxidized Ti-rich Ti-Mt bearing basalts reach relatively high values of κ, the basalts with intensely oxidized phase have possessed only very low values of κ. E.g. the basalts from a larger neck Šomoška locality (No. 44, age cca 4.06 my; according to *Orlický, 2004*) reach value of about 62 123 $\times 10^{-6}$ SI units in average, the intensely oxidized basalts from a small remnant of lava flow of Šomoška, No. 45 (cca 50 m on the north-western part from the bottom of basaltic body No. 44; probably of the same age as basalts No. 44 locality) have only 8 282 $\times 10^{-6}$ SI units in average (*Orlický, 2004*). The values of κ are mostly very different also within one volcanic body (in the lava flow of outcrop No. 45 are from about 2 855 to about 22 650 $\times 10^{-6}$ SI units). The composition of Ti-Mt-es and magnetic properties of basalts depend on the degree of low or high temperature oxidation. The degree of oxidation z of studied basalts with the reversed RM (from 13 localities) is in the range from $z = 0.45$ to $z = 0.70$.

X-ray powder diffraction analysis of the oxidized Ti-Mt Ti-Mgh bearing basalt of the Šomoška 3a-1-6 sample was performed by Dr. Dobročka from the Electrotechnical Institute of the Slovak Academy of Sciences Bratislava, Slovakia (*Orlický et al., 2008*). The results of the analysis are: hematite – α-Fe_2O_3 with a = 5.036 Å, ilmenite $FeTiO_3$ (both of rhombohedral structure); maghemite-γ-Fe_2O_3 a = 8.35150 Å, titanomagnetite $Fe_{2.4}Ti_{0.6}O_4$ (both of cubic structure); pseudorutile $Fe_2Ti_3O_9$ a = 2.86670 Å (hexagonal structure).

From the thermomagnetic curve in Fig. 7 we see that this basalt sample contains partly oxidized Ti-Mt phase of $T_C \approx 220 - 230°$ C and the second magnetic phase with the so-called inversion temperature. This starts from about 360 to 370° C and reaches the maximum peak of κ at about 465 to 530° C during continual heating of the sample in air. This inver-

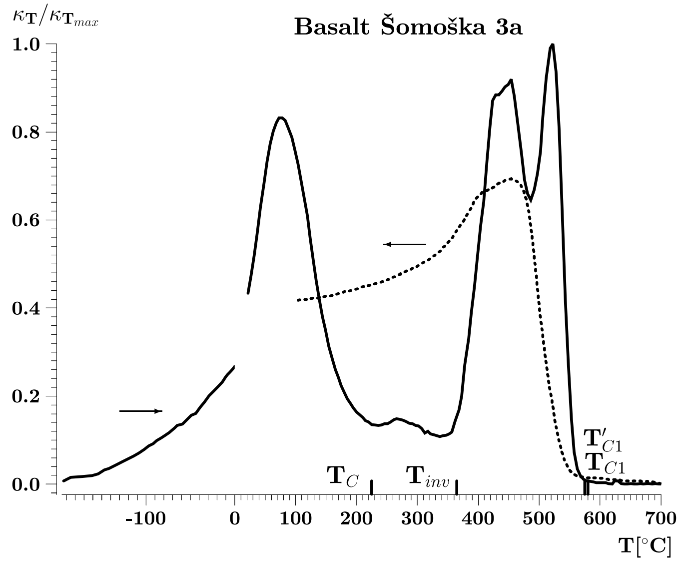


Fig. 7. Thermomagnetic curve of the intensely altered Fe-Ti minerals of the basalt sample Šomoška3A1-6. T_C – Curie temperature of the magnetic phase during heating of the sample, T_{inv} – inversion temperature of magnetic inversion phase – during heating of the sample; T'_{C1} – Curie temperature of the magnetic phase during cooling of the sample; κ_T, $\kappa_{T_{max}}$ – magnetic susceptibility at temperature T and at T_{max}, respectively.

sion phase has usually achieved the Curie temperature at about 580° C. A similar behaviour of κ can be seen for parallel samples in Fig. 2 during step-wise thermal treatment of the sample Šom.3A2-2, also for an inducing of PTRM of the sample Somos3a-1-5. A reflection of the presence of an inversion phase started in both samples with the increase of κ in the interval from cca 360° C. But while the maximum κ is about 530° C for the sample Šom.3A2-2 during thermal treatment, the maximum of κ about 400° C was achieved for the Somos3a-1-5 during inducing of PTRM of the sample. This difference in maximum κ is associated with the degree of oxidation of the samples. As was commented above the temperature when the rock achieved maximum of κ coincided with the minimum of the induced PTRM, which is due to the self-reversed component of the PTRM during thermal treatment.

In Fig. 8 are the results of the Bitter Pattern method (BP) and in Fig. 8a are the results of the magnetic force microscopy (MFM) of oxidized Fe-Ti oxide. The BP and MFM methods were performed by Dr. Funaki in the

Laboratory of the National Institute of Polar Research, Tokyo, Japan. More detailed results of BP were published in *Orlický and Funaki (2008).* The picture of the MFM image has been taken from the conference presentation *Orlický et al. (2008).* In the Somoska3a-1-6 sample, selected grain of cca 40 μm in diameter have shown a maze like the type of domain structure (Fig. 8). The same grain was analyzed also by the MFM method (Fig. 8a). The MFM response depends on the gradient of forces between sample and magnetized tip. The MFM images of domains and domain walls differ from those obtained by the BP techniques. In this technique there is a dependence on the magnetic field gradient (*Krása et al., 2005*). Lamellar domains can be found if the grain is magnetized predominantly parallel to the surface. If the direction of magnetization is oriented perpendicularly to the viewing plane, neighbouring domains appear alternately dark and bright as their magnetization is directed antiparallel and the field leakage is highest above the interior of the domains. In the Somoska3a-1-6 sample (Fig. 8a) the direction of magnetization was about 60° with regard to the polished surface. We see that the domains appear alternately dark and bright (batch-like domains) on the cca 40 μm large grain.

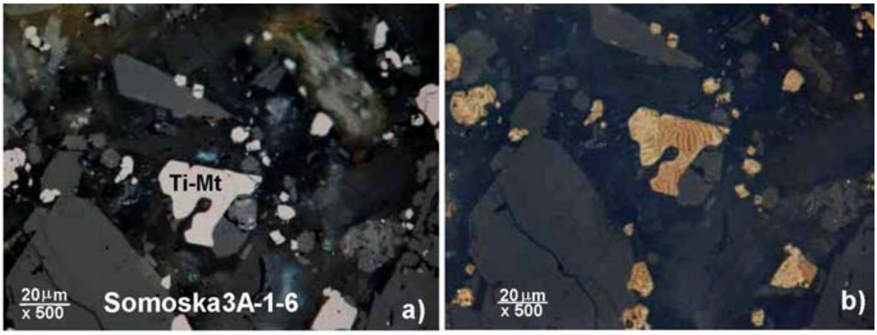


Fig. 8. The ore microscopy image (a) and the Bitter patern – microscopy image (b) of the titanomagnetite (Ti-Mt)-titanomaghemite (Ti-Mgh) grains of the basalt sample Šomoška3A1-6.

Some knowledge about the Ti-Mt-es and their derivatives

I can point out how complicated compositions and magnetic behaviour the titanomagnetites and their oxidized forms possess. A simple preliminary

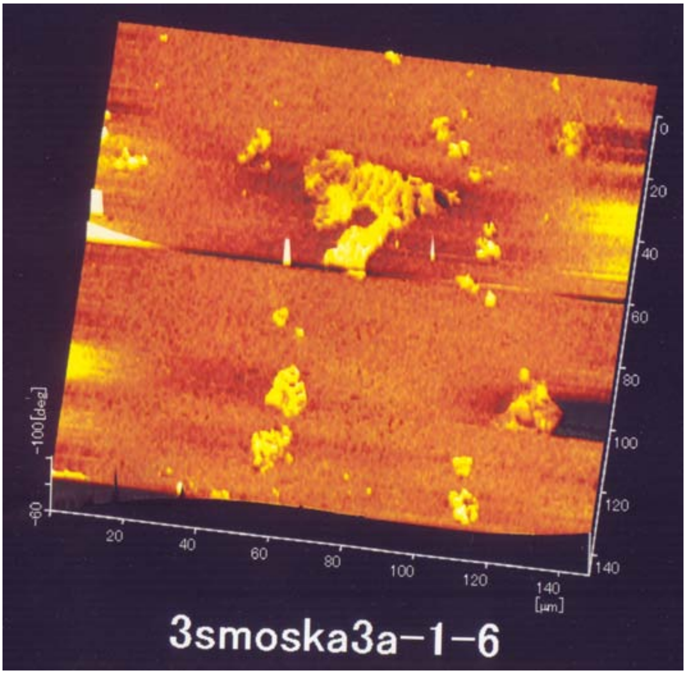


Fig. 8a. The magneto-force-microscopy (MFM) image of the titanomagnetite (Ti-Mt)-titanomaghemite (Ti-Mgh) grains of basalt sample Šomoška3A1-6.

model for the domain state of Ti-rich Ti-Mt-es was suggested by *Appel and Soffel (1984, 1985)* to illustrate an intricacy of the Ti-Mt-es themselves. According to this model, the particles of multidomain (MD) grain size should consist of MD, single domain (SD) and spin cluster regions. The consequence is a decrease of the effective grain size (division into subvolumes) and an enhanced importance of spin rotation process.

From the study of the Ti-Mt-es of basalts from Central Slovakia, Nigeria (*Orlický et al., 1992*) the Mössbauer absorption spectrum yields the hyperfine magnetic fields in the iron nuclei on A- and B- sites. Some characteristic behaviour of the Mössbauer spectra of the Ti-Mt-es can be shown according to *Orlický et al. (2005)*. While the spectra of the Ti-rich Ti-Mt-es are very broad – not Lorentzian and their evaluation showed preferably the octahedral positions for the original Ti-rich Ti-Mt-es at room temperature, the six-line components exhibit a magnetic hyper-fine field about 48

Tesla at liquid nitrogen temperature (LNT). The relative areas of paramagnetic components decrease with decreasing temperature, whereas the area of magnetically split components increase. The tetrahedral positions are characteristic at LNT ($T = -196^\circ$ C) for the same sample.

In *Orlický et al. (1992)* the Curie temperatures of the Ti-Mt-es in air were studied, after evacuated and heated to 620°, and several of them also after being evacuated and heated to 850°, and kept in vacuum at this temperature for about 120 minutes. The results of the measurements were completely different, when compared with those in air. In the samples with Ti-rich Ti-Mt-es the first phase was of about $\mathrm{Fe_{2.28}Ti_{0.72}O_4}$ composition and the secondary – more oxidized phase was about $\mathrm{Fe_{2.62}Ti_{0.38}O_4}$, ($z \approx 0.12 - 0.32$). After the thermal treatment of samples in vacuum, the secondary, low-temperature oxidized phase was significantly suppressed during heating in the interval $25 - 620^\circ$ C and cooling to laboratory temperature. After thermal treatment at 850° C in vacuum and the measurements at laboratory temperature, the low-temperature oxidized phase completely disappeared.

Some samples have a dominant phase with the Curie temperature of $\approx 520 - 570^\circ$ C, and derived composition of $\mathrm{Fe_{2.95}Ti_{0.05}O_4}$, derived oxidation state $z \geq 0.45$. Among the 22 samples of nepheline basanite from Brehy locality (the age about 130-220 ky) three types of Fe-Ti oxides are present. After heating in vacuum and thermal treatment at 850° C this originally one phase was expressively suppressed and new phases with the $T_C \approx 200 - 400^\circ$ C and the second phase of $T_C \approx 580^\circ$ C of very small portion were created. Some samples had a dominant magnetic phase with $T_C \approx 580^\circ$ C and a Verwey temperature $T_V \approx -150^\circ$ C. After thermal treatment in vacuum at 850° C two phases with $T_C \approx 580^\circ$ C and second one with $T_C \approx 500^\circ$ C were created. From the results it is evident that the presence or lack of oxygen in the system is a crucial phenomenon. According to *Pechersky et al. (1981)*, magnetic properties of basalts depend upon the degree of oxidation z in single-phase oxidation. We see from the results that such behaviour is present only for a state in which there was not created the magnetite in the Fe-Ti system, when the oxidation degree z was less than 0.4 according to *Pechersky et al. (1981).*

We can deduce that the ionic bond of the $\mathrm{Fe^{2+}}$ and $\mathrm{Fe^{3+}}$ ions with the ($\mathrm{O^{2-}}$) ions in the oxidized Fe-Ti system is very weak. According to *Kropáček*

(1986) the process of oxidation of the Ti-Mt-es proceeds by means of the diffusion mechanism. A part of Fe and Ti ions diffuse from the B sublattice of Ti-Mt and the Fe^{2+} ions are replaced by vacancies in octahedral (B) positions of the Ti-Mt. Such type of oxidation is present up to 400 to 450° C. The cation-deficient Ti-Mt (Ti-Mgh) is created from the Ti-Mt grain. The creation of small portion of the ilmenite also takes place in parallel way. When there is the magnetite dominantly present in the Fe-Ti system, only magnetite and a high-Curie temperature oxidized Ti-Mt (or Ti-Mgh) were created during thermal treatment in vacuum.

According to *Stacey and Banerjee (1974)* the oxidized Ti-Mt-es are non-stoichiometric compounds. They contain cation-vacancies, due to either the low-temperature ($\leq 600°$ C) oxidation of some of the Fe^{2+} ions in what were originally stoichiometric Ti-Mt-es, or because the cation-deficient compositions were stabilized at moderate-to-high temperature ($\geq 600°$ C) during cooling in the presence of various stabilizing magmatic gases. They are of single-phase cubic crystal structure but metastable like maghemite. On heating in air to temperatures above 200° C, an oxidized Ti-Mt decomposes into a two-phase structure, a Ti-poor Ti-Mt with exsolved Ti-rich titanohematite or ilmenite lamellae. The decomposition is a thermally activated rate process. The electron hoping activates the thermal decomposition of oxidized Ti-Mt-es. The titanomaghemites are strictly oxides in which ($Ti^{4+} + Fe^{2+}$) replaces $2Fe^{3+}$ in maghemite. The general formula for titanomaghemite (Ti-Mgh) is:

$$Fe^{3+}_{8/3-2x}Fe^{2+}_{x}Ti^{4+}_{x}\square_{1/3}O^{2-}_{4}$$

(this is valid only for $y = 1$, i.e. if oxidation is complete).

The oxidation product is a highly non-stoichiometric cation-deficient spinel (titanomaghemite, $Fe_aTi_b\square_3O_4$, a+b+c=3, where $\square$ is a lattice vacancy), according to *Özdemir (1987)*. The metastable Ti-Mgh inverts to a multiphase intergrowth when heated above $250 - 300°$ C. The inversion is an intrinsic property of Ti-Mgh-es, whether heating is in air or in vacuum. The peak of magnetic parameter around $400 - 450°$ C is due to the first stage inversion product. Its height increases with increasing degree of oxidation. On cooling, this product inverts to the final stable product, which should be a spinel phase close to stoichiometric Ti-Mt plus a rhombohedral phase close to ilmenite. After an inversion, an increase in magnetic viscosity and the change of other parameters reflect the subdivision of originally single domain (SD) homogeneous Ti-Mgh grains into SP, or nearly SP-size subgrains. The spinel phase in the intergrowth was near-stoichiometric Ti-Mt or magnetite. The rhombohedral (inversion) phase included ilmenite, hematite, anatase, and pseudobrookite. The composition of the final inversion product depends on the x and z parameters.

The Fe-Ti ferrimagnetic oxides and their characteristic properties

In this paragraph a basic characteristics of the Fe-Ti ferrimagnetics are described. The titanomagnetite (Ti-Mt) solid solutions belong into the ferrimagnetic group of magnetic minerals, with the inverse spinel structure, similar as in the magnetite. The Ti-Mt-es are composed from the two end members – magnetite and the ulvöspinel. The composition of the magnetite is: ($\mathrm{Fe}^{3+}[\mathrm{Fe}^{2+}, \mathrm{Fe}^{3+}]\mathrm{O}_4^{2-}$) and of the ulvöspinel is: ($\mathrm{Fe}^{2+}[\mathrm{Fe}^{2+}, \mathrm{Ti}^{4+}]\mathrm{O}_4^{2-}$).

The outer-shell of unpaired electrons of the sublattices A and B are responsible for the atomic magnetic moments (i.e., $9\mu_B$ per molecule for the B-site iron atoms and $5\mu_B$ per molecule for the A-site iron atoms at low temperature for the magnetite. If the A- and B- site iron atoms were ferromagnetically coupled, spontaneous magnetization I_S at 0° K would correspond to $(9 + 5)\mu_B = 14\mu_B$ (*Stacey and Banerjee, 1974*).

In the Ti-Mt the A-tetrahedral site is coordinated tetrahedrally with four oxygen ions and B-octahedral sub-lattice the each B site ion is coordinated octahedrally with six oxygen ions (the A and B sub-lattices are pictured in Fig. 9, according to S. K. Banerjee in *Lindsley (1991)*. In the following formula the composition of B sub-lattice is in square bracket:

$$\mathrm{Fe}^{3+}_{(2-2x)a}\ \mathrm{Fe}^{2+}_{1-(2-2x)a}\ [\mathrm{Fe}^{3+}_{(2-2x)(1-a)}\ \mathrm{Fe}^{2+}_{(x-(2-2x)a}\mathrm{Ti}^{4+}_x]\mathrm{O}_4^{2-}.$$

The Ti^{4+} ions are only in B positions. The magnetic moment influenced by the interactions and after replacement of ions Fe^{2+} and Fe^{3+} in A and B positions is: $\mu = (1 - x)(6 - 4a)\mu_B$. A distribution of cations of Ti-Mt and its magnetic moment are dependent only on x (compositional parameter) and parameter a.

At room temperature I_S of the magnetite is 90 e.m.u./g. Its saturation magnetization (M_S) is 480×10^3 A/m (*McElhinny and McFadden, 2000*). The magnetite is a ferrimagnetic mineral. Ulvöspinel ($\mathrm{Fe}^{2+}[\mathrm{Fe}^{2+}\ \mathrm{Ti}^{4+}]\mathrm{O}_4^{2-}$) is also the inverse spinel. The outer-shell unpaired electrons of Fe^{2+} ions in

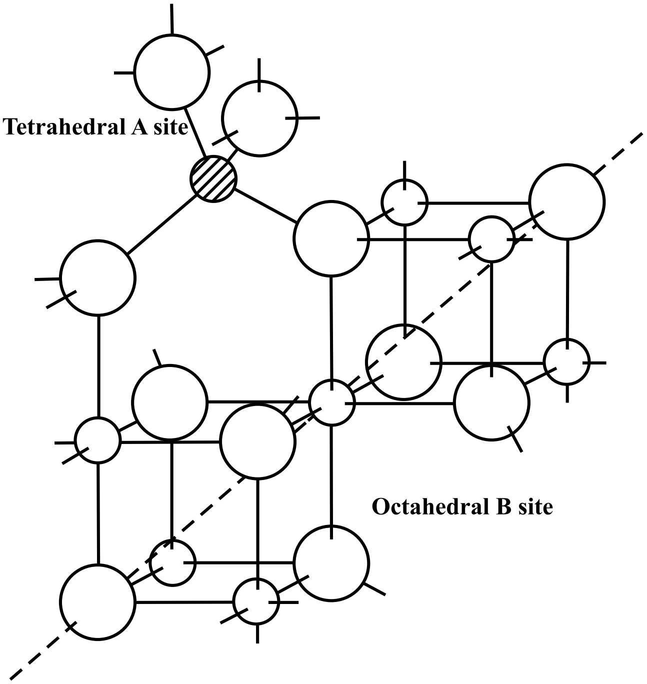


Fig. 9. A part of spinel structure to display octahedral B site iron ions (small open circles) and tetrahedral A site iron ions (small hatched circles). The larger open circles represent oxygen anions. The dashed line is the body diagonal [111] axis, the easy axis of magnetization in magnetite (with kind permission, according to *O'Reilly (1984)*.

the sublattices A and B are responsible for the atomic magnetic moments (i.e., $4\,\mu_B$ per molecule for the B-site iron atoms and $4\,\mu_B$ per molecule for the A-site iron atoms at low temperature). In the titanomagnetites, the substitution of Ti ions for Fe ions in the magnetite lattice, with conservation of total ionic charge, means that (Ti^{4+} and Fe^{2+}) replace $2Fe^{3+}$, i.e., substitution of Ti^{4+} for Fe^{3+} requires also the conversion of second Fe^{3+} ion to Fe^{2+}. The Fe^{2+} ($3d^6$ ions) have a magnetic moments of $4\,\mu_B$ for both A and B sublattices, and since the atoms are coupled antiferromagnetically, the net magnetic moment I_S should be $4 - 4 = 0\,\mu_B$. The Ti^{4+} ion has a zero magnetic moment. *Readman et al. (1967)* studied I_S and magnetic susceptibility (κ versus T, as well as Mössbauer absorption in Fe_2TiO_4). They concluded that the orbital angular momentum in the B-site Fe^{2+} ion is only partially quenched while that in the A-site Fe^{2+} ion is fully quenched. The result is that while the A-site Fe^{2+} ion in ulvöspinel has a magnetic moment of $4\,\mu_B$, the magnetic moment in B-site Fe^{2+} ion due to spin and some orbital momentum is $4.5\,\mu_B$. In very strong fields and close to 0° K the value of I_S for ulvöspinel is $0.5\,\mu_B$ per molecule (*Stacey and Banerjee, 1974*). New investigations have revealed that the sub-lattices are not completely equivalent and so the net magnetic moment of the ulvöspinel is $0.02\,\mu_B$ (S. K. Banerjee in *Lindsley et al., 1991*).

In natural basalts, the Ti-Mt solid solutions, are frequently of the composition ($Fe_{2.4}Ti_{0.6}O_4$; Ti-Mt 60). When we compare the values of M_S of magnetite ($M_S = 480 \times 10^3$ A/m) with that of Ti-Mt 60 in *McElhinny and McFadden (2000)* we see that there are inconsistencies in the level of volume magnetic susceptibilities in volcanics (namely in basalts). While the Ti-rich Ti-Mt bearing basalts have the κ in the range of $50 - 80 \times 10^3$ SI units, the magnetite bearing volcanics (basalts or basaltic andesites) reach the values of about $20 - 30 \times 10^3$ SI units which is only half of the values of Ti-rich Ti-Mt-es. How to explain it? In *Orlický (2006, 2006a,b,c)* there are the curves of the temperature dependence of magnetic susceptibility of the Ti-rich Ti-Mt bearing basalts. In selected curves there is rapid increase of κ from $-196°$ C to higher temperatures up to the maximum. Closely below the Curie point the κ has abruptly fallen down. In the low magnetic fields the permeability (μ), also the (κ) ($\mu = 1 + 4\pi.\kappa$) of the mixed ferrites (the solid solutions of the inverse ferrites with the Zn^{2+} ferrites) increases with increasing of temperature and it reaches the extreme maximum at the temperature closely to that of the Curie temperature in *Brož (1962)*. Such behaviour have shown also the Ti-rich Ti-Mt-es. This effect is a consequence of a rapid decrease of the magnetic crystallographic anisotropy with temperature. With the increasing of Zn^{2+} ions in the ferrite decreases the intensity of the exchange interactions, what results in a decrease of the Curie temperature of the ferrite. Also the Curie temperature of the Ti-rich Ti-Mt-es decrease with an increase a portion of Ti^{4+} ions (the ions of Zn^{2+} and Ti^{4+} ions have no magnetic moments). So, an increase of the non-magnetic Ti^{4+} ions in the titanomagnetite creates the conditions for enhanced magnetic susceptibility. Previously I accepted an idea about the super paramagnetic-like behaviour of the Ti-rich Ti-Mt-es. It has not been adequate and it must be definitely abandoned.

The basic condition for the existence of the ferrimagnetic behaviour of the respective mineral is a presence of ions with non-zero magnetic moments in both mineral sublattices. In this system the magnetic configuration contains two ferromagnetic sub-lattices bonded mutually antiparallelly, one to each other. If the magnetic moments of both sub-lattices are equal to each other and the sum of the magnetic moments is zero – the magnetic structures are as those of *antiferromagnetics.* The ordered structures with the resultant moment $M = M_B - M_A$ different from zero are as *ferrimagnetics.* For the ferrimagnetics there is a characteristic spontaneous magnetization. In the ferrimagnetics the magnetization is a vector sum (M_S) of magnetizations (M_1, M_2) of the two antiparallel sub-lattices: $M_S = [M_1 - M_2]$. The thermal dependence of M_S must be of course expressed separately for M_1 and M_2 of individual sub-lattices, because M_1 and M_2 can have very different temperature dependences themselves. According to *Néel (1948)* model, M_B – the sub-lattice magnetization (I_{SB}) then has a stronger temperature dependence than the M_A sub-latice (I_{SA}). This can lead to a reversal of spontaneous magnetization at a *compensation point* (θ). This is consistent with a model in which the strongest superexchange coupling is the negative one (i.e., antiparallel) A-B interaction and the smaller B-B interaction between B-site iron atoms is also negative one. The negative B-B interaction hastens thermal disorder (*Stacey and Banerjee, 1974*). From the three different types of the exchange interactions the AB interaction is the strongest one, and its exchange integral is always the negative one. It always makes the antiparallel orientation of magnetic moments of individual sublattices. The AB interaction is an example of the indirect exchange. With regard to large distance among neighbouring metallic ions, there is a very weak direct exchange effect. The indirect exchange is interfaced by the negative oxygen's ions. They create some *bridges*, and they make possible, the electrons, which make the non-zero magnetic moments of the metallic ions of the A sublattice to approach the respective electrons of the metallic ions of the B sublattice and so integrating with them. Because A and B sublattices are nor magnetically, nor crystallographically equivalent (they contain different numbers of ions and different quality types of non-zero ions) the resulting spontaneous magnetic moment of these sublattices is different and equal to a difference of magnetic moments of both individual sublattices.

An explanation of κ and the reversed PTRM behaviour, the ionic reordering in the Ti-Mt – Ti-Mgh systems

The reversed RM (probably of CRM – chemical origin) of the rocks is carried by a secondary, low-temperature oxidized magnetic phase, which originated during oxidation of some portion of Ti-rich Ti-Mt-es, either during initial cooling of basalt magma in the field, during an additional heating of the volcanic body, or during existence of basaltic bodies on the Earth's surface in the post-volcanic time. The Fe-Ti phases have positioned themselves into a certain compositional and magnetic stage in which the self-reversed RM was acquired in a positively oriented geomagnetic field. In the laboratory experiments the further portion of the Ti-Mt-es and Ti-Mgh-es (if any) was transformed and the reversed PTRM is acquired at the temperatures 300 to 475° C and an increase of κ with a maximum peak of about 450-475° C is detected. What role for the increase of κ do the spontaneous magnetic moments of both A and B sub-lattices of the Ti-Mt Ti-Mgh ferrimagnetics play? I assume that the changes in the rearrangements of Fe^{3+}, Fe^{2+} and Ti^{4+} ions and so a change of spontaneous magnetic moments is reflected in the change of κ during heating of the Ti-Mgh sample. The resultant spontaneous magnetization of the ferrimagnetics is a difference of both A and B sublattices. So, we are able to explain an increase of magnetic susceptibility of basalts in the interval cca 300 – 500° C and the decrease of PTRM (reversed orientation of PTRM) on the basis of the following model: I can deduce that there is only the Ti-rich Ti-Mt in the magma at the beginning of its emplacement on the Earth surface. From the Akimoto and Néel-Chevalier models it follows that the Fe^{2+} and Ti^{4+} ions are dominantly in the B - octahedral position for $x \geq 0.5$, and the Fe^{3+} ions are in the tetrahedral – A position for $x \leq 0.5$. The evaluation of the Mössbauer spectra showed preferably the octahedral positions for the original Ti-rich Ti-Mt-es at room temperature. The carrier of magnetism is the Ti-rich Ti-Mt in original state of basalt. After an alteration of original Fe-Ti oxides the process of oxidation of the Ti-Mt-es proceeds and the cation-deficient Ti-Mt (Ti-Mgh) is created from the Ti-Mt grain. This process acts by means of the diffusion mechanism. A part of Fe and Ti ions diffuse from the B sublattice of Ti-Mt and the Fe^{2+} ions are replaced by vacancies in octahedral B sublattice of the Ti-Mt. Such type of oxidation is present up to 300 to 450° C. There is a gradual decrease of Fe^{2+} ions and increase of non-magnetic vacancies in B sub-lattice. The original *in situ* relations of cations of the Ti-Mgh, and thus the magnetic moments in the inter-knots of the A and B sub-lattices are reordered in such a way that in the B octahedral positions a number of normally oriented magnetic moments has decreased and in the A tetrahedral positions a number of oppositely (reversed with regard to the external field) oriented magnetic moments has increased. The total magnetic moment M_B of the B sub-lattice is less intense than that of M_A of the A sublattice (in absolute value). (According to *Schult (1968)* if one assumes that the vacancies are all on the B-site the magnetization of the B-sublattice becomes smaller with oxidation and it is possible that for a certain composition the spontaneous magnetization of B-sublattice equals that of the A-sublattice or becomes even less). In the ferrimagnetics the magnetization is a vector sum (M_S) of magnetizations (M_B, M_A) of the two antiparallel sub-lattices: $M_S = [M_B - M_A]$. The thermal dependence of M_S must be of course expressed separately for M_B and M_A of individual sub-lattices, because M_B and M_A can have very different temperature dependencies themselves. According to *Néel (1948)* model, M_B – the sub-lattice magnetization (I_{SB}) then has a stronger temperature dependence than the M_A sub-latice (I_{SA}). This can lead to a reversal of spontaneous magnetization at a *compensation point*. We take into account that all the alterations of magnetic minerals of rocks in nature and magnetizing processes in the laboratory took part under the influence of external, the geomagnetic field. According to *Vonsovskij (1971)* each magnetic ion in ferrimagnetic mineral is under the influence of an effective magnetic field H_{ef} during a magnetizing process. The effective field H_{ef} is the sum of the external field H_{ext} and molecular fields of respectively H_B and H_A sub-lattices. The acquired reversed PTRM of rocks has shown that if the self-reversed mechanism is a reflection of the ordering of magnetic moments of A and B sub-lattices the resultant effective magnetic field during magnetizing of rocks must be opposite (reversed) with respect to the normally oriented geomagnetic field.

As has been noted above, the resultant spontaneous magnetization is the difference between the magnetizations of B and A sublattices. If we accept an idea that the changes of spontaneous magnetization in both B an A sub-lattices have contributed to the changes of magnetic susceptibility, then the increase of κ in the interval from 300 to about 450° C (or somewhat more) can explain an increase of the oppositely oriented spontaneous magnetization in the A sublattice, according to formula $M_S = [M_B - M_A]$.

There have existed the results of other authors about the ionic reordering and an origin of the self-reversal RM in Ti-Mt-Ti-Mgh-es. *Verhoogen (1956)* proposed that the ionic reordering in Ti-Mgh could result in self-reversal of magnetization. During oxidation, a Ti-Mt with a dominant B sublattice magnetic moment can be transformed to a Ti-Mgh with a dominant A sublattice moment, thus producing a self-reversal of spontaneous magnetization (M_S). Remanent magnetization, if controlled by contribution from single-domain grains, will also self-reverse. *Verhogen (1962)* pointed out that some oxidized Ti-Mt-es were likely to have a net spontaneous magnetization due to the tetrahedral sub-lattice moment, M_A exceeding the octahedral sub-lattice moment M_B. Thus a stoichiometric Ti-Mt, in which the B site ions dominate the net magnetization, could, after a suitable degree of oxidation, acquire a dominant A-site magnetization, a reversal in direction of the net moment ($M_B - M_A$). It is probable that oxidation results first in a decrease in I_S, followed by an increase as exsollution occurs. The low-temperature oxidation can result in a CRM remanence in basalts. Since the oxidation also causes a decrease in the number of magnetically anisotropic Fe^{2+} ions in the lattice, coercive force, H_C is expected to be lowered, and hence, the new CRM is expected to be relatively unstable.

Schult (1971) pointed out that when in Ti-Mt-es a cation deficient spinel phase is produced by oxidation, the ratio of the sublattice magnetization at 0° K M_A/M_B should increase with increasing oxidation degree. The Ti-Mgh-es which undergo self-reversal by ionic reordering should exhibit N-type with a compensation point above room temperature or Q'-type M_S (T) curves. *Doubrovine and Tarduno (2004)* suggested that the single domain and the pseudo-single domain grains of Ti-Mgh with N-type thermomagnetic behaviour and compensation points above room temperature are responsible for the self-reversed PTRM.

Krása et al. (2005) deduced that the remanence direction might reverse due to modifications of the occupancy of the magnetic sub-lattices in Ti-Mt-es/Ti-Mgh-es in the course of low temperature oxidation. In non oxidized Ti-Mt-es the B or octahedral sub-lattice has a higher spontaneous magnetization than the A or tetrahedral sub-lattice at room temperature. If, during low-temperature oxidation, cation vacancies are only formed on B sites, the inverse of the A sub-lattice might eventually get stronger than the

B sub-lattice magnetization. In such a case an *irreversible* self-reversal not reproducible by imparting a laboratory TRM would be observed.

The *Néel (1948)* model has a specific applicability in a computation of the magnetization curves of the ferrospinels (or ferrispinels). According to *Goodenough (1963)* the cations in the tetrahedral positions of the ferrospinel $\mathrm{Zn}^{2+}_{x}\mathrm{Fe}^{3+}_{1-x}[\mathrm{Me}^{2+}_{1-x}\mathrm{Fe}^{3+}_{1+x}]\mathrm{O}_4$ create one of the two collinear sub-lattices with antiparallel spinels and in octahedral positions create the second sub-lattice. If $x = 0$, magnetic moments of Fe are mutually compensated and $M_0 = N' g_M\, \mu_B\, S_M$ (N' = number of molecules in a volume unit). The presented magnetization curves separately for Mn, Fe, Ni, Cu, Mg and $\mathrm{Co}(\mathrm{Li}^{+}_{0.5}\mathrm{Fe}^{3+}_{0.5})$, in which the magnetic ion Me^{2+} was substituted by the non-magnetic Zn^{2+} ion in the ferrospinel $\mathrm{Me}^{2+}_{1-x}\mathrm{Zn}_x\mathrm{Fe}_2\mathrm{O}_4$, increased up to about $x = 0.4$ and then decreased.

3. Discussion and conclusions

The paleomagnetic investigation and a study of the remanent magnetization of rocks are strictly linked with the geomagnetic field and its dynamics. A primary question in the dynamics of the geomagnetic field is the change of its polarity during a geological past. An idea about the reversals of the geomagnetic field has been derived only on the basis of the normal and reversed polarities of RM of rocks. No other experimental magnetic evidences were in a disposal for the construction of the paleomagnetic scales and an explanation of the reversed RM on the basis of the so-called field-reversal hypothesis. The one-sided view that the source of the reversed RM of rocks has been only the reversed direction of the geomagnetic field may be misleading. Taking into account my results of the reproducible inducing of the self-reversed PTRM of the intermediate volcanics (see the former cited articles), an acquiring of the self-reversed PTRM of natural basalts and the artificially prepared basaltic rock, dominantly containing the oxidized Ti-Mt-es, the basaltic samples containing the hematite-ilmenites (Hem-Ilm-es), the results of other authors about the self-reversed RM of rocks, and some theoretical aspects, there have might existed several mechanisms of generation of self-reversal reversed RM in the rocks. The above presented results can serve as a basic evidence to an idea of the self-reversal origin of the reversed RM of rocks, and to contribute to my general idea that no reversals of the geomagnetic field have so far occurred.

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