An exceptional role of small magnetic particles in magnetism and paleomagnetism of rocks

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A b s t r a c t : Here we present the results of the investigation of basalts of the Pleistocene to Quaternary age from southern Slovakia, Miocene to Pliocene age basalts from České Středohoří Mts., North Bohemia, Cretaceous age basalts from the Syrian Arab Republic and the Cretaceous age basalts from Nigeria. The selected basalts have the reversed or at least anomalous, partly reversed RM. These types of basalts contain either dominant, or at least small part of Ti-rich titanomagnetites (Ti-Mt-es) and a more oxidized magnetic phase, probably titanomaghemite (Ti-Mgh). The reversed RM was acquired during cooling of magma from about 400 to 500° C (mostly of 450° C) to atmospheric temperature in the nature. The laboratory experiments have proven that κ of these basalts reached the maximum value in the interval 400 to 500° C, mostly at 450° C. Such behaviour of κ coincides with the results of continual measurements of the change of κ of the Ti-Mt-es with temperature. During inducing partial thermoremanent magnetization (PTRM) in the samples, the minimum value of the PTRM has been reached at the temperatures corresponding to maximum values of κ. This behaviour supports the idea that the above mentioned maximum κ corresponds to a presence of superparamagnetic (SP), small magnetic particles in the respective basalts. This is a very important phenomenon. Rather similar behaviour of κ was registered in basaltic andesites and other intermediate andesites containing residual portions of the T-rich Ti-Mt-es, from volcanic fields of central and eastern Slovakia. I deduce, that the above described behaviour of Ti-rich Ti-Mt-es with a secondary Ti-Mgh, and an acquisition of the self-reversed PTRM by the small magnetic particles in the rocks, is a general world-wide phenomenon. All results have proven very strong tendency in favour of the self-reversal PTRM of the rocks with small magnetic particles. The basalts of only 3 localities with normal RM from southern Slovakia were studied to compare their properties with those of the reversed RM. Normal RM is linked either with the non-stoichiometric magnetite or with the more oxidized Ti-Mt phase in these type of basalts. It has been shown, that the rocks containing the magnetic minerals with the thermodynamically stable domain structures are only of normal RM, but the rocks, containing the small magnetic particles, as the carriers of magnetism, may acquire predominantly the reversed RM during magnetization, depending on the size and the shape of the particles.

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Key words: Basaltic rocks, small Fe-Ti magnetic particles, the carriers of the self-reversal RM.

1. Introduction

The study of magnetic properties of rocks has reached a wide application in paleomagnetism, geology and petrophysics. But the understanding of the basic magnetic properties, the magnetic state, the behaviour of magnetic minerals of rocks, namely, their exceptional position in creation of the so called self-reversal RM, has lag behind all other similar branches. In many applications in the physics of magnetic materials is desirable to achieve high values of the remanent magnetization (RM). The RM depends on the material parameters, as well as on the size and shape of magnetic particle. So, frequently small magnetic particles are used to achieve this goal. In magnetism and paleomagnetism we are concerned with the RM of magnetic minerals in volcanic, sedimentary an metamorphic rocks. During the creation and alteration of rocks, the different types and grain sizes, including SP particles of Fe-Ti, or Fe magnetic minerals originate. So, like in magnetism of synthetic materials, also in paleomagnetism of rocks, the small magnetic particles play a very important role. The so called superparamagnetic limit of the particles is frequently respected, because this term describes the critical size, below which small magnetic particles may continually reverse their magnetization direction due to thermal agitation (*Bode et al., 2004*). Néel and Brown in (*Bode, 2004*) calculated the switching probability under the assumption of coherent rotation, i.e., at any time - even during the reversal, the magnetic moments of the entire particle remain magnetized in the same direction, behaving like a single giant spin (superparamagnet). The magnetization in a uniformly magnetized sample is usually stabilized by an easy-axis anisotropy of crystalline or demagnetizing origin. For microscopic single-domain particles, the coercivity can reach high values, since the state of reversed magnetization has first to be nucleated. Such particles exhibit an extremely high long-term stability of the magnetization (*Braun, 1994-II*). This fact renders them suitable for information storage in recording media, and as constituents of rocks they preserve the value of the local magnetic field as the temperature has drops below the blocking temperature of the particle. According to *Braun (1994-II)*, with decreasing of particle size the effect of thermal fluctuations becomes increasingly important. For particle size of a few nanometers and at room temperature the magnetization fluctuates randomly over the anisotropy barrier, and a SP state results with vanishing average magnetic moment. The Néel-Brown model in *(Wernsdorfer et al., 1997)* is widely used in magnetism, in order to describe the time dependence of magnetization of collections of particles, thin films, and bulk materials. With regard to solution of such problems, the magnetization reversal phenomenon is very important. The mentioned authors propose that a good understanding of the problem of magnetization reversal in the complex systems requires an understanding of magnetization reversal processes of single magnetic particle. Most of the single-particle properties were hidden behind some distribution functions of particle size, shape, etc.

Only several sentences mentioned above have outlined that a behaviour of small magnetic particles is very seriously considered in physics of synthetic magnets. In paleomagnetic study *(Orlický, 2006)* brought evidence that the reversed RM of basalts with Ti-rich titanoamgnetites is linked with a presence of SP-like Fe-Ti magnetic minerals. Moreover, he has revealed that the Ti-rich titanomagnetites behave as the SP-like materials *(Orlický, 2004)*. So, they have properties corresponding to small magnetic particles. These particles have not possessed a classical, thermodynamically stable domain structures. The idea about a behaviour of small magnetic particles of Landau and Lifshitz in (*Hubert and Schäfer, 1998*) has been accepted, to identify small magnetic particles. Thermal agitation plays a role only in small particles or at temperatures close to the Curie point. Under normal circumstances the equilibrium magnetic microstructure - minerals with a well developed thermodynamically stable domain structure must be considered athermal. It means that the measurements of the change of magnetic parameter (e.g. magnetic susceptibility – κ) with temperature may identify a presence of small magnetic particles. Continual measurements of the change of κ of magnetic fraction with temperature (Curie temperature measurements) with the apparatus and by the methodical procedures suggested by *Orlický (1990)*, and discrete measurements of the change of κ of compact rock samples during their thermal magnetization were done for many petrographical types of rocks, from many localities. The particular results concerning the basaltic rocks from Southern Slovakia, Bohemian Massif, the Syrian Arab Republic and Nigeria are presented in this article. Because the basic physical principles of a behaviour of synthetic and natural magnetic minerals have a common theoretical basis, we will accept some results from the physics of small synthetic magnetic materials to explain a behaviour of natural magnetic minerals in the rocks.

2. Methodical setting and evaluation of experimental data

The experiments concern the basaltic rocks with the Ti-rich Ti-Mt-es and the basalts with a partly altered Ti-Mt-es, which have been characterized dominantly by the reversed RM or by an anomalous direction of the RM (the basalts come from 11 places of the Podrečany and Cerová basalt formations, located in the geographical coordinates $\varphi = 48.399$ to $48.170°$ and $\lambda = 19.608$ to $19.992°$, and from the locality Devičie, the geographical coordinates $\varphi_L = 48.306°$, $\lambda_L = 19.103°$). Moreover, the basalts from abroad – from the Bohemian Massif, the Syrian Arab Republic and from Nigeria were investigated also. The delineation of the respective localities is done by the geographical coordinates below the Figs. 5a-7a. The basalts with more oxidized Fe-Ti oxides of normal RM from the localities Blhovce-Buda, Šomoška, Ostrá skala were investigated, as well, to compare the magnetic behaviour of these basalts with the basalts of the reversed RM. For all samples the change of magnetic susceptibility (κ) with temperature and Curie temperature measurements were realized.

Thermal magnetization and Curie temperature measurements. We take into account that during the original volcanic activity the process of cooling of basaltic magma proceeded from higher to lower temperatures in the field (from about 1200° to atmospheric temperatures). In the laboratory, the magnetization of the samples was realized in the opposite direction, from lower to higher temperature intervals. The magnetization of magnetic minerals of cooling magma takes place in the permanent geomagnetic field of the intensity H $\approx 50\mu$ T. The laboratory magnetization of samples in the geomagnetic field of intensity H $\approx 48\mu$ T in different temperature intervals has a peculiar course. Evidently, the magnetization is not of the type of a simple, like classical domain type magnetization, but it has the properties of some special flower and vortex type, corresponding to magnetization of small, including the SP-like type of magnetic particles.

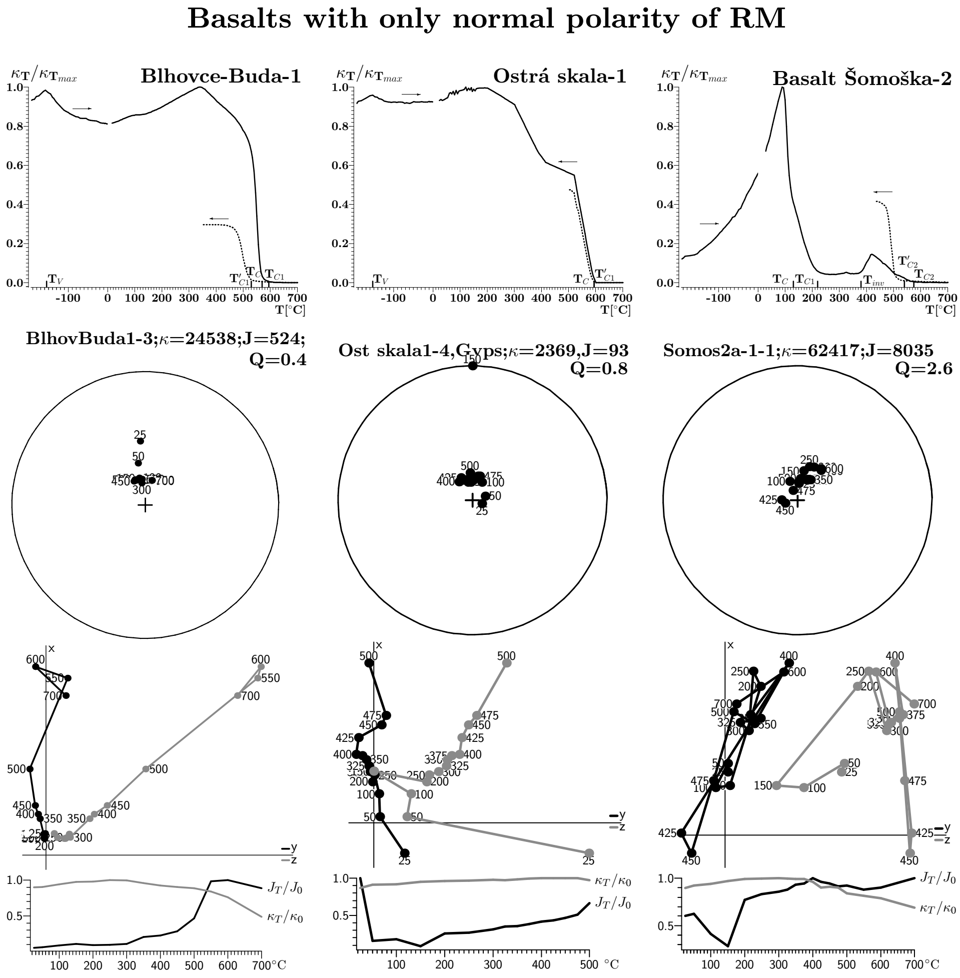


Fig. 1. Thermomagnetic curves of samples from the localities Blhovce Buda (age = 1.73 Ma; $\varphi_L = 48.266°$, $\lambda_L = 19.957°$), Ostrá skala (age = 2.6 Ma; $\varphi_L = 48.246°$, $\lambda_L = 19.943°$), Šomoška (4.06 Ma; $\varphi_L = 48.170°$, $\lambda_L = 19.842°$); Thermal magnetization (PTRM) of samples (stepwise heating to 700° C); Zijderveld diagrams and stereographic projections. T_V - Verwey transition temperature. Other explanations see in Fig. 2.

Most of samples of a cubic or a cylindrical shape were demagnetized by AF field of about 400 mT before the magnetization procedure. After this procedure they were held in the container of high magnetic permeability, to protect them against any influence of the external magnetic field.

An inducing of the partial thermoremanent magnetization (PTRM) in the samples by the magnetic field of the positive polarity and the intensity of H $\approx 48\mu$ T was realized by the following procedure: The samples were placed on the holder of the non-magnetic furnace, with the orientation of the sample in the positive direction of H - component of the field. The speed of heating of the samples was about 3°/min. During about 20 min., the magnetization was realized at the concrete temperature ($T_{concr.}$), and then the samples were cooled to laboratory temperature (approximately at the same speed as was during heating of the samples). The following temperature steps were applied: 50, 100, 150, 200, 300, 350, 400, 450, 500, 600, 700° C. The reason for the magnetization of the samples from the lower to higher temperatures is the following: In the nature, the hot magma cools from high temperatures to atmospheric temperature (the temperature of a basaltic magma reaches about 1200° C, in some cases more, before it cooled on the Earth's surface). The conditions, similar or the same as those, which were valid in the field, cannot be simulated in the laboratory. We have known that the dominant carriers of magnetism in basaltic rocks are the Fe-Ti oxides, mostly Ti-Mt-es. These minerals have very high affinity to oxygen, and so they are very sensitive to oxidation in a presence of oxygen, namely at high temperatures. It means that if we apply a procedure of their cooling from high temperatures to room temperature during laboratory experiment, the original composition of Ti-Mt-es would be either completely or partly destroyed and transformed into the non-precisely defined oxidized Fe-Ti oxide, mostly of high-oxidation Fe-Ti product, and the laboratory PTRM of the sample would be completely overprinted by the phase, corresponding to this product. This unfavorable effect can be partly suppressed by heating and cooling the samples either in high vacuum, or to proceed from lower, laboratory temperature, gradually to higher temperatures (by this way a rule of additivity of magnetizing of the samples is fully respected).

The RM of the samples was measured by the spinner magnetometer JR-5 at room temperature after each laboratory temperature treatment. In parallel way, the change of κ was measured by the susceptibility meter KLY-2, after each laboratory treatment of the samples (see Figs. 1-4, 5a, 6a, 7a; In Figs. 5, 6, 7 the results are presented of measurements of κ change with temperature, the results of microprobe analyses and the microphotographs of basalt samples from Bohemian Massif, the Syrian Arab Republic and Nigeria, respectively).

Evaluation of the results of basalts with only normal polarity of RM. We see from the thermomagnetic curves in Fig. 1, that the dominant magnetic phase in the samples Blhovce Buda-1 and Ostrá skala-1 have the T_C of about 580° C and the Verwey temperature (T_V) about −155° C. They correspond to magnetite. There are no variations of κ during temperature treatment of the sample Blhov Buda1-3, only over 520° C a decrease of κ due to alteration of original Fe-Ti mineral is evident. In this sample gradual increasing of PTRM up to about 450° C is evident, from RM = 524 nT and then an abrupt increase to PTRM = 9420 nT at 600° C. The sample Ost skala1-4 has been prepared artificially (for the thermal magnetization), as the softly grinded grains of basalt fixed in non-magnetic gypsum. No extraordinary variation of κ is seen during thermal treatment. In this sample an abrupt decrease of PTRM from 98 nT, near to zero at 150° C is evident and then a gradual increase of PTRM up to 60 nT at 500° C. No typical increase of κ and decrease of PTRM at about 450° C for both mentioned samples (like for basalts with the reversed RM) has been detected.

The samples from large basaltic neck of Šomoška locality come from two places. They have normal RM, but the directions of RM are little bit different. There are three magnetic phases in these basalts (see the thermomagnetic curve of the sample Basalt Šomoška-2). The first one with $T_C \approx 120°$ C, corresponds to primary Ti-rich Ti-Mt. This phase is a source of high values of bulk κ (Fig. 1, 62417×10^{-6} SI Units) of basalts and is of SP magnetic state. The second phase is of $T_C \approx 230°$ C (250° C). The 3rd phase with $T_C \approx 560°$ C (570° C), corresponds to Ti-Mgh-emite, with high portion of non-stoichiometric magnetite. Both, the 2nd and 3rd phases originated due to low and high temperature oxidation of the primary phase. All three mentioned phases of the basalt neck's body are not of the same portion and magnetic arrangement. So, the direction of RM of all samples has shown some deviations. In the sample Šomoška2-3 the RM decreases only very softly up to 560° C, NRM and RM after thermal demagnetization are of

Basalts containing the Ti-rich Ti-Mt-es of the reversed RM

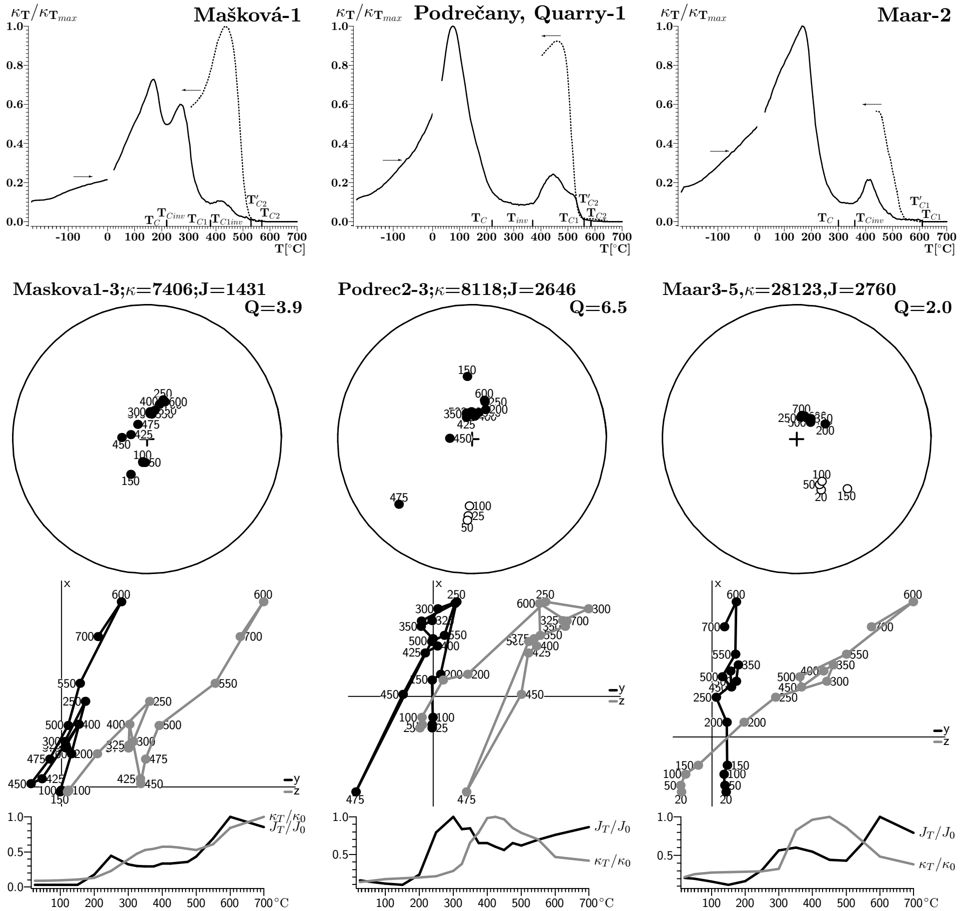


Fig. 2. Thermomagnetic curves of samples from localities Mašková (age = 7.17 Ma; $\varphi_L = 48.320°$, $\lambda_L = 19.576°$), Podrečany (age = 6.44 Ma; $\varphi_L = 48.390°$, $\lambda_L = 19.625°$), Maar ($\varphi_L = 48.249°$, $\lambda_L = 19.954°$; the results of microprobe analyses: $TiO_2 = 18.538 - 19.042\%$, FeO=68.381-69.913%), $T_{C,C1,}$ - Curie temperatures) and thermal magnetization (PTRM) of samples (stepwise heating to 700° C); Zijderveld diagrams and stereographic projections; • (∘)-positive (negative) polarity of RM; κ- magnetic susceptibility: κ_T at T, κ_0- at 25° C; values of κ in pictures are x 10^6 in SI units; J_T (J_0)- remanent magnetization in nanoTesla (nT) at T and at 25° C.

positive direction and are relatively stable. Many samples from this locality have shown intense values of NRM. Very important phenomenon is, that the thermodynamically stable domain structure has developed in the dominant part of the Fe-Ti high-thermally oxidized phase. Owing to its high stability, only normal RMP in the basaltic body was induced by geomagnetic field

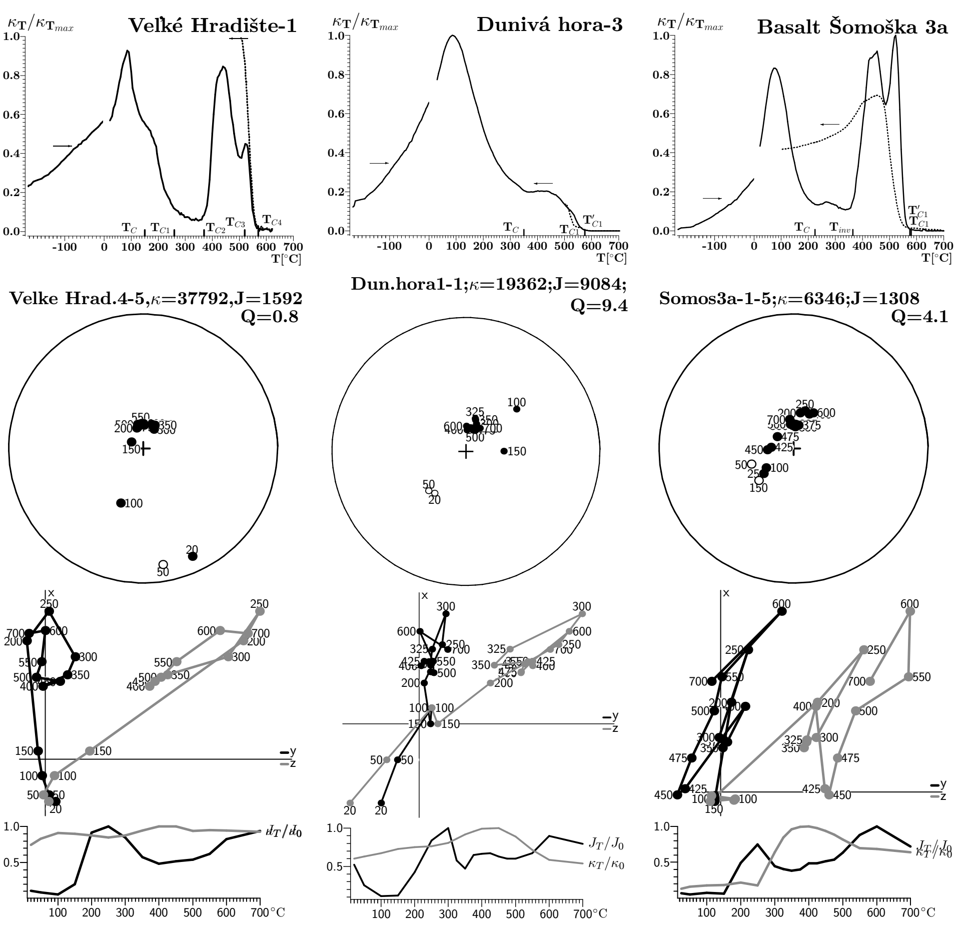


Fig. 3. Thermomagnetic curves of basalts Veľké Hradište (age = 5.43 Ma; $\varphi_L = 48.268°$, $\lambda_L = 19.742°$), Dunivá hora (age = 1.3 Ma; $\varphi_L = 48.176°$, $\lambda_L = 19.871°$), Šomoška3a (age = 4.06 Ma; $\varphi_L = 48.170°$, $\lambda_L = 19.842°$) $T_{C,C1}$, - Curie temperatures; Thermal magnetization (PTRM) of samples (stepwise heating to 700° C); Zijderveld diagrams and stereographic projections. Other explanations see in Fig. 2.

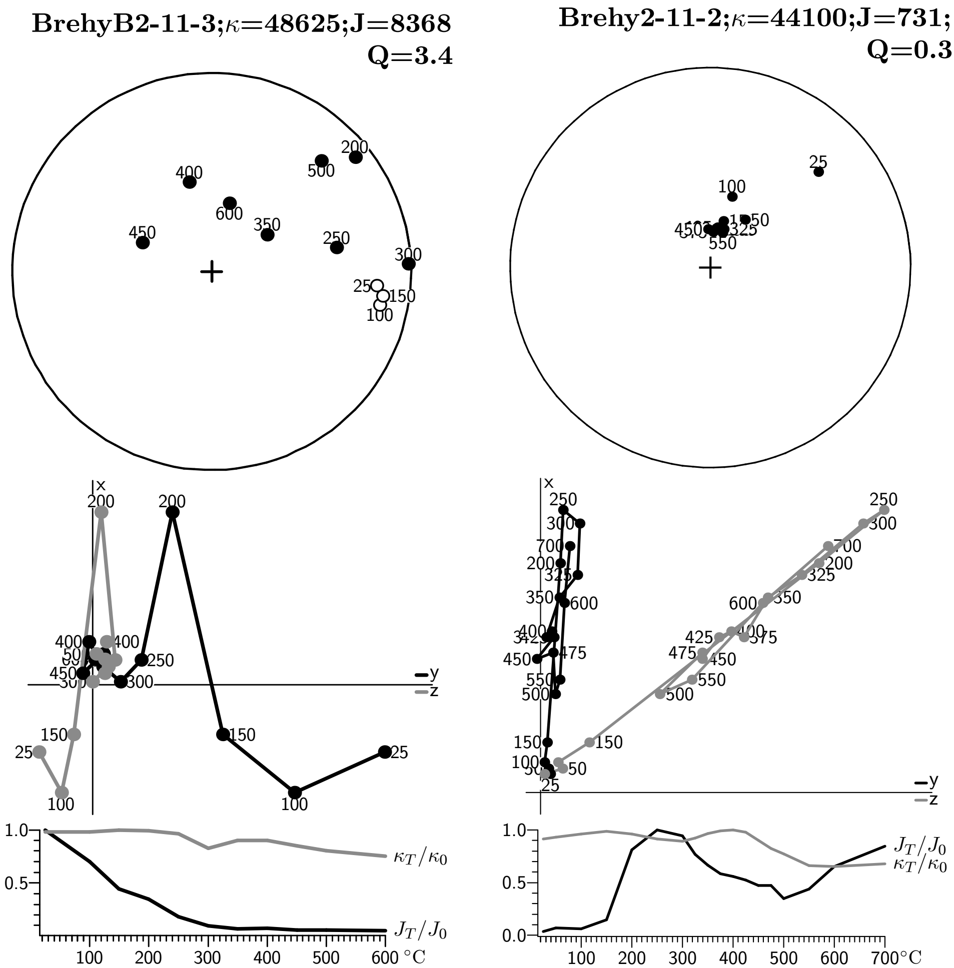
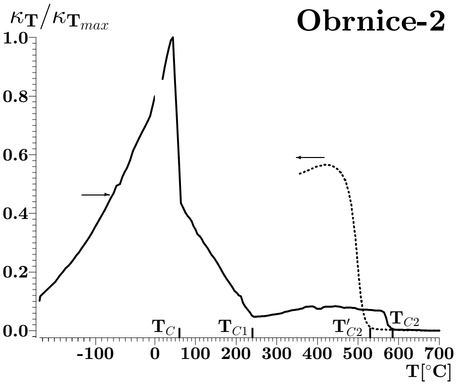
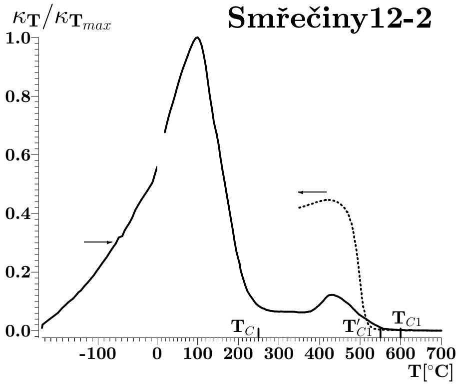


Fig. 4. Thermal demagnetization of basalt sample Brehy B2-11-3 and thermal magnetization (PTRM) of basalt sample Brehy 2-11-2 in normally oriented magnetic field (age of basalts = 0.13-0.16 [0.22] Ma; the geographical coordinates of locality: $\varphi_L = 48.410°$, $\lambda_L = 18.650°$); stepwise heating to 700° C; Zijderveld diagrams and stereographic projections; • (◦) means positive (negative) polarity of RM; κ_T (κ_0) - magnetic susceptibility at T (at 25° C, respectively); J_T (J_0) - remanent magnetization at T (at 25° C, respectively).

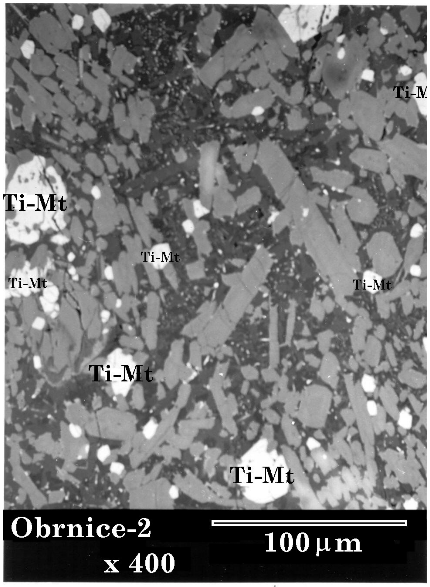
during cooling. As we see from Fig. 1, the sample Šomoška2a-1-1 has shown only very soft variation of κ during thermal magnetization. The original RM during thermal magnetization strongly decreased at 150° C, but after that





Gr.s.	TiO_2 (%)	FeO (%)
60	24.37	74.81
8	23.56	75.82
8	24.07	75.15
6	23.63	75.44
6	23.23	75.61

Gr.s.	TiO_2 (%)	FeO (%)
70	18.90	78.51
10	20.74	77.98
10	19.98	78.14
9	19.48	79.21
7	21.91	76.53



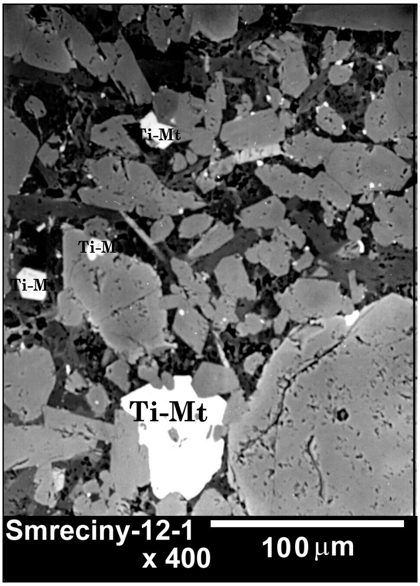


Fig. 5. Thermomagnetic curves, the results of microprobe analyses and microphotographs of Fe-Ti grains (mostly of light, or light-dark tint on the image) of the basalt samples from localities Obrnice ($\varphi_L = 50.428°$, $\lambda_L = 13.751°$) and Smřečiny12-2 (12-1) ($\varphi_L = 50.632°$, $\lambda_L = 15.279°$); T_C, T_{C1}, T'_C, T'_{C1} – Curie temperatures during heating and cooling (T') of the sample; $\kappa_T/\kappa_{T\max}$ – magnetic susceptibility at temperature T, and maximum magnetic susceptibility ($\kappa_{T\max}$). Gr. s. – grain size in μm.

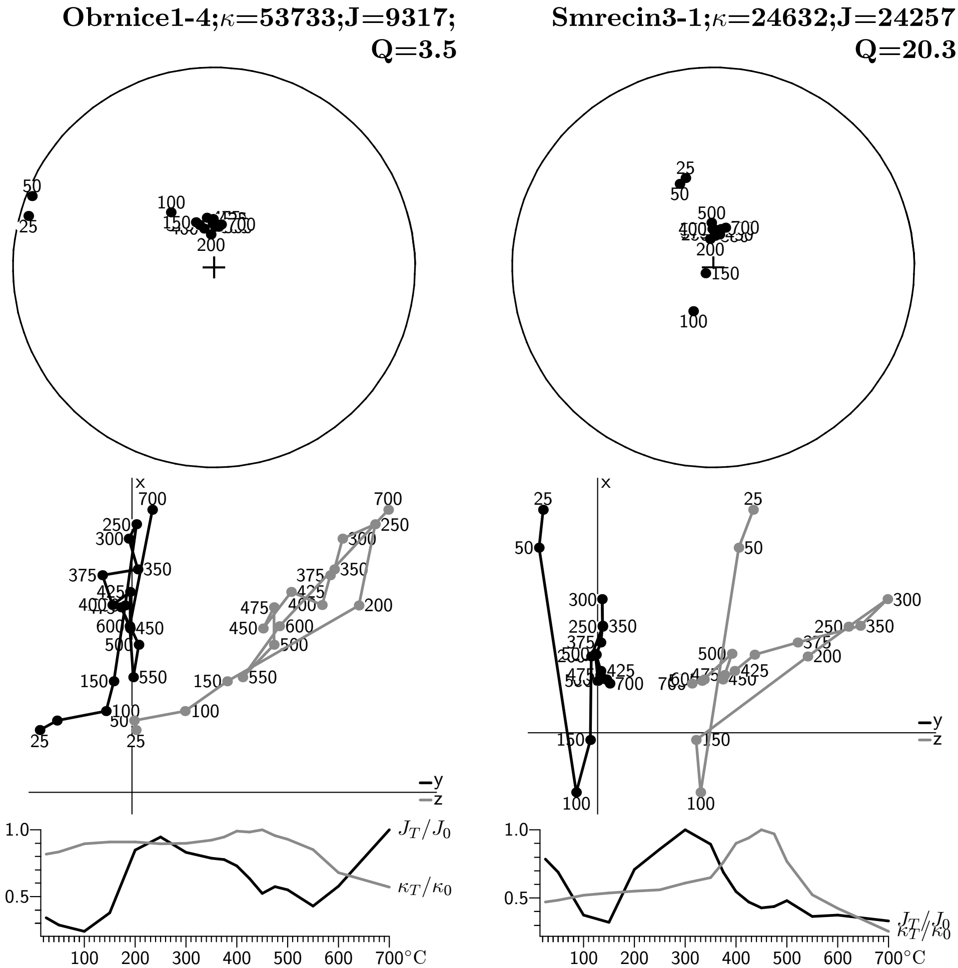
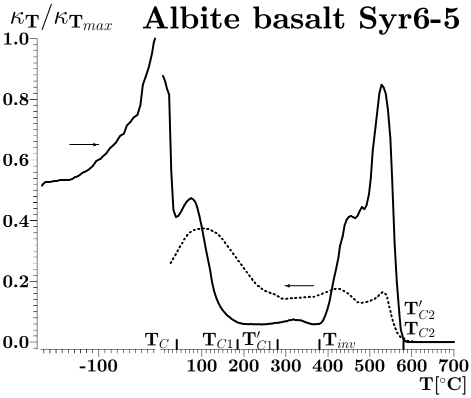
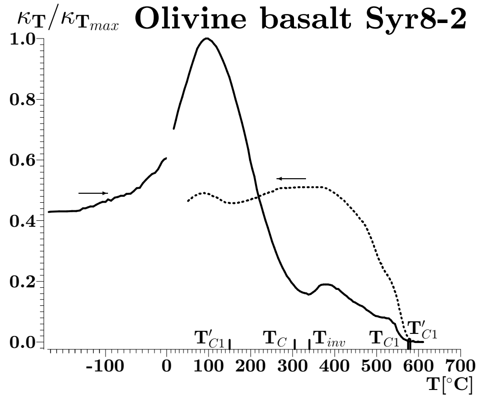


Fig. 5a. Thermal magnetization (PTRM) of basalt samples of Obrnice locality and Smrečiny locality (both of the same geographical coordinates as in Fig. 5); The inclination and the declination of original RM of rocks are respectively I = -31.3°, D = 229.3° (Obrnice-2-2-1), I = 16.4°, D = 328.6° (Smrečiny12-1-7); stepwise heating to 700° C; Zijderveld diagrams and stereographic projections; • (○) means positive (negative) polarity of RM; κ_T (κ_0) - magnetic susceptibility at T (at 25° C, respectively); J_T (J_0) - remanent magnetization at T (at 25° C, respectively).

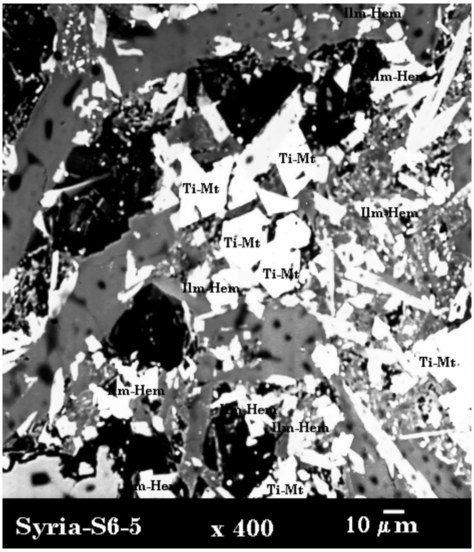
the PTRM increased from a value of 8035 nT to 11220 nT at 700° C. No typical increase of κ and decrease of PTRM at about 450° C (as for basalts





Gr.s.	TiO_2 (%)	FeO (%)
40	24.27	70.49
6	27.23	69.79
8	24.98	70.64
7	25.56	70.42
10	25.60	70.57

Gr.s.	TiO_2 (%)	FeO (%)
40	25.48	73.85
8	23.66	75.50
6	25.68	72.55
10	25.60	73.98
8	25.50	75.58



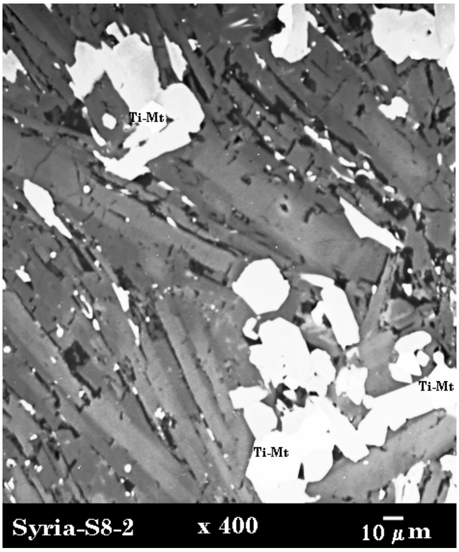


Fig. 6. Thermomagnetic curves, the results of microprobe analyses and microphotographs of samples of Cretaceous basalts from Syrian Arab Republic: Syr6-5 - albite basalt and Syr8-2 - olivine basalt ($\varphi_L = 34.77$ to $35.38°$, $\lambda_L = 36.16$ to $36.30°$); T_C, T_{C1}, T'_C, T'_{C1} – Curie temperatures during heating and cooling (T') of the sample; κ, $\kappa_T/\kappa_{T\max}$ – magnetic susceptibility, at temperature T, and maximum magnetic susceptibility; Ti-Mt – titanomagnetite; Ilm-Hem – ilmenite-hematite; Gr. s. – grain size in μm.

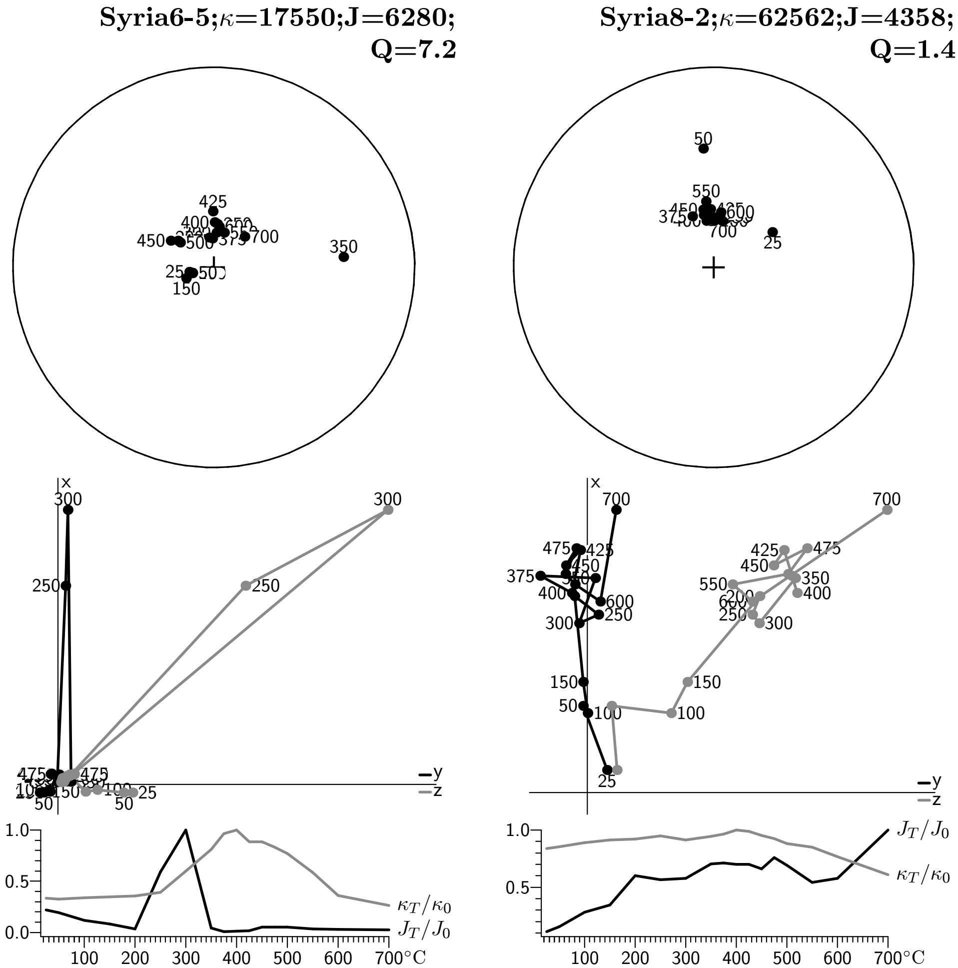
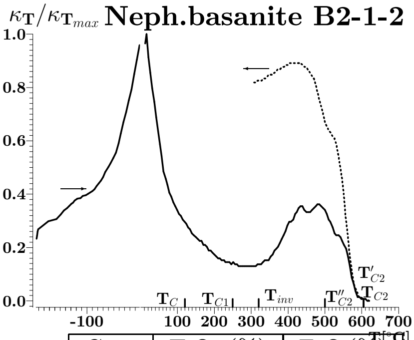
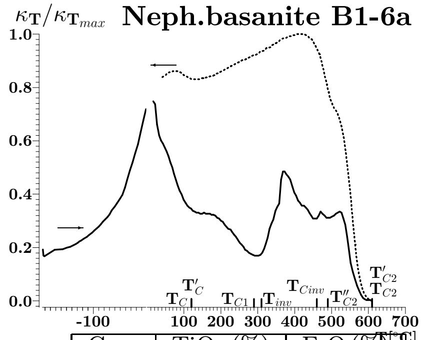


Fig. 6a. Thermal magnetization (PTRM) of basalt samples Syria6-5 and Syria8-2 in normally oriented magnetic field (both samples from the localities of the same geographical coordinates as in Fig. 6). The inclination and the declination of original RM of rocks are respectively I = −16.2°, D = 209.8° (Syria6-5-2), I = −19.4°, D = 191.3° (Syria8-2-2); stepwise heating to 700° C; Zijderveld diagrams and stereographic projections; • (◦) means positive (negative) polarity of RM; κ_T (κ_0) - magnetic susceptibility at T (at 25° C, respectively); J_T (J_0) - remanent magnetization at T (at 25° C, respectively). Other actual descriptions and explanations see in Fig. 6.

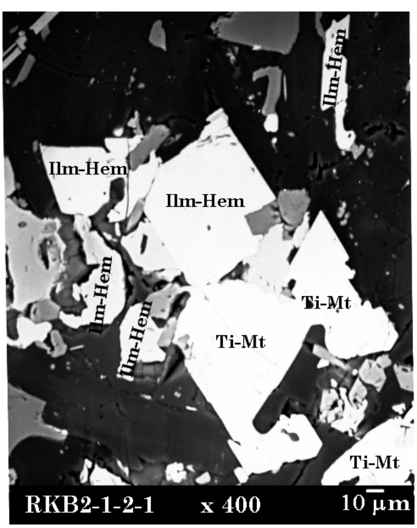
with the reversed RM) has been detected. The basalts with such type of behaviour have not been found in other places of the area under study.

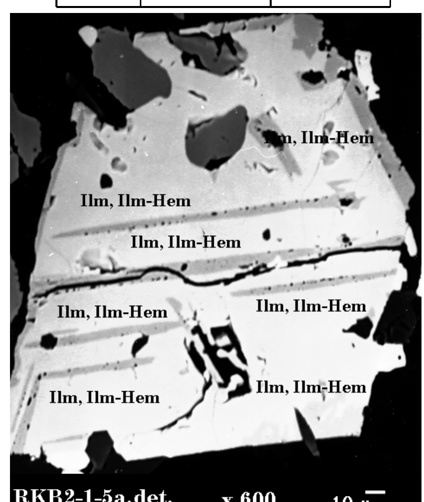


Gr.s.	TiO_2 (%)	FeO (%)
90	25.99	72.98
10	25.03	74.33
15	25.13	74.04
12	51.60	48.06
7	21.48	77.95



Gr.s.	TiO_2 (%)	FeO (%)
80	24.23	75.43
110	51.23	48.45
8	23.82	75.07
9	26.61	72.95
6	50.45	48.98





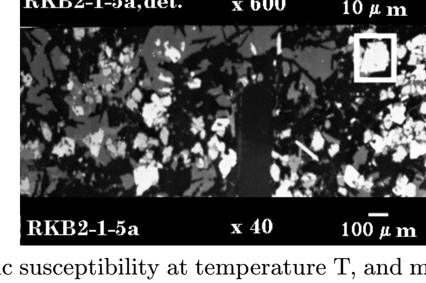


Fig. 7. Thermomagnetic curves, the results of microprobe analyses and microphotographs of the Cretaceous to Jurassic nepheline basanite samples RKB2-1-2-1 and RKB1-6a (φ_L = 12.503 to 12.505°, λ_L = 7.186°); T_C, T_{C1}, T'_C, T'_{C1} – Curie temperatures during heating and cooling (T') of the sample; κ, $\kappa_T/\kappa_{T\max}$ – magnetic susceptibility at temperature T, and maximum magnetic susceptibility; Ti-Mt – titanomagnetite; Ilm-Hem – ilmenite-hematite; Gr. s. – grain size in μm.

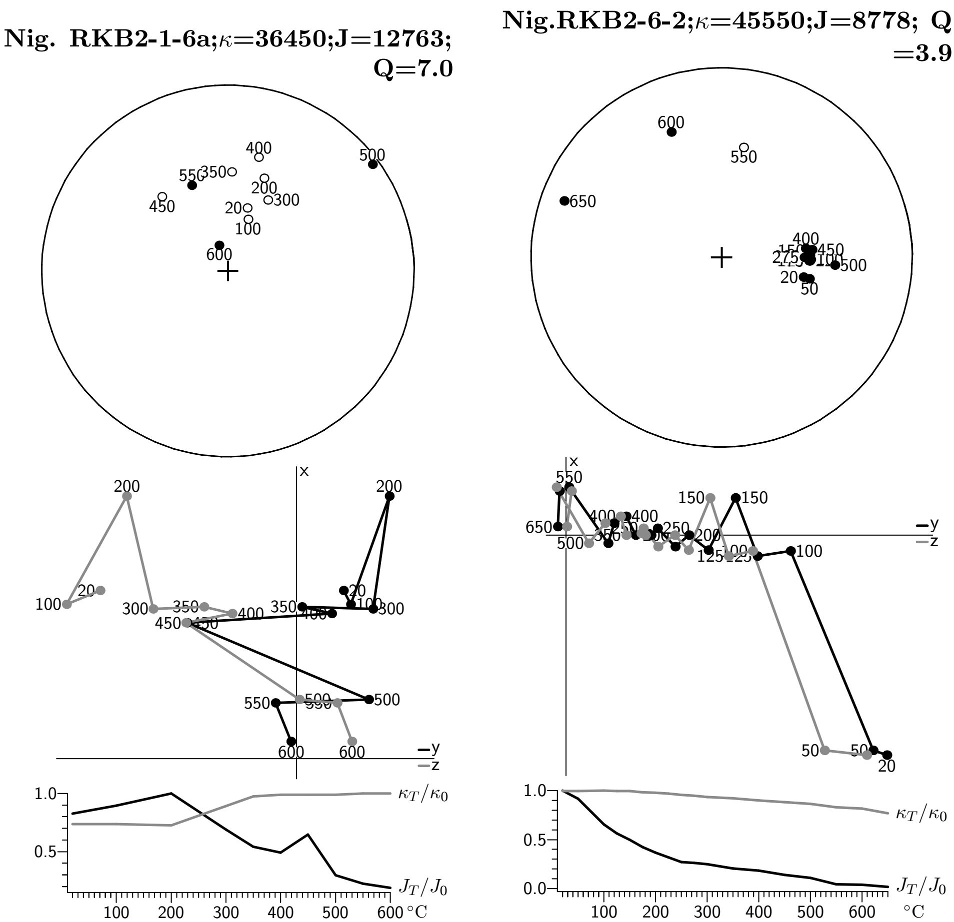


Fig. 7a. Thermal demagnetization of nepheline basanite samples from Nigeria. The inclination and the declination of original RM of rocks are respectively I = −10.3°, D = 213.4° (RKB2-1-6a), I = −13.4°, D = 34.9° (RKB2-6-2); stepwise heating to 700° C; Zijderveld diagrams and stereographic projections; • (○) means positive (negative) polarity of RM; κ_T (κ_0) – magnetic susceptibility at T (at 25° C, respectively); J_T (J_0) – remanent magnetization at T (at 25° C, respectively). Other actual descriptions and explanations see in Fig. 7.

Evaluation of the κ change measurements, magnetizing curves and Zijderveld diagrams of basalts with only reversed RM. From all examples in Figs. 2-5, 5a, 6, 6a, 7 it has been shown that there is an increase of κ, mostly at about 450° C, for continual measurements (the 2nd phase in a frame of the Curie temperature measurements) and discrete measurements of the change of κ with temperature. At the same time, the minimum value of the PTRM is induced around 450° C. We see from the Zijderveld diagrams (Figs. 2-4, 5a, 6a), that the projection of the end vector of RM to both, xy and xz planes has decreased its value and has an opposite (reversed) direction with respect to the original magnetized direction of PTRM, up to about 300° C to 450° C. We can interpret it as an acquiring of the self-reversed PTRM. By the behaviour as described above are characterized not only the basalts presented in Figs. 2, 3, 5, 5a, 6, 6a and 7, but also the basalts from one locality of the Krupinská Planina Highland (Devičie; $\varphi_L = 48.317°$, $\lambda_L = 19.030°$) and from further three localities of the Cerová Vrchovina Highland (Steblová skala: $\varphi_L = 48.250°$, $\lambda_L = 19.986°$: East of Steblová skala: $\varphi_L = 48.317°$, $\lambda_L = 19.992°$; Velký kopec: $\varphi_L = 48.228°$, $\lambda_L = 19.948°$. What is exceptionally important is that such behaviour is shown also by the basalts from two places of 22 investigated locations of the basaltic body of the Brehy locality. This nepheline basalt is the youngest volcanic rock in the Western Carpathian Mts. Its age is about 0.13-0.16 (0.22) Ma. The microprobe analysis of 5 grains (6 to 20 μm in diameter) of the sample B2-16 (similar to B2-11) established the concentration of TiO_2 = 22.42 to 25.63% and FeO = 73.48 to 76.64%. From Fig. 4 is evident that an original RM of the sample BrehyB2-11-3 is of the reversed inclination up to 150°, during thermal demagnetization. We need to stress that no reversed polarity of the geomagnetic field existed in the age described above, according to the world-wide scales.

3. Discussion and conclusions

In my professional works I have so far outlined the way for the realistic interpretation of the reversed RM of rocks, dominantly for the volcanic rocks, based on an idea of the self-reversal mechanism, not due to a reversed orientation of the geomagnetic field.

So far, in many intermediate volcanics - andesites, basaltic andesites and rhyolites of the Neogene age, the reproducible self-reversal partial thermo remanent magnetization (srPTRM) was induced in the laboratory field of normal polarity and the intensity of H $\approx 48\,\mu$T. Together, the samples from about 177 localities from central and eastern Slovakia acquired a srPTRM of the intensity in the range of 2.0 to about 200 nT (*Orlický, 2001, 2002, 2002a, 2002b, 2002c, 2003, 2003a*). Similarly, the srPTRM in selected acidic types of volcanics of the Eocene to Miocene age from the Bohemian Massif was induced in the laboratory (*Orlický, 2002d*). An interpretation of the reversed, or partly reversed RM of the Neogene volcanic and volcano-sedimentary rocks of central Slovakia was made on the basis of non-complete self-reversal origin (*Orlický, 2003b*). Partial and total srTRM was studied in the rhyodacite of the Haruna Volcano (Japan) and the dacite ash of Mount Pinatubo from Philippines. As has been well known the carriers of the self reversal TRM in these rocks are the Ilmenite-hematites of the range Ilm_{45} Hem_{55} (*Orlický, Funaki, Pagáč, 2000; Orlický, Funaki, 2000; Orlický, Funaki, 2001; Orlický, Funaki, 2002*). In a recent time the srPTRM was induced in two titanium rich titanomagnetites of basalts from southern Slovakia (*Orlický, 2006*). Considering an idea about the self-reversal origin of the reversed RM of volcanic rocks, a realistic interpretation of magnetic and paleomagnetic results of basaltic rocks from southern Slovakia was presented by *Orlický (2006a)*. Especially, the great attention has been paid to the Ti-rich titanomagnetite (Ti-Mt) bearing basalts with the self-reversal RM. They contain small, SP-like behaviour magnetic particles. So far not well understood idea about the possibility of the self-reversed origin of RM of rocks, containing small magnetic particles, has been accepted in paleomagnetism. In this article I present the experimental results of the laboratory magnetization of the natural basaltic samples, which can be accepted for the establishment of a relevant theory. For this purpose, the so far accepted theory for the explanation of the reversal process in physics of synthetic small magnetic particles outlined in the introduction of this article will be modified for the processeses in paleomagnetism.

Experimental evidences about the origin of RM of rocks

1. This paper presents the results of the investigation of basalts of: a) The Pleistocene to Quaternary age from southern Slovakia; b) The Miocene to Pliocene age from České Středohoří Mts. of North Bohemia; c) The Cretaceous age from the Syrian Arab Republic; d) The Cretaceous age from Nigeria. The selected basalts have the reversed or at least

anomalous, partly reversed RM. These types of basalts contain either dominant, or at least small part of Ti-rich Ti-Mt-es, as the carriers of magnetic properties. In all basalts also a more oxidized magnetic phase has been detected. The basalts of only three localities with only normal RM from southern Slovakia were selected to compare their properties with those of the reversed RM. These types of basalts from locality Blhovce-Buda contain only more oxidized Ti-Mt phase, near to magnetite. The basalts of the locality Šomoška have also the Ti-rich Ti-Mt-es, but evidently the RM is linked to the more oxidized phase.

2. As has been documented above, the basalts containing the Ti-rich Ti-Mt-es and Ti-Mgh secondary phase of all investigated localities have shown the reversed, or at least anomalous, partly reversed directions of RM. The reversed direction of these samples survived up to about 400 to 500° C (mostly to 450° C) during thermal demagnetization in a fully compensated external magnetic field. In these types of basalts the κ reached the maximum value, during temperature treatment, in the interval 400 to 500° C (mostly at 450° C), exceptionally at 550° C (for more oxidized basalts). At this point, we need to take into account the results of continual measurements of the change of κ of magnetic fraction of the Ti-rich Ti-Mt-es of samples. It is evident that these results coincide with the results of the change of κ of the compact samples in discrete temperature intervals during magnetizing process. What is very important, the minimum PTRM was acquired by samples at the temperatures corresponding to maximum values of κ. This behaviour has supported the idea that the above mentioned maximum κ corresponds to a presence of small magnetic particles in the respective basalts. We need to recall the additivity law of PTRM or TRM acquisition in magnetic minerals with the developed thermodynamically stable domain structure. We see that the results have not proven the idea about the acquiring of TRM at temperatures close to the Curie temperature of magnetic mineral, or close below T_C. This is a very important phenomenon, that the reversed RM was induced at the temperatures corresponding to maximal values of magnetic susceptibility. Rather similar behaviour of the κ was registered in basaltic andesites and other intermediate andesites containing residual portions of the T-rich Ti-Mt-es, from central and eastern Slovakia volcanic fields.

Some consideration about the titanomagnetites. We do not know precisely how, and from what sources, the original Ti-Mt-es have created. According to *Bonatti (1994)*, the basaltic magma comes from the peridotites, which contain three important silicate minerals: olivine-silicate containing Mg and Fe, orthopyroxene and clinopyroxene, they have also small quantities of spinel, and the oxide chromium. We can only assume that the Ti-Mt-es arose in a nascent state, as a very tiny particles.

The results of this work have evidently shown that the basalts containing dominantly the Ti-rich Ti-Mt-es, or some portion of them (except of more oxidized derivatives of the Ti-Mt-es), have acquired originally the reversed RM in the field. They have also a tendency to be magnetized by the self-reversal mechanism in the normally oriented magnetic field.

The second important phenomenon, which supposedly plays an important role in the acquisition of the reversed RM is a magnetic state of magnetism carriers in the rocks. The Ti-Mt-es, including those with the Ti-rich ones, have been recently considered as the multi-domain ferrimagnetic materials. I have revealed that the titanium-rich titanomagnetites behave dominantly as the SP-like particles (*Orlický, 2004*). So, they have properties corresponding to small magnetic particles. They have not a thermodynamically stable domain structures. Analyzing the results of continual measurements of dependence of κ of Ti-rich Ti-Mt-es on the temperature, we see that the minimal κ lies at about $-196°$ and the maximum value is in the range of about 50 to 100° C (these characteristics are dependent on the x value; for a higher x value the maximum κ is shifted to lower temperatures). The Curie temperature for the magnetic phase with low T_C is at about 120 to 190° C. The κ has evidently changed with the temperature. Such behaviour of κ corresponds to small magnetic particles, according to Landau and Lifshitz in *Hubert and Schäfer (1998)*. According to the authors, the magnetic particles with the developed thermodynamically stable domain structure are athermal, which means that they do not change any magnetic characteristic with the temperature. They change only around their Curie temperatures.

Analyzing the microphotographs of the scanning microscope in Figs. 5-7, and those not presented in this article, we see, that the larger Ti-Mt (or Fe-Ti) grains are frequently present in the rocks of normal RM. But in the rocks with the reversed RM are frequently present the grains of a diameter less than 10 μm, mostly below a size around 1 μm or less. The grains of larger sizes are present also in the mentioned rocks, but they are quite rare in a less portion (e.g. in Fig. 5 we see that in the sample Obrnice-2 (on a plain 200×260 μm) there are about 40 grains of a diameter 1 to 30 μm, but more than about 100 grains with a diameter less than 1 μm. On the sample Smreciny-2 (on a plain 200×260 μm) there are about 5 grains of a diameter 1 to 40 μm, but more than 45 grains of a diameter less 1 μm (more precise image has been achieved with the magnification of the picture). The small grains may have not developed the classical domain structure, respecting an idea, that the classical domains are preferentially created in larger sizes of grains. The particles of smaller sizes probably behave as small magnetic particles, if not as a complete grain, at least in its particular part.

Orlický (2004) studied the problem of high values of volume magnetic susceptibility (κ) of Ti-rich Ti-Mt bearing basalts. On the basis of only magnetic parameters he deduced, that high values of κ do not correspond to high concentration of Ti-Mt-es in the samples, but it is a consequence of the SP-like behaviour of small magnetic particles of Ti-rich Ti-Mt-es in the respective basalts. In *Orlický (2004)* there were described the principles of the superparamagnetism (according to *Stacey and Banerjee, 1974*). The SP particles are supposed to be largely spread in volcanic and sedimentary rocks [(e.g. according to *Creer (1961)*, the magnetic minerals in red sands are dominantly in a SP state)]. Its existence is of very large importance with respect to magnetization of these rocks. Unfortunately, so far no comprehensive description about an important role of small magnetic particles has been adopted in the paleomagnetic literature.

During long time the SP particles in the Ti-rich Ti-Mt-es were not proven by conventional analytical methods. It has been influenced by the fact, that commonly the electron microprobe analysis was applied to study the composition of Fe-Ti oxides in the rocks, but this method is not able to analyse the grain size below 1 μm. We have known from experimental works and published knowledge, that the Ti-Mt-es have a strong tendency to be very easily oxidized in the presence of oxygen. On the other side, if protected by high vacuum and heating them over 800°, the oxidized Ti-Mt-es have a tendency to be reduced to ones with low Curie temperature and relatively high value of x, (*Orlický et al., 1992*). But, the Ti-Mt-es of low Curie temperatures and relatively high compositional parameter $x \approx 0.6$ commonly occur in the continental, as well as in marine basalts (in the marine basalts the composition around of 0.6 is the most predominant one). Under what conditions do they occur and are preserved in nature? In magmatic conditions there is commonly a lack of oxygen. From the results of *Zhou et al. (1997)* it is evident that a high portion of the Ti-rich Ti-Mt-es in basalts contained in the small globules which are surrounded by a glassy matrix, and so they are protected against any oxidation during the ascent of magma onto the earth's surface.

In magnetism and paleomagnetism of rocks an idea has been generally accepted that the Fe-Ti magnetic minerals are characterized by the thermodynamically stable domain structure. This idea has been taken into account also for the establishment of the theory for inducing thermoremanent magnetization (TRM) of volcanic rocks, according to *Stacey and Banerjee (1974)*. So far the idea about the dominant position of a thermodynamically stable domain structure was applied also to the titanomagnetite (Ti-Mt) magnetic minerals, which are the main carriers of magnetic properties in basaltic rocks.

A preliminary simple model for the domain state of Ti-rich Ti-Mt-es has been published by *Appel and Soffel (1984, 1985)*. Following the idea of the model, particles of multidomain (MD) grain size should consist of MD, single domain (SD) and spin cluster regions. The consequence *is the decrease of the effective grain size* (division into subvolumes) and an enhanced importance of spin rotation processes. In observations of saturation remanence in TM60 grains *Halgedahl and Fuller (1980, 1983)* saw both SD and MD states over a wide range of grain sizes. If TM60 is stress-free, it has a cubic anisotropy (with $K_1 \geq 0$) and L_{SD}^{rem} is highly sensitive to small changes in composition, shape and crystallographic orientation. In practice, domain observations of TM60 suggest that the anisotropy is usually dominated by stress and is uniaxial. Depending on the orientation of the stress-induced easy axes, this may increase or decrease the size range for SD remanence. *Halgedahl and Fuller (1980, 1983)* claimed that the nonuniform states occurred because defects promote nucleation, but easy axis orientation is another possible mechanism. When $L_{SD}^{rem} \geq L_{SD}^{coerc}$, the coercivity will decrease considerably before the remanence starts to decrease.

Radhakrishnamurty et al. (1981), who studied the magnetic domain state of synthetic Ti-Mt-es with x = 0.0–1.0, for the composition of the range x = 0.3 to 1.0 has suggested, that these materials are not effectively in a MD state and their magnetic behaviour in some cases resembles that attributed to spin glasses. In contrast, magnetite (x = 0.0) readily forms multidomains upon synthesis. Materials with x = 0.1 and 0.2 have magnetic properties different from those of other compositions, and a possible MD state for them cannot be excluded. The results of study, involving about a thousand samples of basalts of different ages and modes of origin, indicate that qualitatively the magnetic behaviour of basalts is almost entirely determined by their ferrimagnetic domain structure (MD, SD, SP) or sometimes by lattice composition as with advanced cation deficiency (CD) in magnetite. The synthesized Ti-Mt samples (TM56, TM60) had properties inherent to the Ti-Mt series. The Ti-Mt-es in the range TM30 - TM100 formed only single domains or spin clusters. An implicit assumption in the literature, that all members of the Ti-Mt series can occur in the MD state, has been misleading. While the magnetite easily forms MD structures, the Ti-Mt-es in the range TM30 - TM100, even when coarse-grained, do not exhibit the magnetic properties expected from the MD state. The single-domains behave like a stable-SD fraction or SP fraction. A peak in the κ-T curve of samples containing single domains may reflect the maximum contribution to susceptibility from the superparamagnetism of the single-domains at the blocking temperatures. Low-field hysteresis properties at 300 K indicate the presence of SP particles even in the range TM30-TM68. The authors did not know, whether the inferred SP content increased with the content of ulvöspinel in solid solution with magnetite.

In the marine basalts from the Juan de Fuca Ridge and from the young ($\leq$ 20 ka) basalts from the axis of the East Pacific Rise at 12° N) *Zhou et al. (1997)* revealed three ranges of grain sizes for the Fe-Ti oxides. The largest grains are one to tens of micrometers in diameter, intermediate sized grains are of diameter 1 to 0.4 μm, or even smaller. These types have occurred as the discrete Ti-Mt-es. But in the globules surrounded by a glassy matrix, there were in high concentration very fine grained, submicrometer Fe-Ti oxides. The authors have differed the large, micrometer-sized Ti-Mt-es with relatively constant ulvöspinel contents near TM60, of MD size range, submicron grains of Ti-Mt associated with residual glass and having a range of composition TM25 to TM66. Some of this material has sizes in the SD range. The discrete fine-grained Ti-Mt can be an important source of stable NRM. However, such kind of Ti-Mt-es are less abundant than those originating from the immiscible liquids. These have sizes less than 0.4 μm, corresponding to SD and SP material. They have a broad range of composition, from TM0 to TM79. Judging from the distribution of grain sizes, SD grains appear to be more common than SP grains. A large portion of fine-grained Ti-Mt grains (both discrete and in globules) has Ti/Fe ratios similar to large grains. The authors have estimated that approximately 15% of the fine-grained Ti-Mt has lower Ti contents (x values ≤ 0.6). The morphology and distribution of immiscible globules and the composition of Fe-Ti oxides resulting from crystallization depend on the composition of the initial immiscible liquids and their cooling rate (the initial composition of immiscible liquids vary locally even in the same sample).

On one side, the small particles do not have a classical domain structure and so, they are not able to acquire the remanent magnetization, according to *Mc Elhinny (1973)* and *Mc Elhinny and Mc Fadden (2000)*. On the other side, it has been published by *Krása et al. (2005)*, that these particles have acquired relatively high M_s, magnetized by the intensity of 1.7 T, and the M_s has followed the shape of κ behaviour with temperature. I suppose that small magnetic particles are able to receive also a RM, under precisely defined particle size. The course of magnetization of the basalt samples at discrete temperatures and measurements of PTRM at laboratory temperatures is rather different. In the interval from 25 to about 150° C there is evident demagnetization of original RM. Then conspicuous increase of PTRM is shown with the maximum value at about 300° C. Then decreasing of PTRM starts mostly up to 450° C, what is supposed to be some minimum of PTRM. This is a common feature of the behaviour of Ti-rich Ti-Mt-es with the presence of the Ti-Mgh phase, during their magnetization, influenced by temperature. Except for my results, the above described behaviour of the Ti-rich Ti-Mt-es has been proven also by the results of *Pan et al. (2006)*, who studied the Thellier-Coe paleointensity of Al-substituted Ti-Mt-es of basalts. The authors have revealed that between 300 and 460° C, specimens acquire thermoremanence with a direction antiparallel to the external field direction, leading to intensity decreases.

It can be concluded that the behaviour of Ti-rich Ti-Mt-es and the acquisition of the self-reversed PTRM by the small magnetic particles in the rocks, as has been presented above, is a general world-wide phenomenon, based on my experimental results and those presented by foreign authors. All results have proven very strong tendency for the self-reversal of the PTRM of small magnetic particles in the studied basalts. In the presented stage experimental work was carried out with the assemblages of magnetic Fe-Ti oxides in basalts, so, like in the study of synthetic magnetic minerals, the effects related to details of the size and the shape of individual particles were concealed by averaging process, according to *Bode et al. (2004)*. From the so far achieved results it is quite evident, that the rocks containing the magnetic minerals with the thermodynamically stable domain structures are only of normal RM, but the rocks with the magnetic minerals, dominantly as the small magnetic particles, may attain the reversed RM during magnetization. To prove this assumption it is necessary to investigate the domain structure of the Te-Ti minerals, and to make further effective analyses of the rocks.

Small, including the SP, particles contain no domain pattern even if they are commonly called single-domain particles. According to *Hubert and Schäfer (1998)*, the magnetic domains are not an universal feature of ferromagnetic materials. In low-anisotropy materials there is an intermediate range, in which continuous micromagnetic vortex states, rather than classical domains, prevail. Even if anomalous, continuous magnetic microstructures do not look like domains; this does not mean that domain models, in which continuous transitions are replaced by domains and walls, are useless. Such models offer a first understanding for a complex situation and can be expected to be very useful if properly selected and evaluated. If a small particle is first saturated at high field and the field is then reduced (and reversed, if necessary) it will in general switch at some point to opposite magnetization direction, or to some other state closer to equilibrium. Blocked transitions between these equilibrium states lead to hysteresis. The saturated or almost saturated state is usually metastable on approaching the equal energy limit, it cannot switch without some excitation, as long as there is an energy barrier. The barrier may be overcome by thermal activation, depending on its height and shape. This leads to magnetic viscosity (disaccommodation) and thermally induced loss of magnetization - superparamagnetism (*Hubert and Schäfer, 1998*). The switching field is defined as the point of instability of the near-saturation state.

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