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An explanation of some effects of the titanomagnetite solid solutions ($\mathbf{Fe_{3-x}Ti_xO_4}$) based on the magnetic susceptibilities, the Curie temperature measurements, and the Mössbauer spectra measurements

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Abstract: The natural titano-magnetites (Ti-Mt-es) from basaltic rocks were investigated. The titanomagnetite solid solutions with high content of ulvöspinel ($x \approx 0.6$ to 0.8, and $T_C \approx 80$–110° C) have attained very high values of volume magnetic susceptibility. These Ti-Mt-es are in a superparamagnetic (SP) state. No typical multi-domain structure was developed in these Ti-Mt-es. In some basaltic rocks the Ti-Mt-es with two or more magnetic phases are frequently present. One phase is probably of the original state and the second one has been created due to the so called low temperature oxidation (below 350° C) of original Ti-Mt-es. This second titano-maghemite magnetic phase is characterized by the inversion temperature. In this magnetic phase a stable domain structure of multidomain mode probably developed. Both these Fe-Ti magnetic phases are in a contact with each other. Experimentally it has been proven that mutual magnetostatic interactions between the SP particles and those with the developed domain structure exist. These interactions have probably strongly influenced also the behaviour of the activated material during realization of the Mössbauer spectroscopy and so, the Mössbauer spectra of such material are non-Lorentzian, and very broad. Preferably the octahedral positions have occurred on the Mössbauer spectra of the original Ti-Mt-es at room temperatures, while the tetrahedral positions are characteristic at LNT for the same sample of the Ti-Mt.

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Key words: titanomagnetite, non-Lorentzian MS spectra, magnetostatic interactions, SP particles, multi-domain particles

1. Introduction

Since the first studies of the magnetic minerals, some magnetic properties and the composition of the Fe-Ti oxides of volcanic rocks have been explained on the basis of results of magnetic parameters and those of the Mössbauer analyses. But so far no final idea has been obtained for the explanation of the so called non-Lorentzian very broad, non-interpretable Mössbauer spectra of the Ti ion bearing compounds - namely ulvöspinel rich titanomagnetites (Ti-Mt-es). We know that the Ti-Mt-es are the main magnetism carriers in basalts. *Orlický (2004)* has presented some ideas about the superparamagnetic behaviour of the ulvöspinel rich Ti-Mt-es, based on the consideration of Ti-Mt magnetic moments and their high values of magnetic susceptibilities (κ). He has commented also on some results of Mössbauer spectra, but the spectra of the respective ulvöspinel rich Ti-Mt have not been documented.

Main goal of this article is to enrich the so far obtained results and make a comparison of values of magnetic susceptibility (κ), Curie temperature and the Mössbauer spectra measurements of the Ti-Mt containing basaltic rocks and to find an appropriate explanation of the anomalous properties of the respective magnetic minerals of rocks.

2. The methodical setting and basic results

In many papers the Ti-Mt-es, as the dominant carriers of magnetic properties in basalts have been described in detail. But an exceptional property - that the Ti-Mt-es with high portion of ulvöspinel have behaved as the superparamagnetic (SP) particles - has been reported by *Orlický (2004)*. Moreover these SP particles may produce strong magnetostatic interaction with the particles containing the developed domain structure.

We will observe the changes of Ti-Mt-es depending on heating and cooling of the air, and in some samples also in vacuum. We see in Fig. 1, that in the

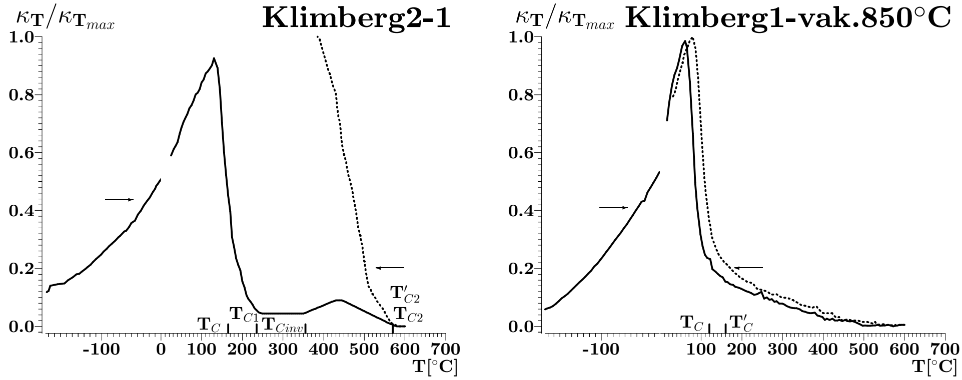


Fig. 1. Thermomagnetic curves of the titanomagnetite samples: Klimberg2-1 – magnetic fraction separated from the basaltic rock - in situ state; Klimberg1-vak. 850° C - magnetic fraction separated from the same basaltic rock as the Klimberg2-1. Magnetic fraction of the sample Klimberg1-vak.850°C was sealed into evacuated ampoule, heated to 850° C, and kept at this temperature during 150 min. After cooling of the sample to room temperature the Curie temperature was measured. κ_T (κ_{Max}) - magnetic susceptibility at the temperature T; κ_{Max} – maximum value of magnetic susceptibility.

magnetic fraction of the sample Klimberg2-1 (basalt from the České Středohoří Mts.– North Bohemia), there is a magnetic phase with $\mathrm{T}_C \approx 165^\circ$ C, $\mathrm{T}_{C1} \approx 225^\circ$ C, a phase with the so called inversion temperature at about 365° C, and magnetic phase with Curie temperature of $\mathrm{T}_{C2} \approx 580^\circ$ C. After heating the sample to 600° C air and successive cooling, the oxidized Fe-Ti phase with $\mathrm{T'}_{C2} \approx 580^\circ$ C originates. This phase contains dominantly non-stoichiometric magnetite. The sample Klimberg1-vak.850° C comes from the same basalt sample as Klinberg2-1 (the sample of Ti-Mt fraction was sealed in the evacuated ampoule, heated to 850° C, kept at this temperature 150 min.). After cooling the sample to room temperature the change of magnetic susceptibility with temperature was measured in the range 600° C. Instead of the phase with $\mathrm{T}_C \approx 165^\circ$ C (in-situ sample), the new dominant phase of lower $\mathrm{T}_C \approx 120^\circ$ C was created (Fig. 1). The phase with the inversion temperature dissapeared and new non-expressive phase without a defined T_C originated. After heating the sample to 600° C and its successive cooling, the phase with $\mathrm{T'}_C \approx 160^\circ$ C originated (nearly reversible with that before heating and cooling). These results have proven that the presence or lack of oxygen has a dramatic influence on the change of magnetic state and composition of the Fe-Ti oxide.

The electron microprobe analyses of Ti-Mt-es

We present the results of analyses of some basalt samples, combined with microphotographs and the thermomagnetic curves of the Ti-Mt samples in Figs 2–4.

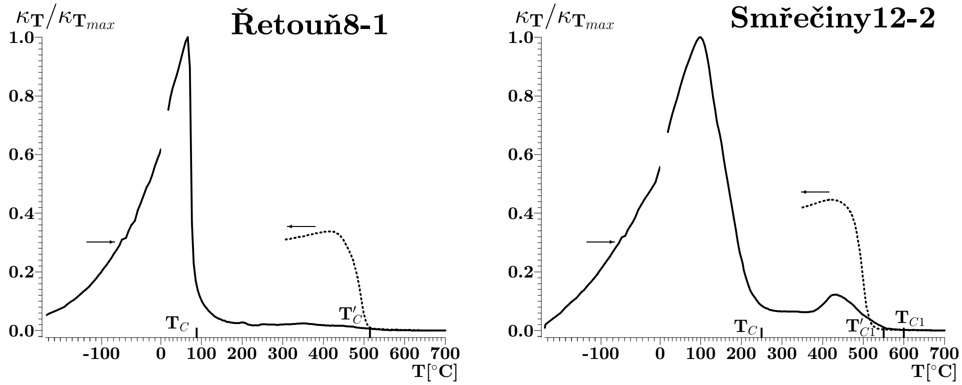
The Fe-Ti grains (6 to cca 70 μm in size) of 5 polished samples of basalts and one sample of basaltic andesite were analyzed. The analyses have proven the presence of Ti-Mt-es as dominant Fe-Ti oxides in the samples. The contents of FeO and TiO_2 in analyzed grains of individual sample is nearly homogeneous, despite their different sizes. With regard to technical parameters of the JEOL analyzer, the grains of the size less than 5 μm could not be effectively analyzed (grain size les than 5 μm lies below the threshold of the effective light beam of the instrument).

Some results of the Mössbauer spectroscopy and Curie temperature measurements of Ti-Mt samples

Several examples of the Mössbauer spectra of the Ti-Mt samples were chosen to point out their specific behaviour.

All the above presented Mössbauer spectra are not appropriate to be easily interpreted. They have too broad non-Lorentzian pattern. We see from all thermomagnetic curves that there are dominantly present the magnetic phases of low Curie temperatures in the samples, which have corresponded to titanomagnetites of high portion of ulvöspinel. But in low portion are present also second magnetic phases of low-temperature oxidation origin – titanomaghemites (e.g. Fig. 7, Steblová skala-3, T_{inv} – inversion temperature). We have assumed that the source of non-Lorentzian spectra is the magnetostatic interactions of Ti-Mt-es in SP state with tinanomaghemites in which the domain structure was developed during low-temperature oxidation.

Except of the above presented results the Mössbauer spectroscopy (MS) was previously applied to analyse small particles in the samples of basaltic rocks from Nigeria (*Lipka et al., 1988*). The measurements were done with the influence of an external field of $H = 0.6$ T (Tesla) at room temperature. The measurements did not prove the presence of small particles. Why? The examined samples contained not only the original Ti-Mt-es, but also



Gr.s.	TiO_2 (%)	FeO (%)
50	21.20	73.00
8	21.87	72.05
7	20.95	73.85
7	20.99	74.26
6	20.70	76.05

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

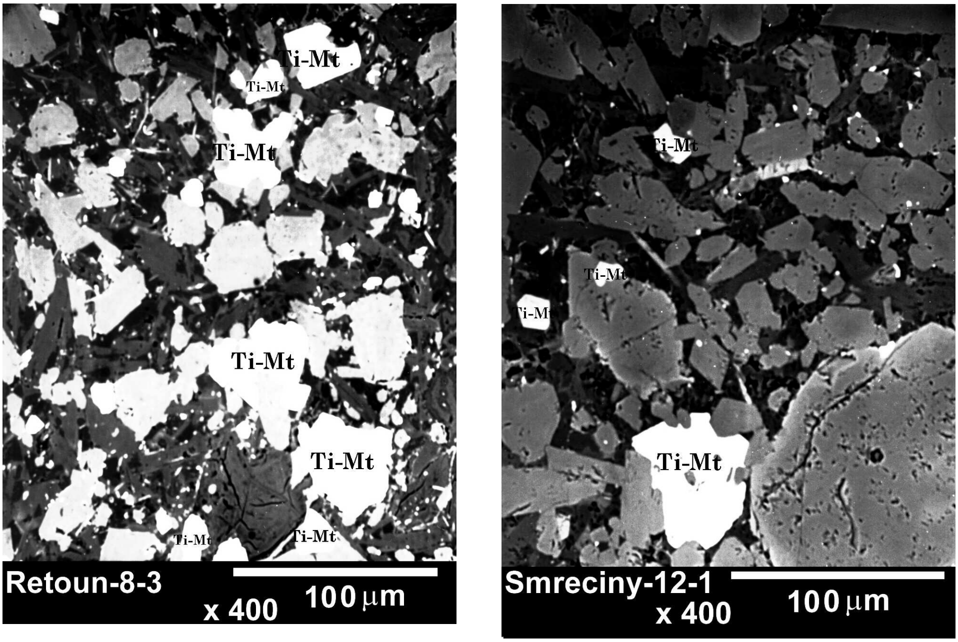
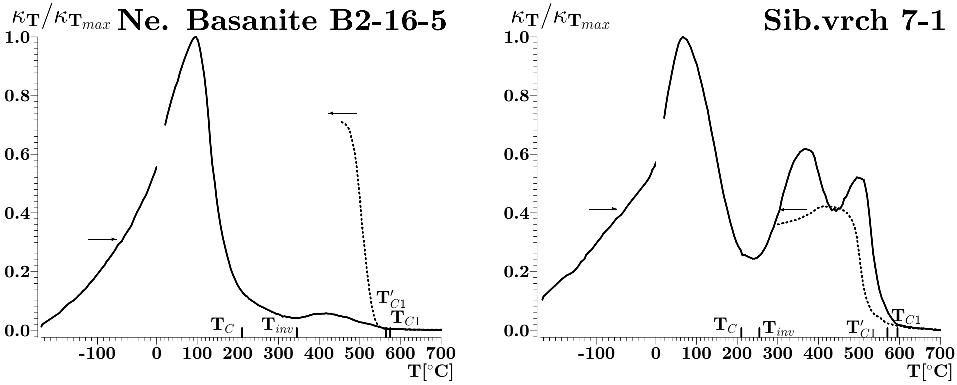


Fig. 2. Thermomagnetic curves, the results of microprobe analyses and microphotographs of Fe-Ti grains (mostly of light, or light-dark tint on the image) of the samples, Řetouň8-1 (8-3); Smřečiny12-2 (12-1); T_C, T_{C1}, T'_C, T'_{C1} – Curie temperatures during heating and cooling (T') of the sample; κ_T/κ_{Tmax} – magnetic susceptibility at temperature T, and maximum magnetic susceptibility (κ_{Tmax}). Gr.s. – grain size in μm.



Gr.s.	TiO_2 (%)	FeO (%)
20	22.42	76.64
10	23.77	74.36
8	23.20	75.84
7	24.57	74.26
6	25.63	73.48

Gr.s.	TiO_2 (%)	FeO (%)
60	24.63	73.23
10	23.97	74.34
8	23.83	74.44
6	23.31	73.79
6	24.60	73.26

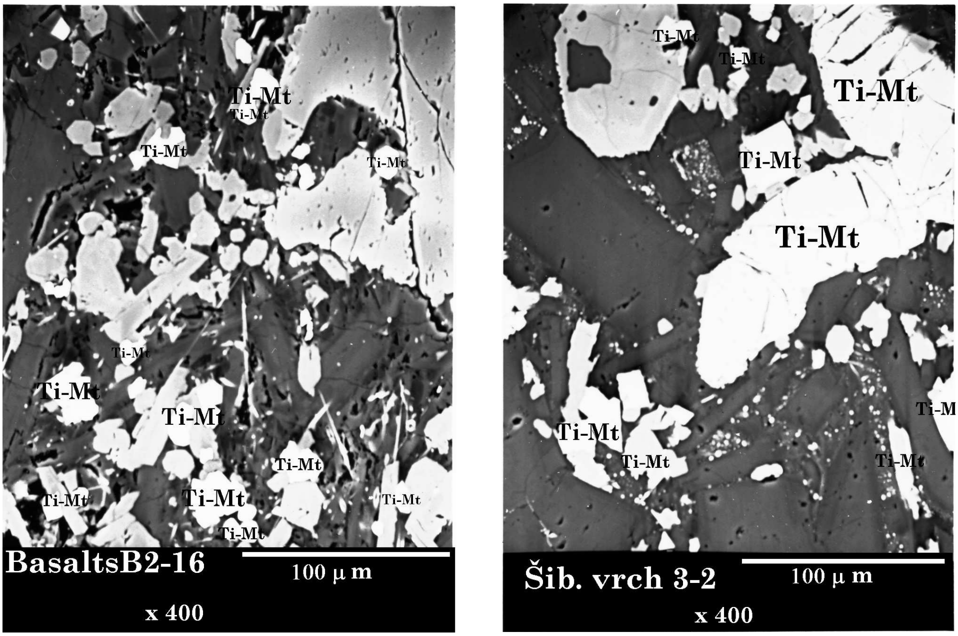
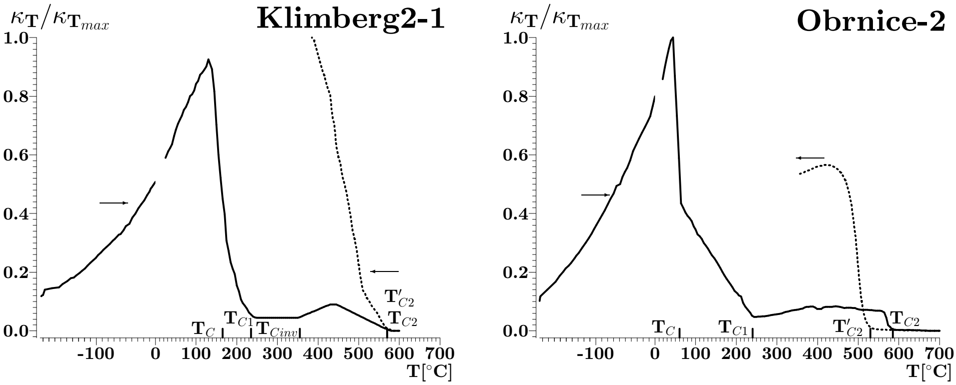


Fig. 3. Thermomagnetic curves, the results of microprobe analyses and microphotographs of Fe-Ti grains (mostly of light, or light-dark tint on the image) of samples: Řetouň 8-1 (8-3); Smřečiny 12-2 (12-1); Other explanations see below Fig. 2.



Gr.s.	TiO_2 (%)	FeO (%)
60	20.83	75.36
12	20.38	77.34
10	22.37	75.67
10	20.04	78.13
7	21.17	75.97

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

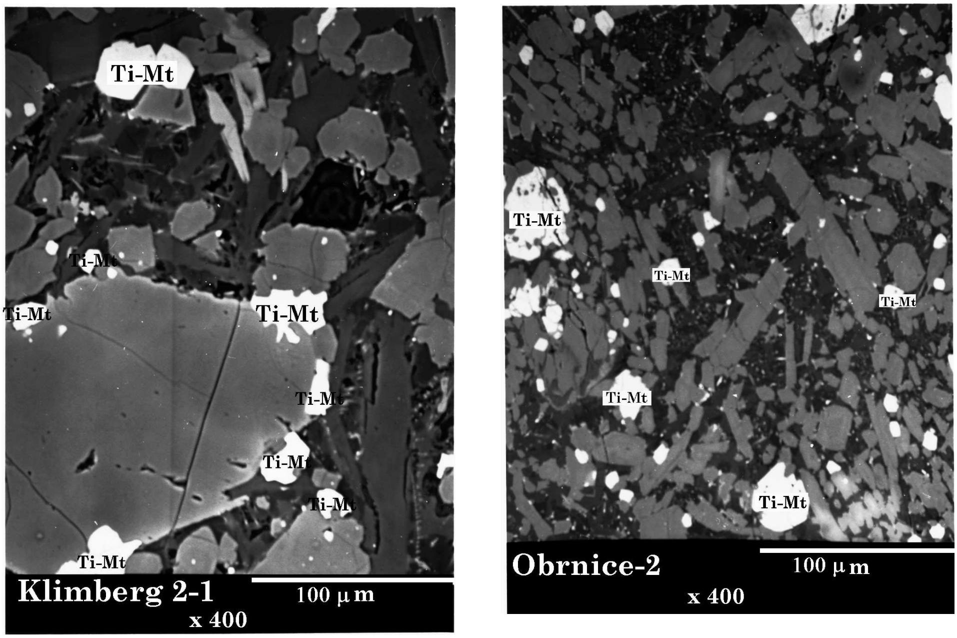


Fig. 4. Thermomagnetic curves, the results of microprobe analyses and microphotographs of Fe-Ti grains (mostly of light, or light-dark tint on the image) of the samples, Klimberg 2-1; Obrnice-2; Other explanations see below Fig. 2.

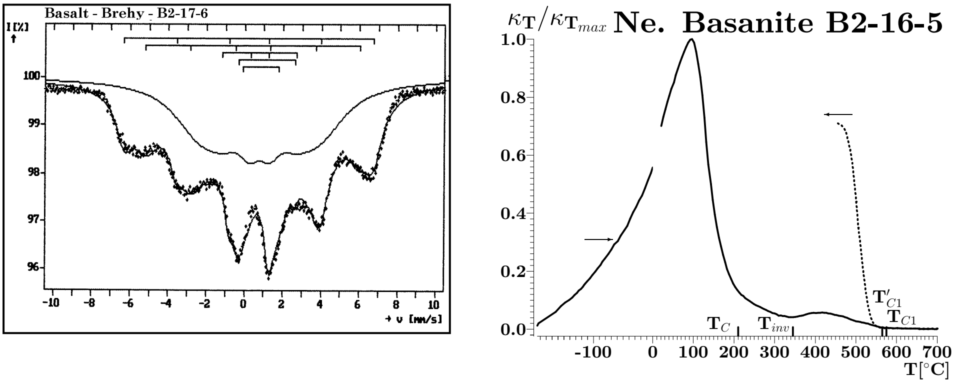


Fig. 5. The Mössbauer spectrum (measured at room temperature) and the thermomagnetic curve of magnetic fraction of the Ti-Mt samples of basalts from Brehy locality.

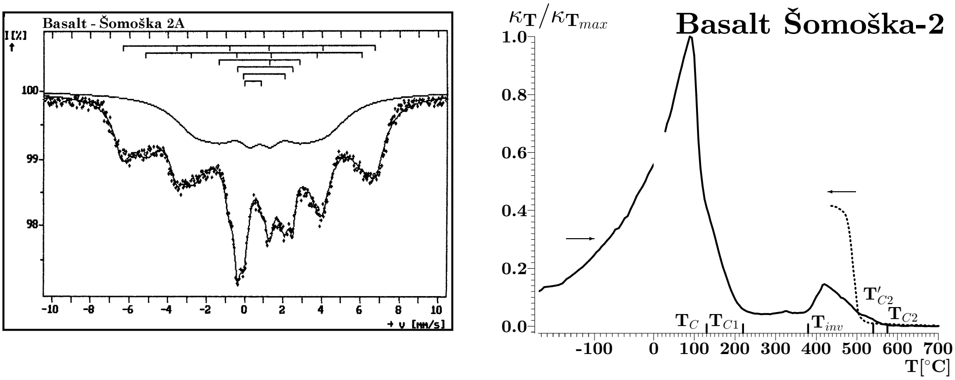


Fig. 6. The Mössbauer spectrum (measured at room temperature) nd the thermomagnetic curve of magnetic fraction of the Ti-Mt samples of basalts from Šomoška locality.

the more oxidized magnetic phases with the presence of the so called *inversion low-temperature oxidation magnetic phase.* Another aspect of the measurements of MS is that simple SP non-interacting single domains are not normally observed in rocks. The properties are modified by grain interactions, which influence the estimates of grain size from the temperature and field dependence. The interpretation of the Mössbauer spectra of additionally studied Fe-Ti oxides of samples from many basaltic rocks from Slovak volcanic fields leads to anticipation that there may be present the Ti-Mt-es with small particles in some basalts. The spectrum of Ti-Mt with

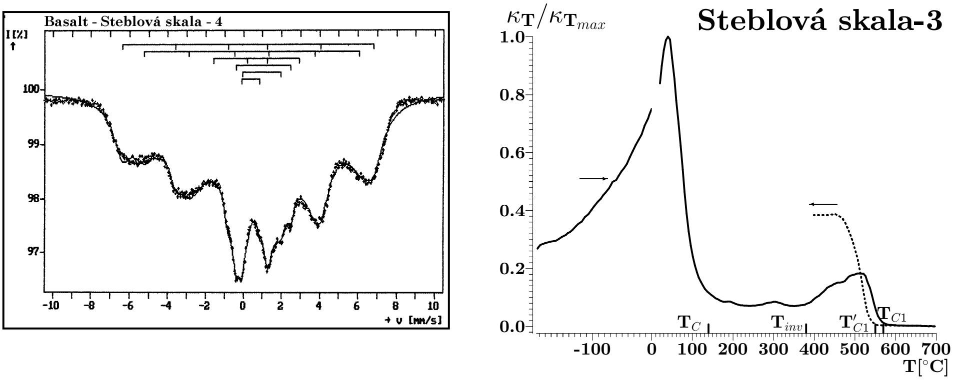


Fig. 7. The Mössbauer spectrum (measured at room temperature) and the thermomagnetic curve of magnetic fraction of the Ti-Mt samples of basalts from Steblová skala locality.

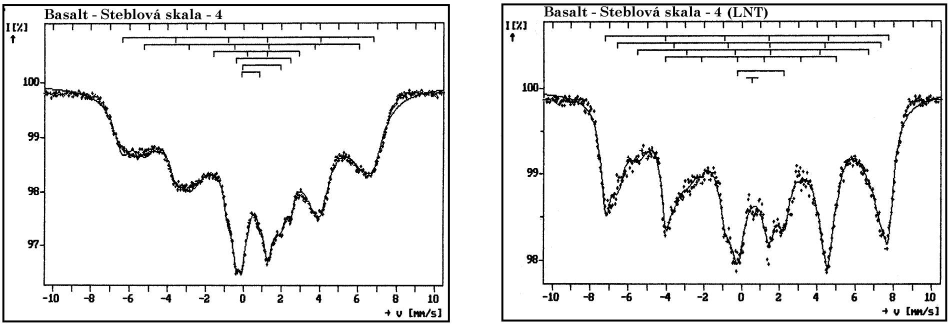


Fig. 7a. The Mössbauer spectra of magnetic fraction of the Ti-Mt samples of basalts from Steblová skala locality. [The spectrum measured at room temperature (left) and at liquid nitrogen temperature – LNT (right)].

high content of ulvöspinel is characterized by dominated paramagnetic doublet.

The detection of a domain structure of TiMt-es by Bitter - pattern method

Work was performed to detect the domain structure of some samples by the Bitter pattern (BP) method and by the Magnetoforce (MF) microscope in Japan (in *Orlický, 2004*). Non expressive, probably ferrimagnetic domain structure of some grains of samples was detected. From many scanning

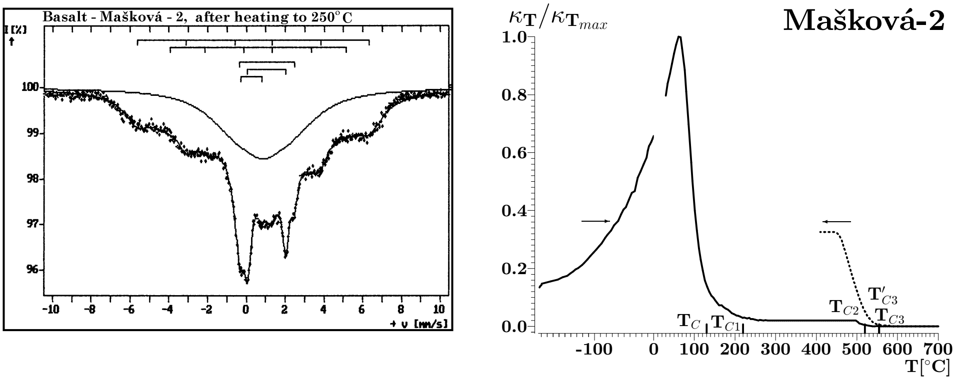


Fig. 8. The Mössbauer spectrum (measured after heating of the sample to 250° C and successive cooling to room temperature) and the thermomagnetic curve of magnetic fraction of the Ti-Mt samples of basalts from Mašková locality.

microscopy images of polished basalt samples are evident large enough Ti-Mt grains (cca 4 μm - 300 μm in diameter) with content of TiO_2 = 24-27%. We know that smaller particles cannot be detected by the mentioned method despite their presence in the rocks. According to *Kropáček (1985)* a threshold between single domain and multidomain structure in Ti-Mt is supposed to be of about 5 μm. The threshold between superparamagnetic and single-domain structure e.g. in magnetite is supposed to be of 0.03 to 0.035 μm, according to *Dunlop (1973)*.

Soffel (1977) investigated the domain structure of the Tertiary basalts with optically apparently homogeneous titanomagnetites (Curie temperatures less than 100°C). He revealed that only the smallest grains with a diameter of less than 6 microns were composed entirely of ferrimagnetic material and showed magnetic domains. The larger grains were only partly ferrimagnetic at room temperature, with large parts of paramagnetic state, or dominantly without domain configuration. This knowledge was a surprise to the author. From the theory and the experiment it has been known that in ferromagnetics and ferrimagnetics the establishment of stable domain structure increases with increasing the size of magnetic particle or the magnetic grain itself. The domain structures of natural grains of Ti-Mt-es (T_C about 240° C) were analysed by *Appel et al. (1990)*, using the magneto-optical Kerr effect. The authors have revealed that the domain structure was very complicated. They commented that internal stress in naturally occurring Ti-Mt-es is high and varies strongly in amount and directions within short distances.

According to *Orlický (2004)* basalts containing dominantly the Ti-Mt-es with high content of ulvöspinel (with x ≈ 0.6 - 0.8 in formula $\mathrm{Fe}_{3-\mathrm{x}}\mathrm{Ti}_{\mathrm{x}}\mathrm{O}_4$) posses high values of volume magnetic susceptibility (κ = 40000 to 80000 × 10^{-6} SI units, while basalts with low content of ulvöspinel and high content of magnetite have paradoxically the values of κ about 1/2 of those of the prior types. Such behaviour has been detected not only in the Ti-Mt-es of basalts from Slovak volcanic fields but also in basalts collected from foreign localities. This phenomenon is supposed to be of world-wide validity. We have known that the magnetite is a very strong ferrimagnetic mineral with the spontaneous magnetic moment of 90 e.m.u/g, while the ulvöspinel is a mineral nearly antiferrimagnetic. A theoretical review on the inadequacy of high values of κ of the Ti-Mt-es with high content of ulvöspinel has been published by *Orlický (2004)*.

From the theoretical review it follows that there is no reason for the ulvöspinel rich Ti-Mt-es to have higher magnetic susceptibilities than those of magnetite rich ones. Large enough Fe-Ti grains are present in the mentioned basalts, according to microscopic images. Unfortunately they do not behave as particles with the developed domain structure. Anomalously high magnetic susceptibilities of the Ti-Mt-es with high portion of ulvöspinel should be attributed to the superparamagnetic behaviour.

About superparamagnetism

According to *Nagata (1961)* there have existed the thermal fluctuations of single-domain magnetic particles in magnetic materials. If a single-domain particle is sufficiently small and the temperature is sufficiently high, in the limiting case an accumulation of particles can reach thermal equilibrium in very short time with regard to duration of an experiment. This phenomenon has been named superparamagnetism.

According to *Stacey and Banerjee (1974)* a single domain grain above its blocking temperature has a magnetic moment μ. This undergoes thermal reorientation, but in the presence of a field H has an average alignment determined by the Boltzmann parameter (μH/kT). Since it has no stable remanence but effectively adopts the equilibrium average magnetization corresponding to the ambient value of H/T, its behaviour is paramagnetic in nature. But since μ is much larger than the atomic moments of a normal paramagnetic material the alignment in a field is much stronger, i.e., the susceptibility may be much higher. Hence the use of the term superparamagnetism to describe its properties. Sufficiently small grains may be SP at room temperature and below, and the phenomenon has been recognized in both laboratory materials and rocks. From the theoretical computations it follows that magnetic susceptibility (κ) of SP grains is much greater than the κ of a material with the same concentration of normal ferromagnetic grains. Thus a small concentration of SP particles can affect the κ quite remarkably. Small magnetic particles are quite commonly present in some rocks. But to separate the individual single-domain or Sp particles from the assemblage of all magnetic fraction is practically impossible in practice. What is of great importance, is that simple SP of non-interacting single domains are not normally observed in rocks. The properties are modified by grain interactions, which influence the estimates of grain size from the temperature and field dependence of κ according to *Creer (1961)*.

The idea about the superparamagnetic state of the high content ulvöspinel titanomagnetites can give us a real chance to explain the source of high values of magnetic susceptibilities of these materials. So far, the relevant methods are missing to verify the existence of SP Ti-Mt particles in rocks.

3. Discussion and conclusions

The considerations presented below have involved not only the specific results mentioned above, but they have been set up on the basis of large own experience and the behaviour of the Fe-Ti oxides in large collections of basaltic and andesitic rocks from Slovak volcanic fields and other foreign areas. An exceptional position among of Fe-Ti solid solutions has the ulvöspinel. Except of properties mentioned above, this mineral does not have a specific low temperature transition, but on cooling to temperatures below LNT, lattice distortions set, in which have been variously identified as Jahn-Teller distortion of tetrahedral Fe^{2+} and as the effect of unquenched orbital angular momentum of octahedral Fe^{2+}. The chief effect of this distortion is a phenomenally large magnetic anisotropy. According to *Lindsley (1991)* the ulvöspinel is an inverse spinel with Ti^{4+} and Fe^{2+} randomly filling the octahedral sites and Fe^{2+} filling the tetrahedral sites. This sort of mixing on the octahedral site is sometimes referred to as *valency disorder* inasmuch as the charge coupled substitution $2L^{3+} = M^{4+} + N^{2+}$ occurs, relative to a hypothetical $Fe^{2+}L^{3+}{}_2O_4$ normal spinel. If one assumes that cations do not exchange sites as mixing occurs, then Ti^{4+} should replace octahedral Fe^{3+} and tetrahedral Fe^{2+} should replace tetrahedral Fe^{3+} accros this join. This model of cation occupation was suggested by Akimoto (in *Lindsley, 1991*) but it does not predict saturation moments accurately. Néel and Chevallier (in *Lindsley, 1991*) suggested a model where Ti^{4+} also replaces octahedral Fe^{3+}, but Fe^{2+} substitutes initially for octahedral Fe^{3+} until all sites contain Fe^{2+}, then additional Fe^{2+} substitutes only for tetrahedral Fe^{3+}. This model also fails to predict saturation moments accurately, but in the opposite sense compared to the Akimoto model. New recent studies have confirmed that the distribution was consistent with the Akimoto model. All samples had Ti^{4+} present only on octahedral sites. However, the Akimoto model appears to be inconsistent with the spinel space group $Fd\tilde{3}m$, which requires that all octahedral sites be equivalent. The model sets the tetrahedral and octahedral Fe^{3+} occupations exactly equal, and further, requires that exactly half of the octahedral sites must be occupied by Fe^{2+}. These restrictions are most easily achieved by symmetry -breaking, e.g., lowering the symmetry to produce inequivalent octahedral sites. But *Wechsler et al. (1984)* suggested that crystal chemical constraints alone may be strong enough to produce the Akimoto distribution. The suggested constraints are: (1) Ti^{4+} has a strong octahedral site preference; (2) The octahedral sublattice may not tolerate more than one ($Ti^{4+} + Fe^{3+}$) per formula unit; and (3) that equal numbers of Fe^{3+} and Fe^{2+} may be favoured on the octahedral sites. Constraint (1) is substantiated by many observations. Constraint (3) appears likely due to the tendency for electron delocalization among identical symmetry sites. This delocalization can be expected to stabilize the structure slightly. Constraint (2) is weaker, but might be necessary for localized charge balancing and electrostatic stability.

There has been number of studies details of which can be found in *O'Reilly (1984)* and *Wechsler et al. (1984)*, which broadly describe the problem of finding the cation distribution pathway(s) by which (a) the octahedral B sites, on going from magnetite to ulvöspinel, end up having only Fe^{2+} ions, instead of an equal Fe^{2+}/Fe^{3+} ratio, and (b) the tetrahedral A sites start with one Fe^{3+} ion per molecule in magnetite, but end up with only one Fe^{2+} ion instead. It is suspected that intermediate titanomagnetites may not display linear variation of M_s (at low temperature and high magnetic fields), which would be the sign of mixed Fe^{2+} – Fe^{3+} occupation of A and B sites throughout, but the M_s data to date have remained murky. Neither Mössbauer nor neutron diffraction data have helped distinguish between models because increasing of titanium substitution in magnetite produces varying local site symmetry for Fe ions, causing large values of diffuse scatter or line broadening *(Wechsler et al., 1984; Boysen and Schmidbauer, 1984; Jensen and Shive, 1973).* Even for the same technique, when neutron diffraction is applied to polycrystals, of a critical composition, $Fe_{2.4}Ti_{0.6}O_4$, the ratio of magnetic moment per iron ion (μFe) as seen in the two sublattices turns out to be different for different authors *(Wechsler et al., 1984; Boysen and Schmidbauer, 1984).* A resolution can perhaps come from polarized neutron diffraction and Mössbauer absorption in high fields and low temperature. As stated before, only such experiments provide individual sublattice magnetic moments, and not merely their difference as given by saturation magnetization measurements.

The results have shown the following:

- When the titanomagnetite solid solutions are present with the only one magnetic phase with high content of ulvöspinel (mostly with x ≈ 0.6 to 0.8, and $T_C \approx 80 - 110°$ C), in all cases they possess very high values of volume magnetic susceptibility regardless of a place of origin (attaining $\kappa \approx 40 to 100 \times 10^{-3}$ SI Units).

- A typical feature of these types of Ti-Mt-es is a strong decrease of κ from laboratory temperature (or from their maximum value) down to liquid nitrogen temperature (LNT). No electron-hopping and the so called Verwey transition temperature has existed in such material, despite the magnetite, though in a negligable portion, is present in these Ti-Mt-es.

- An explanation of high values of volume magnetic susceptibility of these types of Ti-Mt-es cannot be realized either on the basis of high volume portion of Ti-Mt-es themselves, or on the basis of highly magnetic material, because an ulvöspinel which is in dominant portion is nearly non-magnetic. We need to find an explanation in strange - superparamagnetic (SP) behaviour of such Ti-Mt-es.

- Neither the Bitter Pattern (BP) method, nor the magneto-forced-microscopy methods have provided an information about the domain structure of large polished grains of Ti-Mt-es, despite the sizes of grains were more then 50 μm in diameter (such large grains of Ti-Mt-es should be of multidomain structure). We can assume that the domain structure in these types of Ti-Mt-es has not been developed.

- The Ti-Mt-es are very frequently present with the two or more magnetic phases in volcanics, namely in basaltic rocks. One phase which is probably of original state and the second one which has been created due to the so called low temperature oxidation (mostly below 350° C) of part of original phase. This second magnetic phase corresponds to a cation-deficient titano-magnetite - titano-maghemite. This second magnetic phase is characterized by the so called inversion temperature. In this second magnetic phase a stable domain structure of multidomain mode has probably developed. We assume that the more oxidized Ti-Mt (titanomaghemite) is in the same Fe-Ti grain as the original Ti-Mt. Both these Fe-Ti magnetic phases are in a contact with each other.

- Experimentally it has been proven that there existed mutual interactions between the SP particles and those with the developed domain structure. These interactions have probably strongly influenced also the behaviour of the activated material during realization of the Mössbauer spectroscopy, and so the Mössbauer spectra of such material are non-Lorentzian, very broad.

- Dominantly the octahedral positions have occurred on the Mössbauer spectra of the original Ti-Mt-es at room temperatures, while the tetrahedral positions are characteristic at LNT for the same sample of the Ti-Mt.

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