

## Engineering of experiment

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**Microscopic diagnostics of magnetic and domain structures in minerals with ferrocolloid** UDC620.186.8

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**Abstract.** Application of the magnetic ferrocolloidal liquids on samples with magnetic and nonmagnetic parts in their composition actually visualizes the structure of magnetic particles, namely: magnetite-ilmenite intergrowths, heterophase decay structures, grain distribution of zones with different magnetization, and under certain conditions it is possible to diagnose domain structure, including the determination of the domain walls dimensions. In this paper there are presented overviews of experimental works on the study of the magnetic microstructure of samples from various collections with magnetic fluids and micrographs of the studied structures with magnetic contrast usage obtained with a ferrocolloid application.

**Keywords:** *ferrocolloid, domain structure, ferrimagnet, magnetic structure*

### Introduction

Domain structure. Domain structure (DS) properties determine the magnetization and reversal magnetization processes. A magnetic domain structure is a union of regions in the magnetic materials magnetic subsystem which make relations between microscopic magnetic characteristics and their macroscopic properties. A magnetic domain structure exists at temperatures below the magnetic phase transition into a magnetically ordered state temperature and in certain intervals of the external magnetic field strength. An equilibrium magnetic domain structure is determined by the minimum of the magnet total energy, the energy of exchange interaction, magnetic anisotropy, magnetostatic and magnetoelastic energies. The type of the magnetic domain structure depends on features of magnetic anisotropy (the number of easy magnetization axes), the orientation of the surfaces bounding the crystal relative to the crystallographic axes, various defects (magnetic and nonmagnetic inclusions, stacking faults, dislocations, etc.) (Vonsovskii, 1971 (a); Vonsovskii, 1971 (b); Kandaurova et al., 1977; Tikazumi, 1987).

### Methods and results

The concept of magnetic domains in ferromagnets was introduced by P. Weiss in 1907. The magnetostatic energy decreases with a domain structure forming when magnetostatic poles are split on the sample surfaces and the magnetic flux is completely closed inside a ferromagnet. A magnetic domain structure appears only when the energy spent on its formation is less than the loss of magnetostatic energy. A single-domain state is realized in crystals

of small sizes, smaller than the single-domain size  $r_c$ . The short-range exchange interaction plays a more important role than the long-range magnetostatic one at distances  $r < r_c$ . This causes the impossibility of DS formation in crystals with sizes  $r < r_c$ . The domains size in ferromagnets is  $10^{-4}$ - $10^{-2}$  cm.

Changes in temperature, voltage, and magnetic fields provides a great influence on the magnetic DS structure in ferromagnets. Heating and subsequent cooling of samples can lead to changes in their crystal structure and magnetic domain structure. Unlike ferromagnets, there are several magnetic sublattices in ferrimagnets and antiferromagnets. Due to the presence of exchange interaction between sublattices the resulting magnetization practically does not change in magnetic fields up to 10–100 kOe in ferrimagnets. According to this the magnetic properties of ferrimagnets, in particular the formation of a magnetic domain structure, are usually identical to the properties of ferromagnets. In strong magnetic fields (of the order of 100 kOe) the orientation of sublattices magnetizations relative to each other can be changed: for example, the collinear position can become noncollinear.

There are no magnetostatic poles on surfaces in antiferromagnets but a magnetic domain structure is present in them. Adjacent domains in antiferromagnets can differ in the antiferromagnetism vector direction and in the strain tensor principal axes direction. The twin magnetic domain structure formation during antiferromagnet deformation occurs at temperatures below the Néel point. The reasons of the magnetic domain structure formation in antiferromagnets are not fully understood. Magnetic DS is experimentally observed using the magnetic suspension method, methods based on the Kerr effect and the Faraday effect, electron microscopic methods, magnetic neutron diffraction method, etc.

There are a huge number of DS configurations in magnetic crystals and amorphous magnets. The DS depends on many factors: the shape, size, and crystallographic orientation of the samples in one and the same magnetic material. The scientific interest lies in finding their common features in this huge variation of the DS configurations and calculating the quantitative parameters of the DS depending on the fundamental magnetic properties (saturation magnetization, anisotropy, exchange) and geometric characteristics (size, shape, orientation of the bounding surfaces). The observed relationship allows us to show the basic patterns of the DS behavior under external influences: field, temperature, mechanical stresses, etc. This allows the construction of the lowest energy DS model (Hubert et al., 1998).

**Akulov-Bitter powder figure method.**

During the first observations of DS the Akulov-Bitter powder figure method (magnetic suspension) was applied, which confirmed DS existence. The Akulov-Bitter powder figure method consists of applying a magnetic suspension (single-domain particles of magnetite  $\text{Fe}_3\text{O}_4$  suspended in a liquid) onto a well-polished surface of a magnetic material. These particles are attracted to domain boundaries regions because the greatest field gradient is near the edge of the domains. And domain boundaries are shown. It is important to carefully prepare surfaces during observations with a magnetic suspension. One of the first methods for visualizing a magnetic field is the method of "magnetic filings" and magnetic suspension. This imaging method utilizes small (5-20 nm) magnetic particles dispersed in various media (magnetic fluids or suspensions). Due to the concentration of these particles in areas with the larger magnetic field gradients and optical anisotropy forming under the magnetic field influence the optical contrast is achieved.

#### Practical application of ferrocolloid in scientifically significant samples

All micrographs were obtained with an Olympus BX51M microscope at the Borok Geophysical Observatory, Institute of Physics of the Earth, Russian Academy of Sciences.

A) magnetite-ilmenite intergrowths. The micrographs (Fig. 1a, b) show large magnetite grains (dark background of the grain) with ilmenite intergrowths (light background).

B) structures of heterophase decay. The micrographs (Fig. 2) show titanomagnetite grains with both the initial stage of heterophase oxidation (the ilmenite appearance along the crack, along the grain edge, rare accumulations under the influence of external factors), and well-formed lamellas of the

$\text{FeTiO}_3$  composition. It is possible to determine the regions of cationic diffusion on titanomagnetite grains according to these photographs. It is easy to estimate the width of the ilmenite lamellae without using another high-tech equipment.

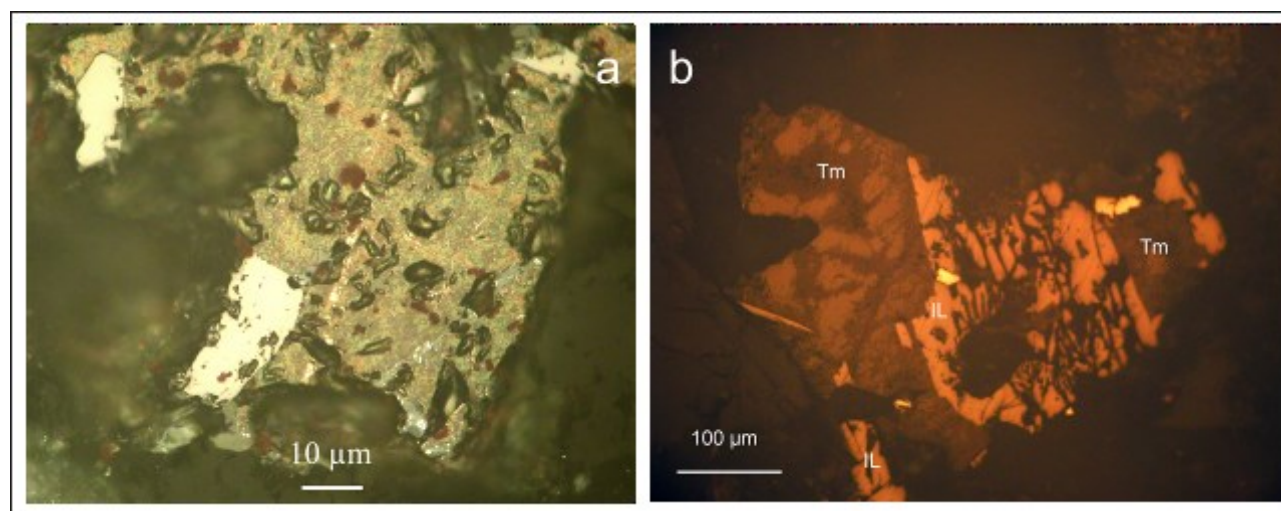
C) distribution of zones with different magnetization in the grain. Due to the ferrocolloid a well-visualized color contrast is obtained on the investigating sample. It is possible to evaluate the zones with different magnetization on the magnetic grain according to this image. The darker color shows stronger in magnetic power magnetite zones. The light ones shows the magnetite with substituted by impurities cation vacancies and reduced magnetization and a nonmagnetic ilmenite.

#### Conclusions

In the course of experimental work of ferrocolloid application on various previously carefully prepared and polished with micron grain size pastes and diamond suspensions natural and synthetic objects scientifically significant micrographs were obtained. In which you can see various structures, such as:

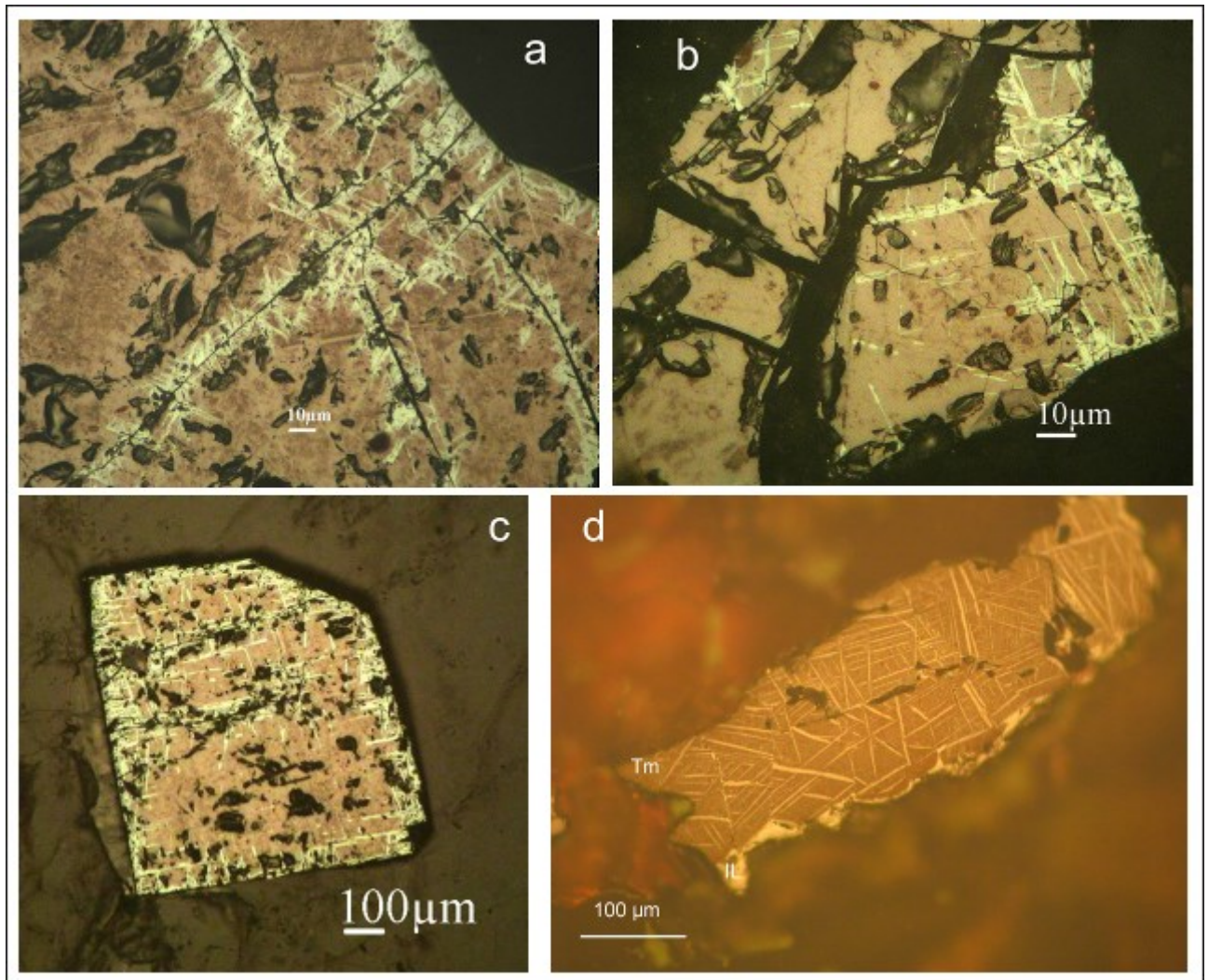
1. Magnetite-ilmenite intergrowths (Fig. 1);
2. Structures of heterophase decay (Fig. 2);
3. The initial stage of HFO along the cracks of the TM grain (Fig. 2a);
4. Granular distribution of zones with different magnetization (Fig. 3b);
5. DS in titanomagnetite grains from basalt (Fig. 4) and on the surface of synthetic titanomagnetite (Fig. 5).

This method has proven itself well and has such advantages as: relatively simple technology, lack of large financial costs, adaptability to field tests, clarity of results, speed of obtaining results.

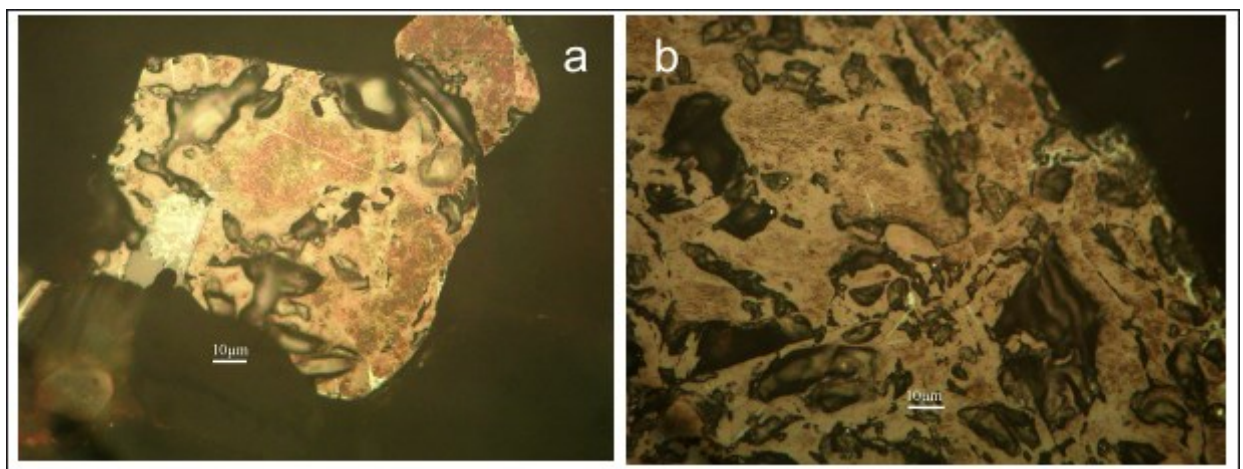


**Fig. 1.** Intergrowths of titanomagnetite and ilmenite

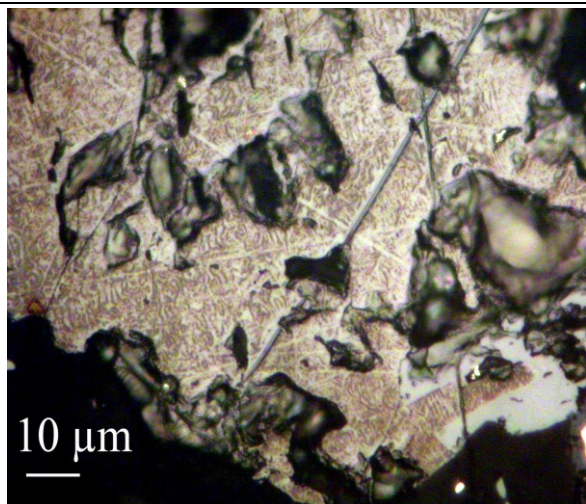




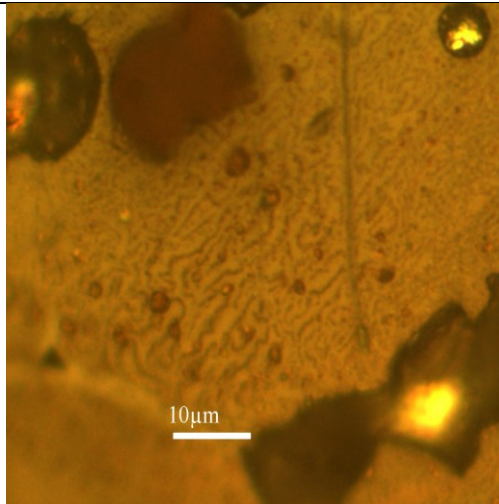
**Fig.2.** The initial stage of HFO along the cracks of the TM grain a) a fragment of the TM grain; b) grain, oxidized on one side; c) grain with the initial stage of HFO on all edges; d) evenly HFO grain.



**Fig. 3** Grains with the initial stage of HFO, inhomogeneous magnetization and visible domain structure



**Fig. 4.** Domain structure of titanomagnetite grains from basalt.



**Fig. 5.** Domain structure on synthetic titanomagnetite.

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