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ELIMINATION OF FERROMAGNETIC PARTICLES AGGREGATION FOR INVESTIGATION BY ELECTRON MICROSCOPY

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It has been described the device for sample preparation of highly dispersed ferromagnetic powders including micropowders for permanent magnets, magnetic carriers, machine and mechanism components' wear products contained in lubricants for investigation of these materials by light and electron microscopy. The device eliminates the coalescence of ferromagnetic particles and improves reliability of the results of such objects investigation. The technique of such device application has been described and exemplified for various materials investigation.

Keywords: *ELECTRON MICROSCOPY, FERROMAGNETIC PARTICLES, AGGREGATION, ULTRASOUND, MAGNETIC FIELD.*

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Highly dispersed metals and their alloys are widely used in various fields of science, engineering and technology [1]. The most important technical characteristics of ferromagnetic powder are particle size and form which are usually monitored by electron microscopy [2]. The reliability of the results obtained by this method is defined mainly by the quality of prepared samples which could lack because of the aggregation of powder particles owing to magnetic interaction between them [3]. The formation of powder clumps complicates the interpretation of optical and electron microscopic images since under these conditions the form and structure of individual particles as well as their size and positional relationship of their structure elements are masked. The indicated difficulties could be eliminated when the prepared sample is simultaneously exposed with a magnetic field and ultrasound. At this, the repulsive forces between the analogous poles of magnetized powder particles as well as the effect of reduction of particles cohesion in ultrasonic field are used [4].

In practice, this exposure is realized using the device combined with ultrasonic disperser "UZDN-2T" or "UZDN-A" [5]. Constructively this device (Fig. 1) represents the cone-shaped nozzle for disperser irradiator. The nozzle material is stainless steel. At the one end of the nozzle 1 the axisymmetric magnet 3 (samarium-cobalt alloy) is fixed on shock-absorbing gaskets 2. The magnet is oriented in such way that force lines of magnetic induction are perpendicular to nozzle working butt. With the other end the nozzle is fixed on the irradiator bloc of a disperser. The nozzle size is chosen in order the nozzle with working disperser irradiator to be mechanical resonant system at ultrasonic vibrations frequency. The ring cavity at the nozzle head serves for the mounting of non-magnetic material-based spring blank holder which fixes the film carrier at the nozzle working surface. Permanent magnet located in the nozzle generates the magnetic induction B, whose value changes depending on the distance h to nozzle butt in the way as it is shown in Fig. 2.

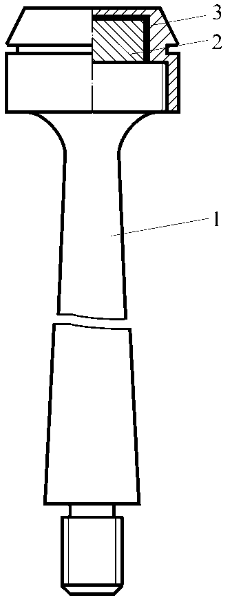

Fig. 1 *– The device scheme: 1 – nozzle; 2 – shock-absorbing gaskets; 3 – magnet*

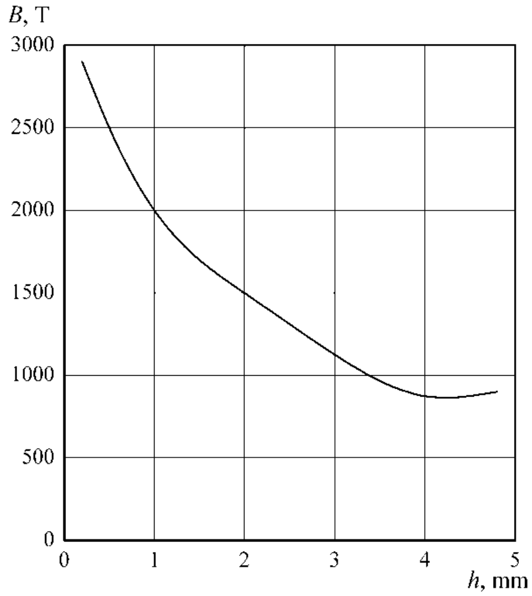

Fig. 2 *– The diagram of magnetic induction change at the nozzle butt*

To use the device, in the centre of irradiating nozzle surface (Fig. 3) the film carrier 2 is fixed in the operating zone of uniform magnetic field from the magnet 1 using the spring blank holder. Magnetic induction at the film carrier site is chosen in the way the ferromagnetic substance under study to magnetize until saturation. After that, ferromagnetic powder or its suspension in volatile liquid is applied onto film carrier. Under exposure with a magnetic field generated by the magnet 1 the magnetic poles appear at the ends of ferromagnetic particles 3. These poles generate the magnetic field partially closed up beyond the particle. Under these conditions the repulsive forces between analogous poles of particles as well as attractive forces between the opposite poles of particles and permanent magnet occur. The friction forces between particles also take place. Under the exposure with this system of forces the particles of ferromagnetic powder form the chains (Fig. 3 a) oriented along the force lines of external magnetic field. When ultrasonic vibrations are superposed at film carrier the friction forces between particles decrease and these particles are directed towards the magnet and simultaneously repel from each other. At this, particles "spread" along the film carrier and are situated separately from each other (Fig. 3 b). The particles excess is piled up at the edge of nozzle working butt in the operating zone of non-uniform magnetic field.

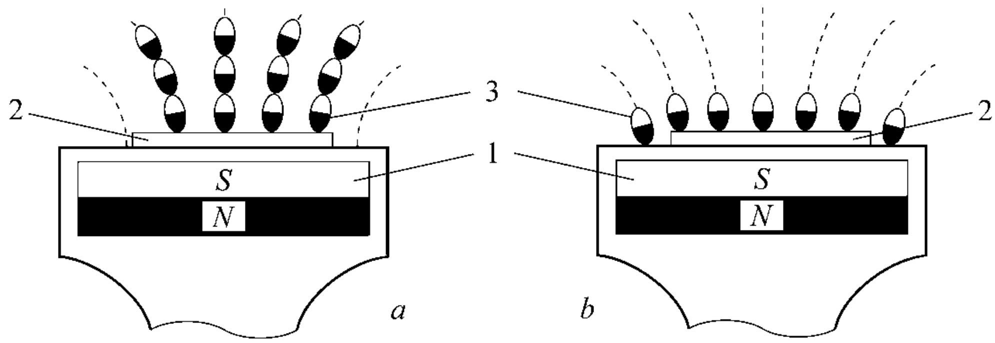

Fig. 3 *– Arrangement of powder particles in the field of permanent magnet: a – without ultrasound; b – under ultrasonic exposure*

In order to provide long-term operation of the nozzle we'll indicate some peculiarities of its usage technique. The nozzle is placed onto the irradiator bloc of a disperser in the position of working surface upwards. For sample preparation the film carrier is fixed in the centre of nozzle butt. At this, the thin rubber gasket (~ 1mm diameter) is placed between the spike of spring blank holder and film carrier. Then ferromagnetic powder is placed onto the film carrier in the way to cover its surface. After that one switches on an ultrasonic disperser preliminary adjusted in resonance with the irradiator, and, gradually increasing the power supplying the irradiator, achieves powder "spreading" along the nozzle butt whereupon a disperser should be switched off. Then the carrier has to be taken off and the particles should be fixed on it via the carbon film sputtering. By this the reduction of a microscope objective lens contamination with ferromagnetic particles is achieved. The prepared sample is ready for investigation.

During every manipulation with the film carrier it is recommended to use the non-magnetic tweezers. The rubber gasket and spike of spring blank holder should be placed closer to the edge of the film carrier. The moment of powder "spreading" along the nozzle butt is a criterion of sample readiness. It may come at various values of power supplying the nozzle and depends on the friction factor between particles of the magnetic powder under study. With an increase of practical skills one may fix the film carrier with a non-magnetic needle instead of the spring blank holder using. Either the collodion support fixed on supporting grid or mica plate may serve as film carrier. The latter may be used for the preparation of magnetic powder suspensions in volatile liquid. It is recommended to use freshly-made collodion film.

The device allows one to shade the prototype system situated in orienting magnetic field orthogonal to the carrier plane. For this purpose the nozzle along with the fixed film carrier after particles distribution are taken off from the disperser irradiator and placed into a vacuum sputtering installation where the shading with some heavy metal is performed. It's not recommended to operate the device in air over a period of more than 10 seconds. The samples of the device usage for investigation of various materials are given in Fig. 4. The images show that aggregation of ferromagnetic particles in the samples prepared by the described above technique is absent. This improves the accuracy of particle size estimation and increases the amount of information obtained from unit area of the image.

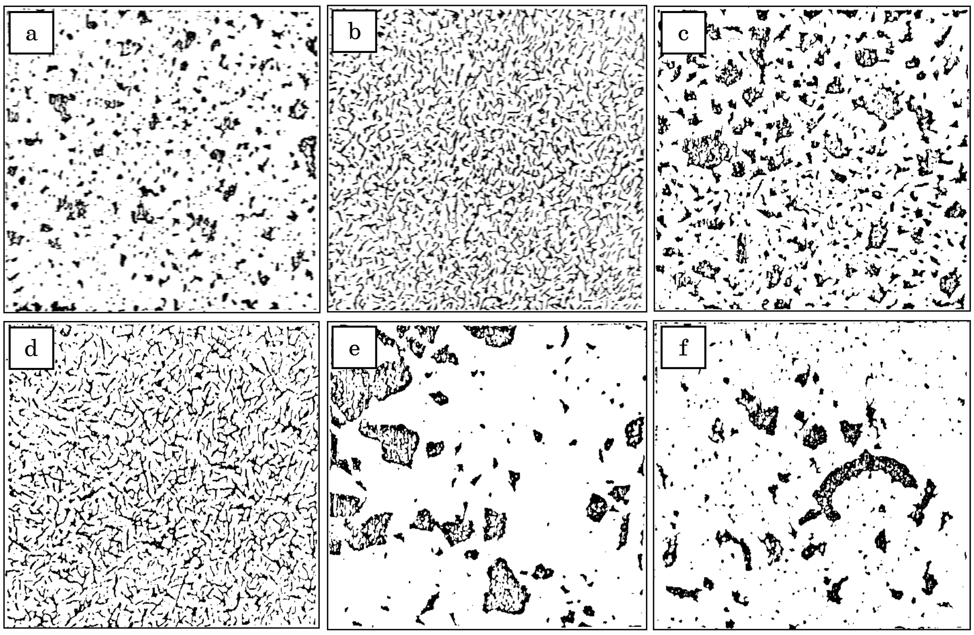

Fig. 4 *– The micrographs of highly dispersed powders: a – nickel (× 15000); b – iron particles obtained via electrolysis (× 10000); c – barium ferrite (× 5000); d – iron oxide (× 10000); e – samarium-cobalt alloy (× 10000); f – machine and mechanism components' wear products, lubricant extract (× 10000)*

Thus, the device could be used to study the properties of ferromagnetic powders as well as for the investigations in chemistry, biology and medicine aiming to provide simultaneous expose of objects and processes with ultrasound and magnetic field.

REFERENCES

1. E.P. Kotov, M.I. Rudenko, *Tapes and Disks in Magnetic Recording Devices* (Moscow: Radio isvyaz': 1986. – In Russian).
2. P.J. Goodhew, J. Humphreys, R. Beanland, *Electron Microscopy and Analysis* (London-NY: Taylor & Francis: 2001).
3. F. Vainberg (ed.), *Devices and Methods of Physical Metallurgy*. Iss. 2 (Moscow.: Mir: 1974. – In Russian).
4. A.K. Dudchenko, A.S. Kuzema, I.E. Grin'ko, N.S. Lyal'ko, A.C. USSR N 1033904, G01N1/28, 1983. Publ. BI, N 29. – In Russian.
5. A.K. Dudchenko, A.S. Kuzema, I.E. Grin'ko, *Priboryi Tekhnika Eksperimenta*. **1**, 239 (1984).