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## THE INFLUENCE OF THE TEMPERATURE OF LIQUID NITROGEN ON THE PHYSICAL PROPERTIES OF POWDER MAGNETIC COMPOSITES

### WPLYW TEMPERATURY CIEKŁEGO AZOTU NA WŁAŚCIWOŚCI FIZYCZNE KOMPOZYTÓW MAGNETYCZNYCH PROSZKÓW

The paper presents the physical properties of soft magnetic iron composites and Nd-Fe-B bonded permanent magnets measured at room temperature and at liquid nitrogen. The objective of research was a determination of influence of liquid nitrogen temperature on the magnetic properties, resistivity and mechanical properties of different powder magnetic materials. Research was carried out for three powder materials: soft magnetic, i.e. Somaloy 700, AncorLam and hard magnetic powder MQP-B used for production of bonded magnets. Composite specimens were prepared by compression moulding technology.

*Keywords:* powder metallurgy, cryogenic condition, soft magnetic composites, hard magnetic composites

### 1. Introduction

Liquefied gases, such as liquid nitrogen and liquid helium, are used in many cryogenic applications, like - cooling of superconducting magnets or supercomputer, and in advanced chemical processes. Electric drive pumps operating in liquid gas atmospheres, may be applied in devices for transport and storage of liquids: nitrogen, hydrogen or oxygen. For example, they can be used in advanced cars or spaceships. It is a reason that demand for electric drives working in cryogenic temperatures permanently growth [1].

Soft and hard magnetic powder composites have many advantages: possibility of tailoring physical properties of elements due to requirement of designers of electric motors, they are inexpensive and, they have three-dimensional distribution of magnetic flux, possibility of execution of complex magnetic circuit structures, which are impossible to be made by traditional material. This type of magnetic composites can be successfully applied in electric motors [2].

All materials, including magnetic materials, change physical properties with changes of temperature. Changes of physical properties of magnetic circuits influence the parameters of machines. Thus, it is very important to have knowledge about changes of physical properties with temperature changes and take them into consideration during the design process of electric devices [2]. The influence of cryogenic conditions on the parameters of magnetic materials is weakly described in the literature. The aim of research is to increase knowledge of the behaviour of composite materials in cryogenic conditions.

### 2. Experimental details

Measurements of magnetization curves and total losses vs. frequency curves of composites made of AncorLam and Somaloy 700 iron powders at room temperature and at liquid nitrogen temperature were conducted. Samples of soft magnetic composites were made in the compression moulding technology in Tele and Radio Research Institute (ITR). The first stage of processing is pressing powder in a die, under pressure of 800 MPa. Then a green compacts were cured; the temperature of curing depends on the bonding agent. The curing temperature of Somaloy 700 was 500°C, Ancorlam was cured at 530°C, and MQP-B was cured at 200°C. The samples for research of magnetic properties are ring-shaped, with size of  $\Phi 55 \text{ mm} \times \Phi 45 \text{ mm} \times 5 \text{ mm}$ . Magnetic properties were determined according to IEC 60404-6 on ring specimens at room and liquid nitrogen temperature.

The magnetic properties of the bonded permanent magnets were tested. Magnets used in the study were made of Nd-Fe-B powder and were bonded by epoxy resin type Epidian 100 (2.5% of weight). Samples of permanent magnets were manufactured in ITR from commercially available powder type MQP-B. This permanent magnets had cylindrical shapes: diameter of 10 mm and 4 mm in length. For measurements of magnetic properties of Nd-Fe-B bonded permanent magnets at room and at liquid nitrogen temperatures, hall sensors were used. In this method magnetic sample was placed in the air gap between magnetic poles of the electromagnet. The electromagnet with air gap length 30 mm was used. Such value of air gap allows to use thermally insulated container filled with liquid nitrogen. In this case it is a necessity to change

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only temperatures of magnetic sample. External values such as  $H_0$  and  $B_0$  were measured, while internal values defining magnetic properties of magnetic samples are designated on the basis of equations considering the demagnetization ratio[3].

Transversal rupture strength (TRS) and compressive strength ( $R_c$ ) were measured. Transverse rupture strength, also called bending strength, were measured on samples with dimensions of  $30 \times 12 \times 6$  mm (Fig. 1). Instron machine type 1115 was modified and mechanical measurements at liquid nitrogen temperature were possible (Fig. 2).

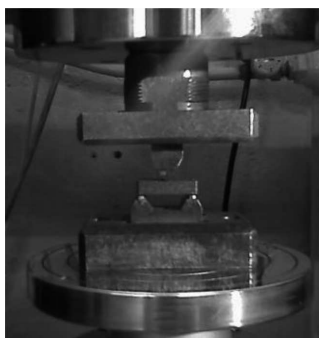


Fig. 1. Sample and sample holder for testing TRS

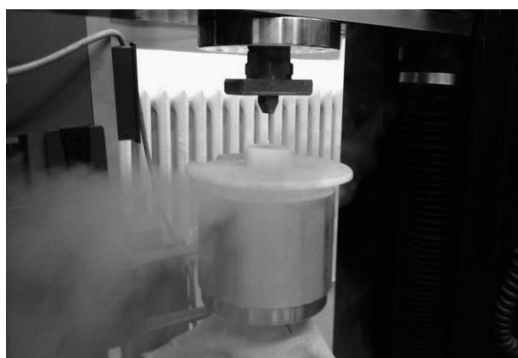


Fig. 2. Container with liquid nitrogen installed in a machine INSTRON 1115

Resistivity of soft magnetic composites were also tested. The resistivity was determined by 4 electrode wires method. The sample and the measurement electrodes during the measurements were immersed in liquid nitrogen. In the four-point method, two electrodes were connected to surfaces side of the sample, and they are current  $I_z$  terminals. Another two measuring terminals positioned at the sample surface, are used to measure the voltage caused by current flow through the sample  $I_z$  (Fig. 3 and Fig. 4). The resistivity is calculated based on the relationship (1):

$$\rho = \frac{S}{l} \cdot \frac{U_p}{I_z} [\Omega m] \quad (1)$$

where: S-cross section area of the sample, l-distance between the measurement electrodes (40 mm). Resistivity measurements were carried out according to the Polish standard PN-C-82055/08: 1990 [4,5].

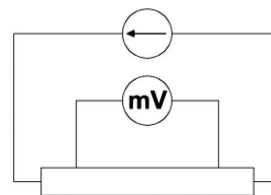


Fig. 3. Diagram of the measuring apparatus

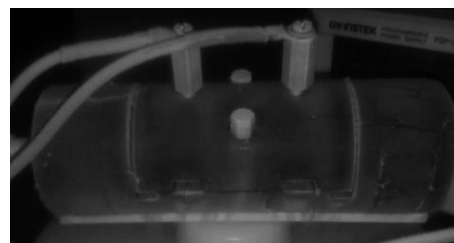


Fig. 4. Handle with measurement electrodes when taken out of liquid nitrogen

### 3. Results and discussion

Figure 5 and 6 shows DC magnetization curves of composites made of Somaloy 700 and AncorLam powders.

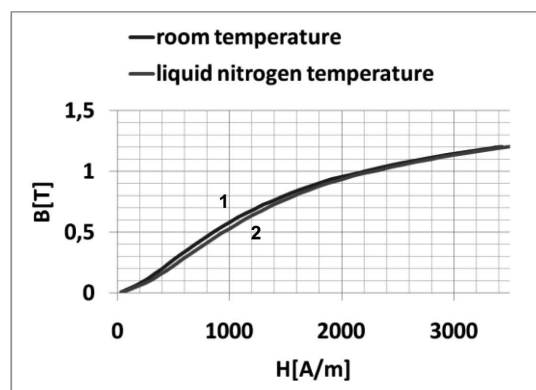


Fig. 5. Magnetization curves characteristics of Somaloy 700 at room (1) and liquid nitrogen (2) temperatures

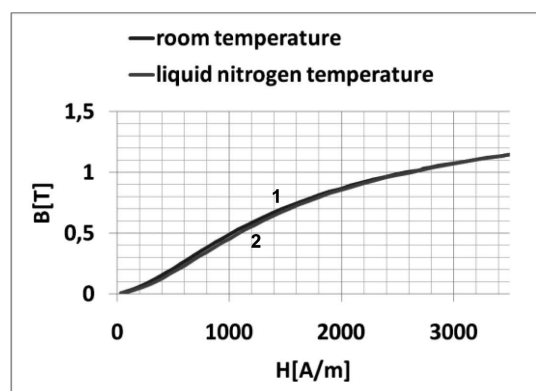


Fig. 6. Magnetization curves characteristics of AncorLam at room (1) and liquid nitrogen (2) temperatures

For both composites made from powders the magnetization curves are very similar in shape at room and liquid nitrogen temperature. Up to 2000 A/m, the flux density of cores made of SMC powders is insignificantly higher at room

temperature compared to the flux at liquid nitrogen temperature. This fact leads to higher relative permeability at room temperature.

Figures 7 and 8 show that decrease of temperature of the sample results in a significant increase in the total power losses in the magnetic circuit. Increase of total power losses is connected mainly with reduction of atom vibrations in crystal lattice. The scope of movements of domain walls is limited. The magnetocrystalline anisotropy will increase at lower temperatures, meaning, that a higher field is needed to reach a given magnetization level. A consequence of this phenomenon is an increase in magnetization energy losses [6].

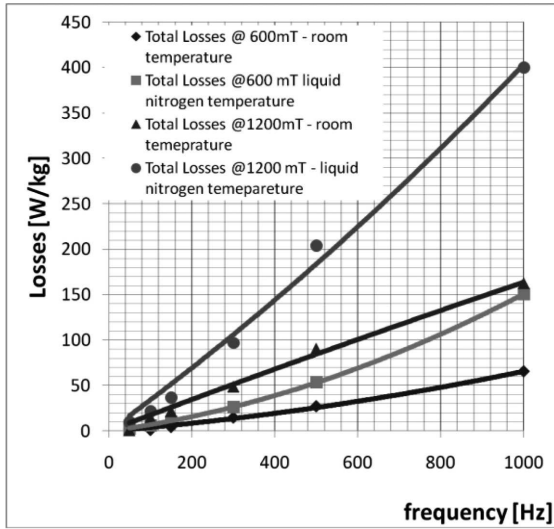


Fig. 7. Losses vs. frequency characteristics of Somaloy 700 at room and liquid nitrogen temperatures

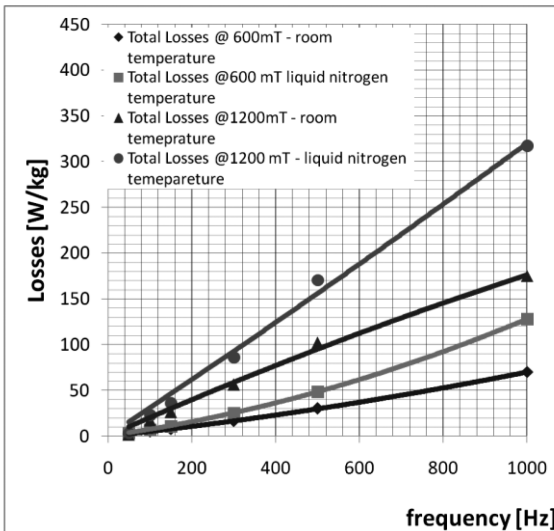


Fig. 8. Losses vs. frequency characteristics of AncorLam at room and liquid nitrogen temperatures

Magnetic properties of bonded permanent magnets were measured. Results of measurements of magnetic properties of Nd-Fe-B hard magnetic composites made of MQP-B powder are summarized in Table 1.

According to results presented in Table 1 decreasing temperature caused significant increase of remanence and coercivity of polarization. As in the case of soft magnetic material

scope of movements of the domain walls is limited. However, this affects the improvement of magnetic properties of the composite. Thanks to this, material is more difficult to magnetize, which increases the coactivity and remanence.

TABLE 1  
Magnetic properties of Nd-Fe-B hard magnetic composites at room and liquid nitrogen temperature

| Temperature                            | $B_r$ [T] | $H_{cJ}$ [kA/m] | $H_{cB}$ [kA/m] |
|--|-----------|-----------------|-----------------|
| Room temperature (20°C)                | 0.672     | 721             | 442             |
| Liquid nitrogen temperature (-195.8°C) | 0.921     | 1323            | 446             |

In many applications the mechanical properties are also very important. It is the reason that mechanical properties of hard and soft magnetic composites were determined. Results are shown on Figure 9 and 10.

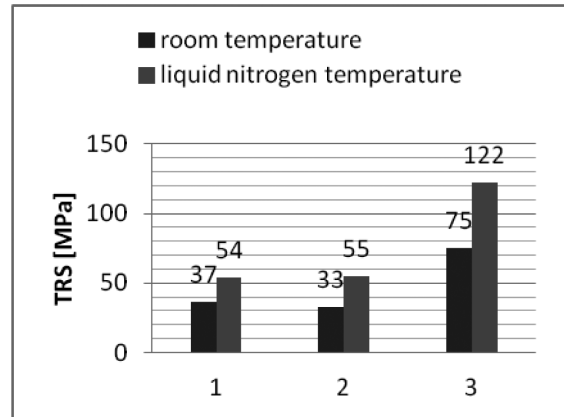


Fig. 9. Transversal rupture strength of composite materials at room and liquid nitrogen temperatures: 1- Somaloy 700; 2-AncorLam; 3-MQP-B +2,5% Epidian 100

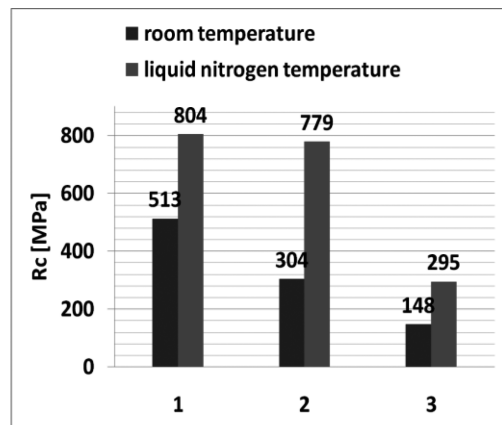


Fig. 10. Compressive strength of composite materials at room and liquid nitrogen temperatures: 1- Somaloy 700; 2-AncorLam; 3-MQP-B +2,5% Epidian 100

Mechanical tests showed a very large influence of liquid nitrogen temperatures on the strength of the magnetic composites. Low temperature causes the significant increase in strength for both type magnetic materials.

The resistivity of soft magnetic composites was also measured. Results are shown in Table 2.

Resistivity of the composite metal – epoxy resin is dependent on the resistivity of the resin and metal. The main factor which influences the resistivity is the resin content. The more resin the higher resistivity a composite. The epoxy resin are a dielectric, the resistivity is practically independent of temperature. Resistivity of soft magnetic composites that consist of iron powder and resin decreases at liquid nitrogen temperature less than resistivity of pure iron. Change in resistivity is very small. This is due to large volume of resin amount in the sample. The resin does not change resistivity as a function of temperature, and therefore low temperature does not substantially affect on resistivity of the composite.

TABLE 2

Resistivity of the SMC at room and liquid nitrogen temperature

| Temperature                            | Resistivity [ $\mu\Omega\text{m}$ ] |          |
|--|-------------------------------------|----------|
|  | Somaloy 700                         | AncorLam |
| Room temperature (20°C)                | 370,7                               | 496,8    |
| Liquid nitrogen temperature (-195.8°C) | 324,6                               | 375,9    |

#### 4. Conclusion

The paper presents the results of research of influence of temperature of liquid nitrogen on the physical properties of soft magnetic composites and bonded permanent magnets. Magnetization curves of soft magnetic materials changes in small range, but total power losses increase. This effect is related to increase of so-called the inter particle eddy-currents and reduction of atom vibrations in crystal lattice. Research shown that magnetic properties of permanent magnets made of Nd-Fe-B powder with decreasing has temperature increase. As in the case of soft magnetic materials less atom vibration, influences on increases the value of the field required for magnetization and demagnetization. What resulting in increased coercivity and remanence of the magnet. In the case

of hard magnetic materials results in an increase of coercivity. Mechanical properties of both types of magnetic elements increases at liquid nitrogen temperature. Small changes in the resistivity of the soft magnetic composites can be a great advantage of this type of materials.

The results could be used by designers of electric motors for operation in liquid gases [7-9].

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