

Microstructure and physical properties of nanocrystalline $\text{Fe}_{75}\text{B}_{10}\text{Si}_{10}\text{Cr}_5$ alloy

**Dominika Baran, Natalia Czyżewska, Szymon Dziuba,
Julia Przenajkowska, Mateusz Piesiak, Mariusz Hasiak**

*Department of Mechanics, Materials Science and Engineering,
Wrocław University of Science and Technology, 25 Smoluchowskiego St., 50-370 Wrocław*

Abstract: *The main of this paper is to study the microstructure and physical properties of the $\text{Fe}_{75}\text{B}_{10}\text{Si}_{10}\text{Cr}_5$ in condition after being produced. The sample was prepared as cylindrical rod with a diameter of 3 mm. The rod was produced using the arc melting method with a suction casting option. It was found that produced alloy was nanocrystalline with average grains of about 15 nm. Due to the different cooling rates in the inner and outer part of the rod, variable sizes of dendrites in the material were observed. The presence of the dendrites was also confirmed on the surface by AFM investigations. The average roughness calculated for chosen profile of the examined area was 6 nm and average hardness of the material equals 844 HV. The mechanical properties were determined in respect to the Oliver-Pharr method. Young's modulus of the $\text{Fe}_{75}\text{B}_{10}\text{Si}_{10}\text{Cr}_5$ calculated from load-displacement curve equals 128 GPa. Magnetic properties of the sample were measured by the vibrating sample magnetometer. The coercivity was equaled 2.184 kA/m, which is relatively low. It means that the $\text{Fe}_{75}\text{B}_{10}\text{Si}_{10}\text{Cr}_5$ alloy is magnetically soft.*

Keywords: *suction casting, nanocrystalline alloy, mechanical properties*

1. Introduction

Fe-based alloys produced with melt spinning method exhibit exceptional properties, both mechanical and magnetic [1]. These alloys are usually produced in the form of thin strips and rods. Due to the high cooling rate, it is possible to obtain alloys in the amorphous form. The nanocrystalline structure is achieved with isothermal heating of the amorphous precursor. Partially crystallized Fe-based alloys exhibit unique combination of the characteristic properties for soft magnetic materials i.e. low coercive field, high induction with saturation magnetization [1]. The addition of such elements as chromium, titanium, manganese or molybdenum to Fe-based alloys leads to improvement of magnetic and mechanical properties [2-3]. From properties point of view, these materials are used in the electrotechnical industry, to production of cores for application in transformers and chokes [4]. The main motivation to conduct the experiment was to produce and study the specific properties of the nanocrystalline alloy which is used to build the transformer cores.

2. Experimental part

The nanocrystalline $\text{Fe}_{75}\text{B}_{10}\text{Si}_{10}\text{Cr}_5$ alloy was produced using the suction casting technology as a cylindrical rod. In order to identify crystalline phases the X-ray diffraction pattern was recorded by using Rigaku Ultima 4 with the cooper lamp. The XRD sample investigation was performed between 20 and 120 (2θ) angles. The sample was also analyzed with the help of Scanning Electron Microscope HITACHI S-3400N, equipped in SE and BSE detectors and material identification mode, with magnification up to 5000. The topography surface of the sample was analyzed using sicon tip and Park XE100 AFM equipment. Nanoindentation was carried out using Micro Combi

Tester produced by CSM. The measurements were carried out using the Berkovich indenter. The magnetic properties of the material were tested using VSM VersaLab Quantum Design. The measurement was carried out for the sample with mass of 86.45 mg. The magnetic properties were investigated for the maximum external magnetic field of 3 T.

3. Results and discussion

3.1. X-ray Diffraction

XRD analysis was used to identify the crystalline phases and to distinguish the amorphous and crystalline phases in the investigated material. Fig. 1 shows the X-Ray diffraction pattern of the $Fe_{75}B_{10}Si_{10}Cr_5$ alloy. The obtained results correspond to the reflexes of bcc Fe phase. The reflex between angles (2 θ) 40 and 50 is broaden. It suggests that some part of the alloy is amorphous, and other nanocrystalline. No other phases were recognized. The average crystallite size were calculate according to the Scherrer equation using the half width of the peak. The constant value used in the Scherrer's formula was 1. The obtained value of crystallite size is 14.8 nm.

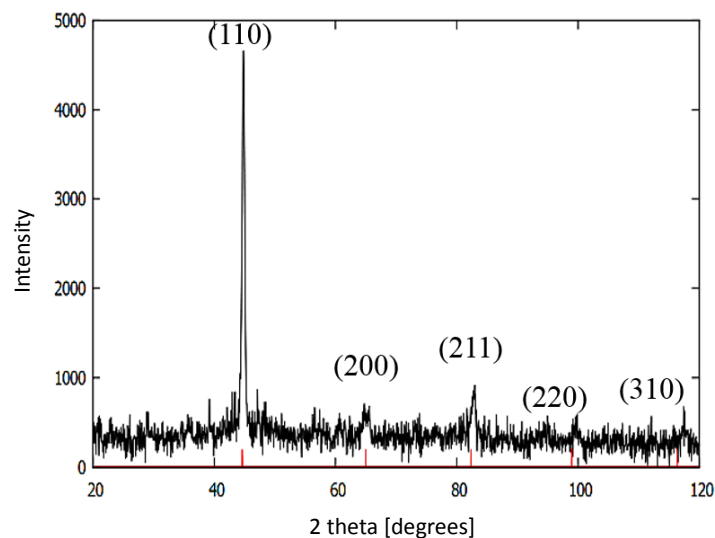


Fig. 1 X-Ray diffraction pattern of the as-quenching $Fe_{75}B_{10}Si_{10}Cr_5$ alloy.

3.2 Scanning Electron Microscopy

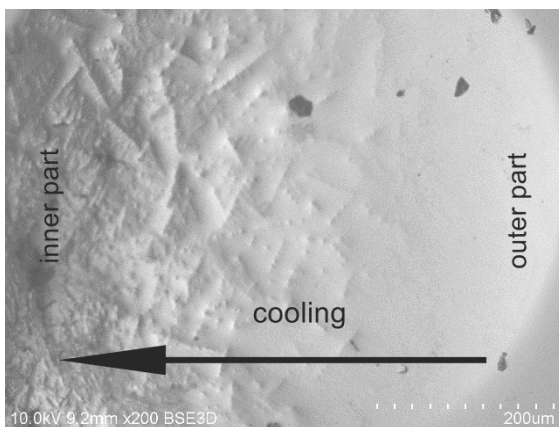


Fig. 2 SEM BSE mode image of the $Fe_{75}B_{10}Si_{10}Cr_5$ alloy with magnification of 200.

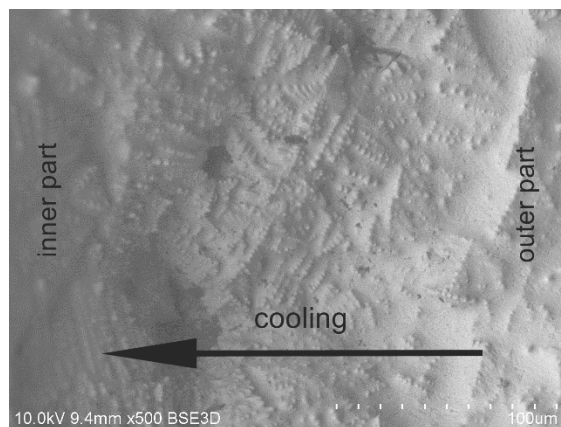


Fig. 3 SEM BSE mode image of the $Fe_{75}B_{10}Si_{10}Cr_5$ alloy with magnification of 500.

The sample was also examined with a SEM microscope with magnification up to 5000 in SE and BSE mode. Polished surface was identified as homogenous. Unpolished surface of shown areas (Figs 2-3) revealed a pattern with recesses, which size and concentration is growing towards to the center of the rode, that is with the cooling direction. The presence of the dendrites was more visible in the inner part of the sample due to the sample's cooling direction.

3.3 Atomic Force Microscope

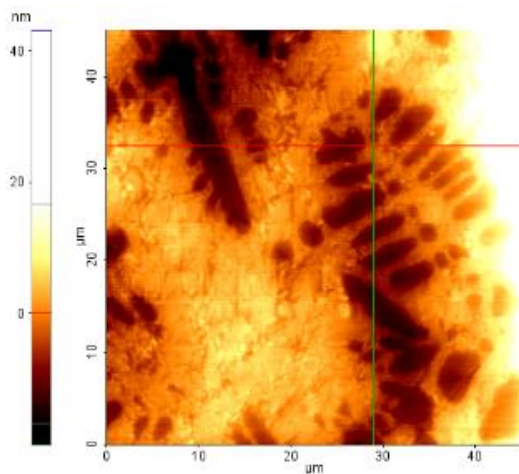


Fig. 4 2D surface topography of the $Fe_{75}B_{10}Si_{10}Cr_5$ alloy for the scanned area of $45 \mu m \times 45 \mu m$.

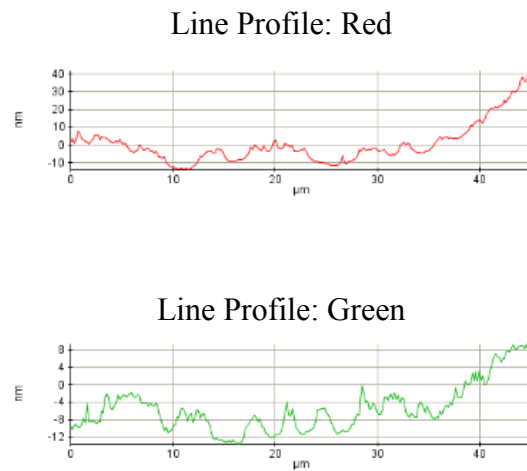


Fig. 5 The profiles (red and green marked in Fig. 4) for $Fe_{75}B_{10}Si_{10}Cr_5$ alloy.

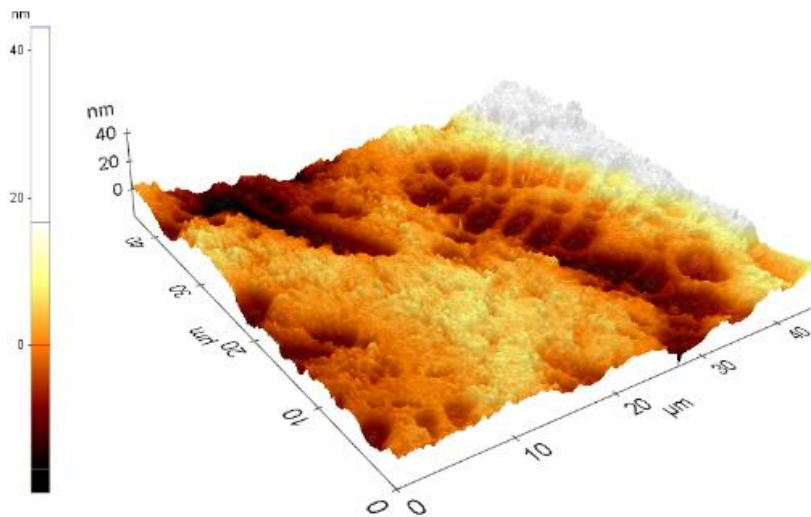


Fig. 6 3D surface topography of the sample.

Figure 4 presents AFM topography image with $45 \mu m \times 45 \mu m$ of the cross section of the produced rod. The average roughness profile (R_a) of the area shown in this figure equaled 6.098 nm, and 10-points mean roughness (R_z) equaled 62.428 nm. However, for the red and green profiles $R_a = 7.536$ nm, $R_z = 38.207$ nm, and $R_a = 4.11$ nm, $R_z = 21.568$ nm, respectively. The maximum height for the red profile was 41.627 nm and for the green profile was 9.181 nm. The minimum height for the red profile is -13.960 nm and for the green profile -13.582 nm. The examined profile reflects to the pattern observed in the figures 2 and 3 obtained from SEM microscope. It was particularly visible in the middle of the produced rod.

3.4 Nanoindentation tests

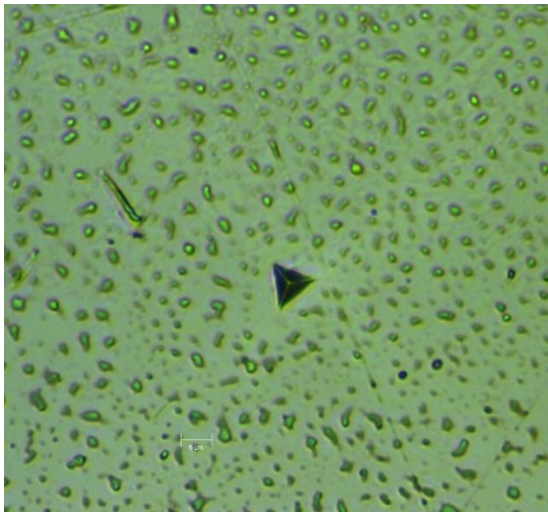


Fig. 7 Imprint of the diamond tip recorded on the surface of the $Fe_{75}B_{10}Si_{10}Cr_5$ alloy.

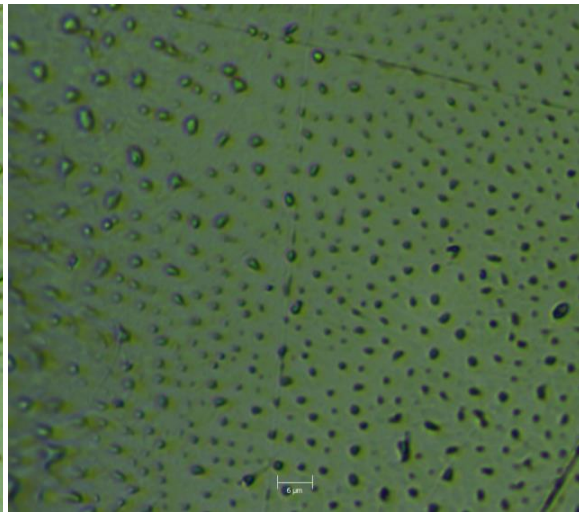


Fig. 8 Surface of the $Fe_{75}B_{10}Si_{10}Cr_5$ alloy observed under the microscope equipped with polarized light.

Figure 7 presents surface of the specimen with imprint recorded in the polarized light for the investigated material. The average hardness value of material is 844 HV. Moreover, the local hardness of 1478 HV was observed for some investigated areas due to the presence of the dendrites. Figure 8 shows difference in the structure observed for the produced material. A similar phenomenon was observed in Figures 2 and 3 from SEM microscope.

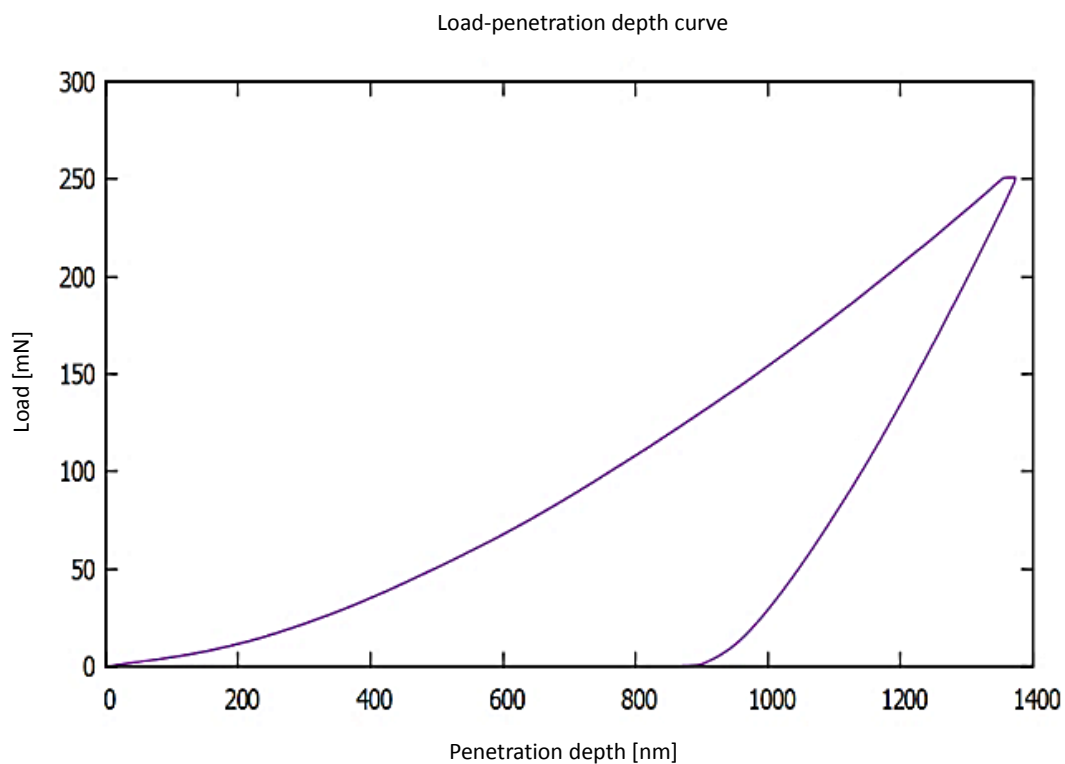


Fig. 9 Load-penetration depth curve recorded for the $Fe_{75}B_{10}Si_{10}Cr_5$ alloy.

Figure 9 shows load-penetration depth curve, where residual depth is 871 nm, contact depth is 981nm and maximal depth is 1373 nm. The analysis of the mechanical properties was based on the Oliver-Pharr method [5]. This method allows to calculate the hardness, Young's modulus and n_{IT} of the tested material directly from the resulting load curve. Table 1 shows received values of parameters i.e. Young's modulus, maximum load, elastic, plastics and total energy, the ratio of the elastic energy to the total energy.

Tab. 1. The Young's modulus, maximum load, strain, W_{elast} , W_{plast} , W_{total} , n_{IT} for the $Fe_{75}B_{10}Si_{10}Cr_5$ alloy.

| Parameters | Values |
|-----------------|--------------|
| Young's modulus | 128.21 GPa |
| Maximum load | 250.71 mN |
| Strain | 0.77 |
| W_{elast} | 50021.40 pJ |
| W_{plast} | 85048.33 pJ |
| W_{total} | 135069.72 pJ |
| n_{IT} | 37.03 % |

3.5 Magnetic properties

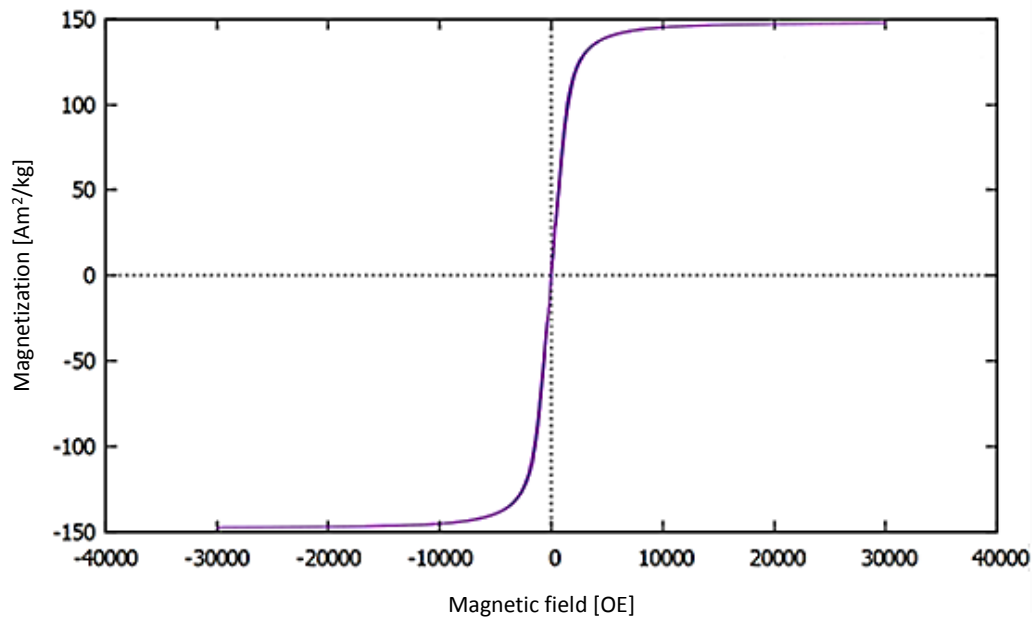


Fig. 10 Magnetic hysteresis loop for the as-quenched $Fe_{75}B_{10}Si_{10}Cr_5$ alloy.

The magnetic properties of the sample were measured by the vibrating sample magnetometer. During the examination, the magnetic hysteresis loop was recorded (Fig. 10). The maximum value of saturation magnetization was equal to $147.67 \text{ Am}^2/\text{kg}$. Based on the magnetic hysteresis loop, coercivity was appointed and its value equaled 2.184 kA/m , which was relatively small. It means that the $Fe_{75}B_{10}Si_{10}Cr_5$ alloy is magnetically soft. Materials with these properties show possibility to use as cores e.g. in transformers.

4. Conclusions

The Fe₇₅B₁₀Si₁₀Cr₅ alloy was produced using the arc melting method with a suction casting technology. The used method belongs to rapid solidification techniques. The produced cylindrical rod of the Fe₇₅B₁₀Si₁₀Cr₅ alloy was identified as partially crystallized with the α -Fe crystalline phase. No other phases were detected in X-ray diffraction pattern. The average crystallite size was 14.8 nm. The grain size was calculated from the Scherer equation. Due to the different cooling rates in the inner and outer part, variable sizes of dendrites were observed in the material's morphology. The mechanical properties were examined by nanoindentation measurements. The value of the hardness for the Fe₇₅B₁₀Si₁₀Cr₅ alloy is 844 HV. In addition, the higher values of hardness were also observed in some regions. Using AFM Park XE-100 system the average roughness for profiles was determined for the 45 μm \times 45 μm scanned area. Moreover, the average roughness of the area was investigated. R_a of this area was 6.098 nm and R_z was 62.428 nm. The Young's modulus of the Fe₇₅B₁₀Si₁₀Cr₅ alloy was 128.21 GPa. It is worth noticing that the produced alloy is characterized by narrow magnetic hysteresis loop. Therefore, the investigated Fe₇₅B₁₀Si₁₀Cr₅ material belongs to the group of soft magnetic materials.

References

- [1] J. J. Sunol, J. Fort, *Materials developed by mechanical alloying and melt spinning*, International Review of Physics, 2007
- [2] D. D. Xu, B. L. Zhou, Q. Q. Wang, J. Zhou, W. M. Yang, C. C. Yuan, L. Xue, X. D. Fan, L. Q. Ma, B. L. Shen, *Effects of Cr addition on thermal stability, soft magnetic properties and corrosion resistance of FeSiB amorphous alloys*, Corrosion Science, vol. 138, 2010, pp. 20-27
- [3] C. Zhang, Z. Zhu, H. Zhang, *Effects of the addition of Co, Ni or Cr on the decolorization properties of Fe-B-Si amorphous alloys*, Journal of Physics and Chemistry of Solids, vol.110, 2017, pp.152-160
- [4] W. Wolf, R. Mohs, U. Koning, *Soft magnetic low-cost amorphous Fe-Si-B alloys, their properties and potential uses*, Journal of Magnetism and Magnetic Material, vol. 19, 1980, pp. 177-182
- [5] Oliver W. C., Pharr G. M., *An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments*, Journal of Materials Research, vol. 7, 1992, pp. 1564-1583.