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Structural changes and Barkhausen effect parameters of amorphous-nanocrystalline alloys after various heat treatments

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Abstract. The influence of the structural state of amorphous magnetic soft Fe<sub>5</sub>Co<sub>10</sub>Si<sub>15</sub>B<sub>10</sub> (λ<sub>5</sub>=0.5·10<sup>-6</sup>), Fe<sub>60</sub>Co<sub>20</sub>Si<sub>5</sub>B<sub>15</sub> (λ<sub>5</sub>=30·10<sup>-6</sup>) and Co<sub>81.5</sub>Mo<sub>9.5</sub>Zn<sub>9</sub> (λ<sub>5</sub> is close to zero) alloys on the magnetic properties and informative parameters of the Barkhausen effect is studied. The structure of amorphous ribbons is investigated using transmission electron microscopy. The results obtained allow the conclusion that the study of the Barkhausen effect provides magnetic and structural information in the alloy. The parameters of the Barkhausen effect are shown to correlate with features of the fine structure (size of nanophases – 5-10 nm) of the investigated devitrifying amorphous alloys.

1. Introduction
The magnetic properties of amorphous alloys depend on their structural state. The use of informative parameters of the Barkhausen effect is a method for detecting changes in the fine structure during devitrification of amorphous alloys. Currently, the transmission electron microscopy (TEM) method is widely used to investigate the structural state of metals and alloys, including amorphous and nanocrystalline alloys. However, up to now there have been no studies relating TEM-studied structural changes of amorphous alloys to the informative parameters of the Barkhausen effect. Therefore, the authors of this paper have not only investigated the influence of the structural state of Fe- and Co-based amorphous alloys on the magnetic properties and Barkhausen effect parameters following different heat (HT) and thermomagnetic (TMT) treatments, but also structural features formed following the indicated treatments of selected alloys. For our investigation, we have selected alloys with unique soft magnetic properties and high thermal stability.

2. Results and discussion
Amorphous ribbons of Fe<sub>5</sub>Co<sub>10</sub>Si<sub>15</sub>B<sub>10</sub>, Fe<sub>60</sub>Co<sub>20</sub>Si<sub>5</sub>B<sub>15</sub> and Co<sub>81.5</sub>Mo<sub>9.5</sub>Zn<sub>9</sub> alloys were obtained by melt-quenching onto a rotating disk. Specimens were ribbon- and toroid-shaped. Heat treatments were carried out at temperatures from 300 to 450°C in a vacuum. Some specimens were subjected to thermomagnetic treatment in a dc magnetic field.

The structure of amorphous ribbons was checked by TEM method using a JEM-200KX microscope. For the TEM observations, foils of 200-300 nm thick were prepared from ribbons by electropolishing.
As an informative parameter of Barkhausen effect, we choose the emf of Barkhausen jump flow $\varepsilon$ averaged over a magnetization-reversal cycle. A Barkhausen jump (BJ) flow was visually observed on an oscilloscope screen. We measured the emf on ribbon-shaped specimens by an applied transducer.

The magnetic properties of alloys after various heat treatments are presented in Table 1.

Figure 1 displays oscillograms of the amplitude envelopes of the BJ flow for specimens of Fe$_{60}$Co$_{20}$Si$_3$B$_{15}$ alloy after both annealing at 300°C and TMT in a dc magnetic field at 400°C.

Figure 2 shows electron-microscopic pictures of the amorphous Fe$_{60}$Co$_{20}$Si$_3$B$_{15}$ alloys after the same treatments.

The oscillograms of the amplitude envelopes of the BJ flow for Fe$_{60}$Co$_{20}$Si$_3$B$_{15}$ specimens (figure 1a) show that BJ distribution over the field obeys Gaussian law owing to the presence of small magnetization-reversal domains with randomly distributed critical start fields. Structural studies showed that the alloy exist in the amorphous state after annealing at 300°C for two hours (figure 2a).

### Table 1. Magnetic properties of amorphous alloys after different treatments

| Alloy            | Treatments | $T_{an}$, °C | $\mu_0$ | $H_{c}$, A/m |
|------------------|------------|--------------|---------|--------------|
| Fe$_{60}$Co$_{20}$Si$_3$B$_{15}$ | 300 | 2 | 1050 | 2.5 |
|                  | TMT in dc field | 400 | 0.4 | 1200 | 2.5 |
|                  | 450 | 1 | 1200 | 1.5 |
| Co$_{81.5}$Mo$_{9.5}$Zr$_9$ | TMT in dc field | 450 | 1 | 3500 | 0.8 |

Then the Fe$_{60}$Co$_{20}$Si$_3$B$_{15}$ alloy specimen was subjected to the TMT in a dc field at 400°C. The oscillogram of the amplitude envelope of the BJ flow (figure 1b) show several jumps with critical start fields and a shift along the field axis caused by a hysteresis-loop displacement. Electron-microscopic investigations (figure 2b) showed that dispersed precipitations (with a size of <5 nm) of Co$_2$Si and Fe$_3$Si phases, in which the magnetization direction is determined by the magnetic-field direction during the TMT, are present in the alloy structure after the aforementioned treatment. From the comparison of oscillograms of the BJ flow amplitude envelopes demonstrated in figure 1 and electron-microscopic pictures of the Fe$_{60}$Co$_{20}$Si$_3$B$_{15}$ alloy structure shown in figure 2, it follows that a correlation between the alloy structure and the chosen informative parameter of the Barkhausen effect is possible. A Gaussian BJ field distribution corresponds to the amorphous structure, while the formation of dispersed precipitations in the alloy leads to the appearance on the oscillogram of several regions of the critical start fields, which correspond to precipitations in the amorphous matrix.

Note that the existence of phase inhomogeneity results in an increase of $\varepsilon$ by 10% as compared to one in amorphous-structured specimens. Magnetization reversal of the specimens subjected to TMT in a dc magnetic field is accomplished by a complex of Barkhausen jumps with close start fields. Figure 3 shows oscillograms of amplitude envelopes of the BJ flow for specimens of the amorphous metalloid-free Co$_{81.5}$Mo$_{9.5}$Zr$_9$ alloy after various HTs and TMTs.

Figure 4 shows electron-microscopic pictures of the amorphous Co$_{81.5}$Mo$_{9.5}$Zr$_9$ alloy after two heat treatments: at 300°C for 2 h and at 450°C for 1 h.

It is seen from oscillograms in figures 3a that the BJ field distribution also obeys the Gaussian law after annealing at 300°C. Electron-microscopic investigations showed that annealing at 300°C for 2 h did not affect the amorphous state of the matrix (figure 4a).

However, in the diffraction pattern of the alloy, in addition to the first and second diffusion halos, single and ring diffraction reflections are observed, where the latter consist of small single reflections. In the dark-field image of the alloy (obtained with the objective aperture of the microscope located on the diffraction ring near the first diffusion halo), crystalline phases are detected; the interpretation of these phases using the interplanar spacings allowed us to consider them as precipitations of $\alpha$-Co and $\beta$-Co phases with sizes of about 2 nm. A weak diffuse diffraction ring that is present at small angles on the diffraction pattern indicates the initial precipitation of small amounts of a dispersed Co$_2$Mo phase.
After annealing at 450°C for 1 h, several critical start-field regions are observed on the oscillogram of the BJ flow amplitude envelope (figure 3b).

The diffraction pattern from the selected area also changes (figure 4b), and additional ring reflections (besides small-angle ones) appear on the diffraction pattern at large angles. Dark-field images of the structure demonstrate coarser precipitates of crystalline phases, the interpretation of which allows us to consider them as $\alpha$-Co, $\beta$-Co, Co$_2$Mo, and Co$_2$(Mo, Zr) phases. Phase precipitates in an amorphous matrix reach 10-20 nm in size, but "coarse" particles are not numerous.

The comparison of structure images and oscillograms of the BJ flow amplitude envelopes of amorphous Co$_{81.2}$Mo$_{9.5}$Zr$_{9}$ alloy after heat treatments under different conditions also reveals a possible correlation between the alloy structure and the chosen informative parameter of the Barkhausen effect. After the TMT in a dc field, one can see a shift along the field axis on the oscillogram of the BJ flow amplitude envelope, caused by a hysteresis-loop displacement (Fig. 3c). Several areas of critical start fields are also observed in the oscillogram. Dispersed phases ($\alpha$-Co, $\beta$-Co, and Co$_2$(Mo, Zr)), with a magnetization direction determined by the direction of the dc magnetic field during the TMT, contained in the structure of this alloy, cause an increase in the BJ flow amplitude with simultaneous narrowing of the critical start-field regions.

After the TMT in an ac magnetic field, the displacement of the oscillogram of the BJ flow amplitude envelope along the field axis disappears, the hysteresis loop becomes symmetrical, and the jump amplitude decreases. The area of the critical start fields becomes narrower (compare fig. 3c and 3d). The emf of the Barkhausen jump flow, $\varepsilon$, averaged over a magnetization cycle and measured after the TMT in an ac magnetic field is 8% lower than $\varepsilon$ measured after the TMT in a dc magnetic field.
Figure 2. Electron-microscopic pictures of the structure of a Fe$_{60}$Co$_{20}$Si$_{15}$B$_{15}$ amorphous alloy and selected-area diffraction patterns after different treatments: (a) heat treatment at 300°C for 1 h and (b) thermomagnetic treatment in a dc field at 400°C for 5 min.

Figure 3. Oscillograms of Barkhausen jump flow amplitude envelopes after different treatments for a Co$_{81.5}$Mo$_{9.5}$Zr$_{9}$ alloy: (a) heat treatment at 300°C for 1 h, (b) heat treatment at 450°C for 1 h, (c) thermomagnetic treatment in a dc field at 400°C, and (d) thermomagnetic treatment in an ac field at 400°C.
Figure 4. Electron-microscopic pictures of the structure of a Co$_{81.5}$Mo$_{9.5}$Zr$_9$ amorphous alloy and selected-area diffraction patterns after different treatments: (a) heat treatment at 300°C for 2 h and (b) heat treatment at 450°C for 1 h.

3. Conclusions
1. The results of the performed investigation very likely testify that the use of informative parameters of the Barkhausen effect allows one to conclude about (a) the degree of devitrification of an amorphous alloy, (b) the presence of precipitated dispersed phases.
2. The Barkhausen effect parameters can be proposed as the parameters of a new method for testing the structure of an amorphous alloy during its devitrification.