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# PHYSICS

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## Structure-Phase States and Properties of High-Entropy Amorphous Soft Magnetic Fe-Co-Si-B-P Ribbons

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# ФИЗИКА

Научная статья

## Структурно-фазовые состояния и свойства высокоэнтропийных аморфных магнитомягких лент сплавов системы Fe-Co-Si-B-P

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***Abstract.*** This paper presents the analysis of the structure-phase states and mechanical and magnetic properties of amorphous $(FeCo)_{82}B_{13}Si_5$ and $(FeCo)_{82}B_{12}Si4P_2$ alloy ribbons produced by spinning. The analysis uses the relevant methods of physical materials science. The ribbons are in an amorphous state, while partial crystallization of the material is detected by scanning the prepared ion-thinned foils with the transmission electron microscopy. The investigated alloys are traced for their distribution of elements, and distinctive separation of Si and B layers is identified. Differential scanning calorimetry reveals the key crystallization temperatures for the both alloys.

***Аннотация.*** Методами современного физического материаловедения выполнен анализ структурно-фазовых состояний, механических и магнитных свойств лент аморфных сплавов $(FeCo)_{82}B_{13}Si_5$ и $(FeCo)_{82}B_{12}Si_4P_2$, полученных методом спиннингования. Ленты находятся в аморфном состоянии, тогда как при приготовлении фольг для просвечивающей электронной микроскопии в режиме ионного утонения выявлена частичная кристаллизация материала. Прослежено распределение элементного состава и отмечено расслоение сплава по кремнию и бору. Дифференциальной

The size of crystallization cells varies between 30–50 μm. It is shown that experimentally determined values of saturation induction (1.7–1.8 T) and coercive force (18–20 A/m) practically do not depend on the variation of elements' compositions in ribbons within the studied elements' concentration ranges. The values of tensile strength (~162 MPa) and elongation at break (~0.23 %) indicate a low plasticity of the investigated ribbons. Meanwhile, the elastic modulus has a high value of 81.5 MPa. A physical interpretation of the observed regularities is provided.

***Keywords:*** soft magnetic alloys, spinning, structure, mechanical properties

сканирующей калориметрией определены ключевые температуры кристаллизации обоих сплавов. Размер ячеек кристаллизации изменяется в пределах (30–50) мкм. Показано, что определенные экспериментально значения индукции насыщения (1,7–1,8 Тл) и коэрцитивной силы (18–20 А/м) практически не зависят от изменения состава лент в исследованном диапазоне содержания элементов. Значения предела прочности (~162 МПа) и удлинения до разрушения (~0,23 %) свидетельствуют о низкой пластичности исследуемых лент. Однако величина модуля упругости имела высокое значение на уровне 81,5 МПа. Дана физическая интерпретация необходимых закономерностей.

***Ключевые слова:*** магнитомягкие сплавы, спиннингование, структура, механические свойства

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At the end of the last century, a new paradigm of alloy design was proposed, based on mixing several elements in an equiatomic or close to equiatomic composition [1, 2]. They are called high-entropy alloys (HEAs) because they have a significantly higher entropy of mixing than conventional alloys. The excellent functional properties of HEAs and especially their magnetic properties are provided by high entropy, slow diffusion, large distortions of the crystal lattice and the coctale effect [3-8]. Literature reviews have noted unusual properties of HEAs materials, including wear resistance, radiation, corrosion resistance, heat resistance, superconductivity, low temperature coefficient of resistance, biological resistance, low thermal conductivity, etc. [9, 10]. When studying the magnetic properties of a number of HEAs, the prospects of developing soft magnetic materials have been shown; moreover, the magnetic properties can be controlled by alloying and annealing [11]. Paramagnetic HEAs of the CuCrFeTiNi composition with a small fraction of the ferromagnetic phase were obtained. Such HEAs as GdTbDyAlM (M=Fe, Co, Ni) based on rare-earth elements and metals of the iron group, are also of interest, as they demonstrate of the magnetocaloric effect.

The development of modern technology calls for the search and development of new materials that have not only higher performance properties than existing ones, but also a combination of different properties (physical, mechanical, chemical) that cannot be achieved on the basis of traditional crystalline materials. Amorphous metal alloys are indicative in this regard.

High-entropy metallic glasses, combining the features of both metallic glasses and high-entropy alloys, can become a new direction in the production of materials with high mechanical properties. Compared with conventional crystalline materials, they have excellent magnetocaloric properties over a wide temperature range [12]. The FeCoNi-based HEAs with additives of P, Si, B, and C are capable of easily forming metallic glasses [13, 14].

Possessing almost perfect phase-structural uniformity and high electrical resistivity, amorphous alloys have extremely low magnetization reversal losses. Another area of modern technology that absorbs amorphous alloys is the radio-electronic industry and instrument engineering. For these branches of technology, cobalt-based amorphous alloys with near-zero magnetostriction

are used, which have excellent hysteresis magnetic properties in small fields.

Over the past decade, a noticeable improvement in the magnetically soft properties of amorphous alloys has been achieved, which was motivated by the industry's need to develop materials with higher saturation magnetization, lower coercive force, and high magnetic permeability. The use of such materials allows for energy conservation, increased efficiency, and miniaturization of electronic and magnetic devices [15].

Of particular interest may be thin strips of HEAs (amorphous alloys) made by quenching from a melt. It is important to regulate and improve the mechanical and magnetic properties of magnetically soft amorphous alloys [16, 17].

The authors of [18–20] developed amorphous alloys based on the FeXoSiB system with a high saturation induction value of Bs (above 1.7 T) in combination with low values of the Hc coercive force (below 20 A/m). In [21], the authors managed to achieve a high Bs value exceeding 1.8 T in an amorphous Fe-Co-based alloy, and the highest Bs value=1.86 T, combined with a low Hc value of 3 A/m, was obtained for ribbons made of an amorphous alloy (Fe0.8Co0,2)83Bi16Si1. It was found that the simultaneous choice of 0.25 Co/Fe components and a boron-rich metalloid plays an important role in achieving high Bs, mainly exceeding 1.8 T.

The purpose of this work is to analyze the structural and phase states, deformation behavior, and magnetic properties of the amorphous Fe-Co-Si-B-P alloy ribbons obtained by spinning.

**Materials and methods of research**

The research material was samples (ribbons) of (FeCo)82B13Si5 and (FeCo)82B12Si4P2 alloys. The ribbons were made by spinning at the URS 1.0 Chermet Central Scientific Research Institute installation (Moscow). Technical parameters of wind turbine spinning: melt temperature — 1480 °C; linear speed of the quenching drum — 32.6 m / sec; the value of the extrusion (excess) pressure — 0.35–0.3 atm; nozzle size — 0.7* 20 mm; the size of the gap between the nozzle and the drum — 0.27 mm. The ratio Fe:Co=50:50 was used, since such a ratio can lead to very high values of magnetic properties (high saturation induction Bs and low coercive force Hc) of magnetically soft materials with an amorphous structure.

The proportions of alloying components — Si, B, and P — were selected based on the criteria of amorphization and data from the scientific literature, where special attention is paid to the effect of the above components on the properties of the obtained materials [22, 23].

For ribbons in the initial state (state of supply), the average thickness was (29–34) microns, width (4.5–6.5) mm, characteristic length (142–156) mm.

**Differential scanning calorimetry (DSC)**

The thermal analysis of the ribbon samples obtained by spinning was carried out on a synchronous thermal analyzer Netzsch STA 449 F3. To perform DSC analysis, samples with a width of 1 mm and an average thickness of 18 microns were cut into fragments with a length of 4 mm, weighed (the mass of the samples was ≈4 mg) and placed in a corundum (Al2O3) crucible. The measurements were carried out by heating in the temperature range from room temperature to 700 °C, followed by cooling and reheating at a heating/cooling rate of 10 K/min in an argon atmosphere. The standard was an "empty" corundum crucible.

**Magnetic properties**

A VSM-250 vibration magnetometer was used to determine saturation induction. The data was processed using the Model DJAW2000 software. The specific saturation magnetization σs was determined in a field of 40 kA/m.

The MK-3E magnetic measuring device was used to measure the coercive force. An induction-pulse measurement method was used, corresponding to the method of static magnetic measurements of magnetically soft materials according to GOST 8.377-80.

**X-ray diffractometry**

The X-ray diffractometer DRON-4 was used for the survey, the radiation was Co-Kα, the measurement error of 2θ is 0.01°. For shooting, ribbons samples from 10 mm to 15 mm long and 4.5 mm to 6.5 mm wide were mounted on a plasticine substrate.

**Scanning and transmission electron diffraction microscopy**

(KYKY-EM6900 and JEM-2100 instruments) were used to study the structure, elemental and phase composition of the ribbons. Foils were produced by ion thinning of plates on an Ion Slicer EM-091001S installation (thinning was carried out by a stream of argon ions) and by electrolytic polishing methods. The deformation curves of the ribbons $(FeCo)_{82}B_{13}Si_5$ were recorded on the Instron5582 installation.

**Research results and their discussion**

**Differential scanning calorimetry**

The thermal analysis of the samples of ribbons obtained by spinning was carried out. The curves obtained by differential scanning calorimetry (DSC) are typical for amorphous alloys with a relatively low boron content (<17 at. %) (Fig. 1).

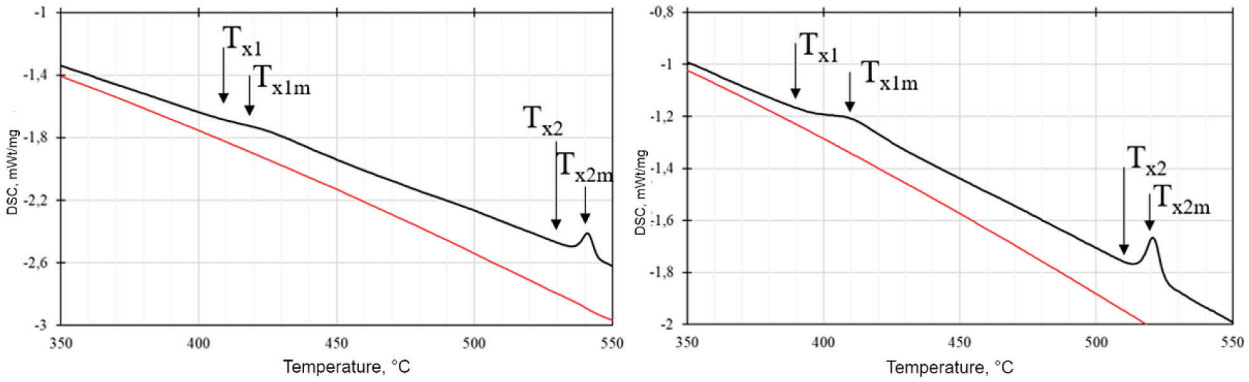


Fig. 1. DSC thermograms of samples $(FeCo)_{82}B_{13}Si_5$ (a) and $(FeCo)_{82}B_{12}Si_4P_2$ (b)

Table 2 shows the key temperatures of the alloy crystallization processes. Two-stage crystallization is observed, which is respectively characterized by two temperatures of the beginning of crystallization, $T_{x1}$ and $T_{x2}$, the values of which are shown in Table 1.

*Table 1*

Temperatures of the crystallization process of amorphous ribbons

| Alloy | $T_{x1}$, C | $T_{x1m}$,°C | $T_{x2}$,°C | $T_{x2m}$,°C |
|---|---|---|---|---|
| $(FeCo)_{82}B_{13}Si_5$ | 390 | 410 | 510 | 520 |
| $(FeCo)_{82}B_{12}Si_4P_2$ | 410 | 420 | 530 | 540 |

*Table 2*

Average values of magnetic properties of ribbons samples in the initial state

| Alloy | $H_c$, A/m | $B_s$, T |
|---|---|---|
| $(FeCo)_{82}B_{13}Si_5$ | 20 | 1,8 |
| $(FeCo)_{82}B_{12}Si_4P_2$ | 18 | 1,7 |

**Measurement of magnetic properties**

Table 2 shows the average values of saturation induction and coercive force for samples $(FeCo)_{82}B_{13}Si_5$ and $(FeCo)_{82}B_{12}Si_4P_2$ in the initial (amorphous) state. It can be seen that the change in composition was not accompanied by a drastic change in the magnetic properties that the studied ribbons samples possessed at the Hc=18–20 A/m level.

**X-ray diffractometry**. The diffraction spectra of the ribbons confirmed the amorphous state of the studied materials.

**Scanning electron microscopy** studies of the etched surface of $(FeCo)_{82}B_{13}Si_5$ ribbons (Fig. 2, a, b) and $(FeCo)_{82}B_{12}Si_4P_2$ and (Fig. 2, c, d), performed by scanning electron microscopy, revealed a dendritic (cellular) structure of the material, the cell size of which varies within (30–50) microns. This may indicate that the ribbons are stratified by the chemical elements that form them.

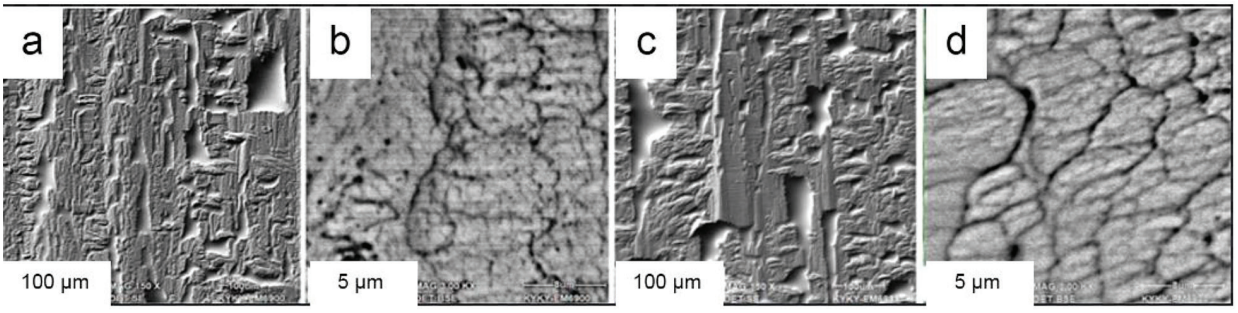


Fig. 2. Electron microscopic image of the etched ribbon structure; a, b — alloy $(FeCo)_{82}B_{13}Si_5$, c, d — alloy $(FeCo)_{82}B_{12}Si_4P_2$

**Transmission electron diffraction microscopy**. Figure 3 shows the electronograms obtained in the study of alloys $(FeCo)_{82}B_{13}Si_5$ (a) and ((FeCo)$_{82}B_{12}Si_4P_2$ (b). The presence of diffuse rings on the electronograms indicates the amorphous state of the studied ribbons, which confirms the results of X-ray phase analysis.

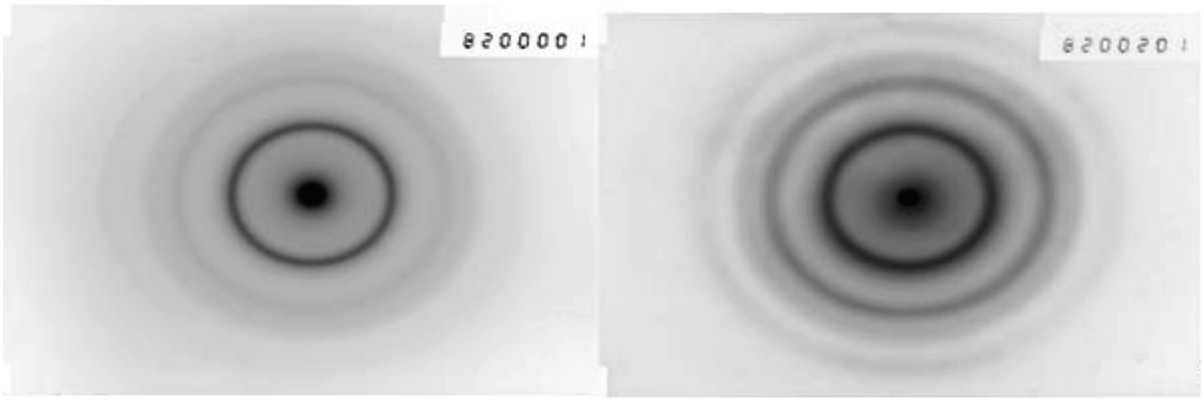

Fig. 3. Electronograms of strips of alloys $(FeCo)_{82}B_{13}Si_5$ (a) and $((FeCo)_{82}B_{12}Si_4P_2$ (b)

Figure 4 shows electron microscopic images of the ribbon structure $(FeCo)_{82}B_{12}Si_4P_2$ obtained by examining a foil made by the electrolytic method (Fig. 4, a, b) and the ion thinning method (Fig. 4, c, d). A cellular type structure with a cell size varying from 150 nm is observed up to 300 nm. Judging by the microelectronogram (Fig. 4, b), the studied ribbon is in an amorphous state. It can be assumed that the revealed cellular structure of the alloy reflects the domain structure of the magnetic material, and the cell size corresponds to the size of the magnetic domains.

Changing the ribbon thinning mode during the foil manufacturing process led to partial crystallization of the material (Fig. 4, c, d). This is indicated by the presence of an amorphous ring and point reflections on the microelectronogram (Fig. 4, d).

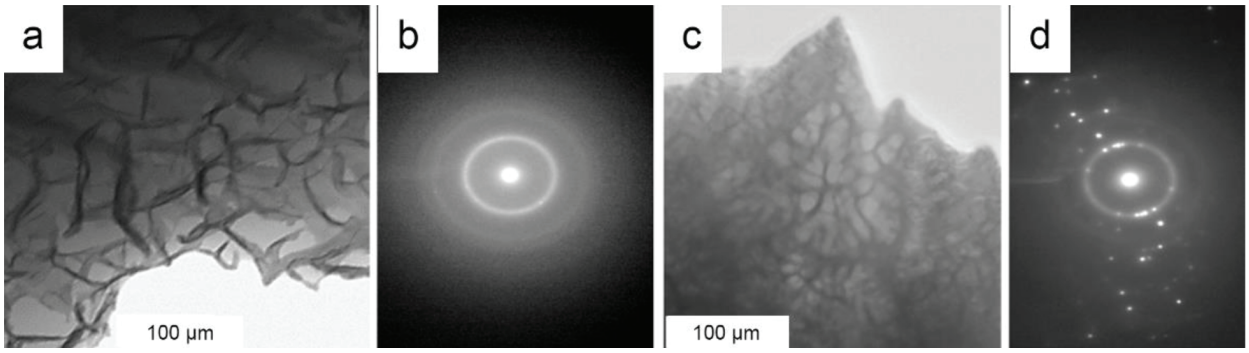


Fig. 4. Electron microscopic image of the ribbon structure subjected to electrolytic thinning (a, b) and thinning by a stream of argon ions (c, d); a, c — light fields; b, d — corresponding microelectronograms

The distribution of chemical elements in the ribbon structure has been studied using micro-X-ray spectral analysis of foils. It was shown that, regardless of the foil preparation method, the stratification of the studied material in silicon and cobalt is observed - the interlayers separating the cells are enriched in silicon and depleted in cobalt. Apparently, the silicon and cobalt stratification of the ribbons occurred during spinning.

**Mechanical properties**. Table 3 shows the initial data of mechanical tests of the ribbons composition $(FeCo)_{82}B_{12}Si_4P_2$, determined according to the loading diagrams, which had an almost linear appearance, and the nature of the fracture can be considered as brittle. The low strength of the samples could be caused by structural defects identified by SEM and TEM methods (Fig. 2–4).

*Table 3*

Mechanical properties
of tested belt samples (FeCo)82B12Si4P2

| Sample | Elasticity modulus E (MPa) | Ultimate strength σв (MPa) | Elongation to destruction εp (%) |
|---|---|---|---|
| 1 | 76,83 | 203,2 | 0,256 |
| 2 | 79,64 | 149,1 | 0,281 |
| 3 | 87,88 | 134,6 | 0,156 |
| Average values | 81,5±5,7 | 162±36 | 0,23±0,07 |

Figure 5 shows electron microscopic images of the fracture surface of the ribbon under tensile testing conditions. It can be seen that a block structure has been formed, characteristic of the amorphous state of the material. The dendritic (cellular) structure of the ribbon is not revealed.

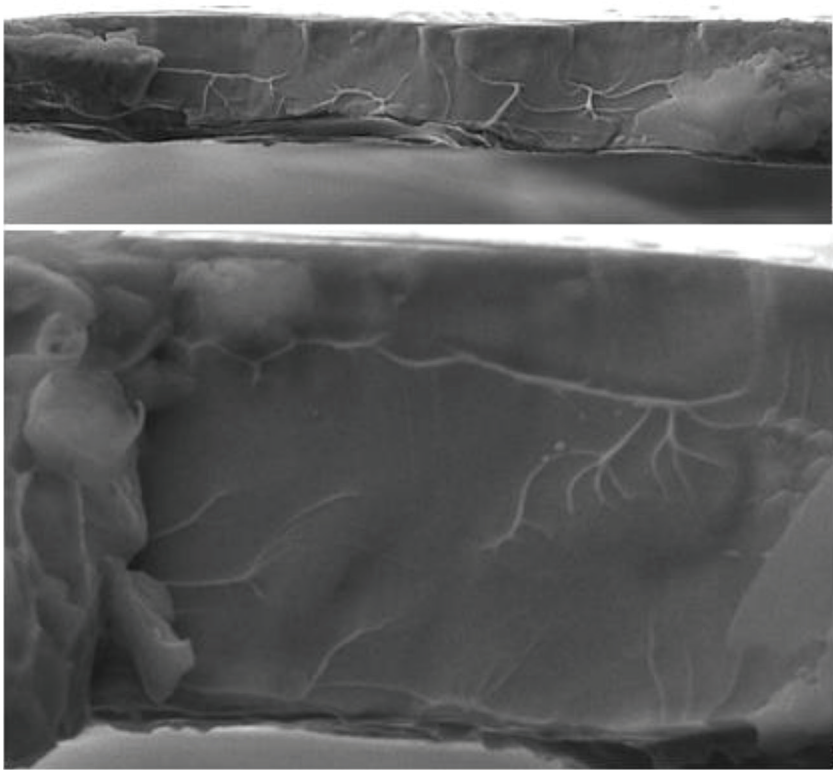

Fig. 5. Electron microscopic image of the fracture surface structure formed by stretching samples of $(FeCo)_{82}B_{12}Si_4P_2$ ribbon

**Discussion of the results**

Amorphous alloys are characterized by a short-range order due to the lack of translational symmetry, so they lack characteristic defects such as dislocations, twins, packing defects, and grain boundaries [18]. As indicated in the cited monograph, amorphous alloys, regardless of the type and concentration of components, are a single-phase system, the structure of which is a supersaturated solid solution. According to the author of the cited work, amorphous alloys, which include those studied in this work, have the high atomic-structural and phase-chemical homogeneity. This allows them to achieve a unique combination of different properties.

The objects of research in this work were two amorphous alloys $(FeCo)_{82}B_{13}Si_5$ and $(FeCo)_{82}B_{12}Si_4P_2$, which are quite similar in composition. In the framework of the conducted studies, it was shown that they practically did not differ in terms of their key functional characteristics, namely, the induction of saturation of Bs. In general, this was not an unexpected result, since the content of the key element controlling magnetic properties (Co) was identical for them. In any case, the achieved level of magnetic properties can be considered one of the best indicators for amorphous alloys.

On the other hand, the introduction of 2 % phosphorus was apparently intended to increase fluidity, since it is known that an increase in phosphorus content in the structure of cast iron is accompanied by the formation of solid inclusions of phosphide eutectic, improving the casting properties of the material. Additionally, the overall hardness and wear resistance can be increased. However, for cast iron, the phosphorus content should not exceed 0.4 %.

Alternetavly the disadvantages of the obtained ribbons include the "stratifications" of cobalt and silicon identified above, as well as low mechanical characteristics. If the first can be attributed to a suboptimal ratio of components, which will be the subject of further research, then the second is clearly related to structural macrouniformity, actually allowed by the longitudinal stratification of the ribbons. At the same time, the modulus of elasticity fixed at 81.5 GPa indicates that, unlike the deformation properties, the strength properties of the alloy ($(FeCo)_{82}B_{12}Si_4P_2$ are more than acceptable.

Thus, the amorphous alloys obtained and studied in this work have a good prospect of being used as materials, for example, for the manufacture of magnetic circuits, where strength properties are not limiting, while it

is necessary to ensure the required indicators of magnetic induction and coercive force.

**Conclusion**

The structural and phase states, mechanical and magnetic properties of soft magnetic ribbons $(FeCo)_{82}B_{13}Si_5$ and $(FeCo)_{82}B_{12}Si_4P_2$ obtained by spinning have been studied. The key temperatures of crystallization of both alloys were determined by differential scanning calorimetry. The values of saturation and coercive force inductions are in the range of (1.7–1.8) T and (18–20) A/m. Analysis of the thin structure of the ribbons by the TEM method with electrolytic polishing of foils has shown that the ribbons are in an amorphous state, whereas partial crystallization of the material is observed in the ion thinning regime.

When spinning, cobalt and silicon the stratification occurs. Scanning electron microscopy revealed a dendritic (cellular) structure, the size of which varies between (30–50) microns. Mechanical tests for uniaxial tension of the ribbon (FeCo)82B13Si5 revealed the low plasticity (εp~0.23 %), as well as the strength (σb~162 MPa) due to the amorphous structure of the ribbon and the presence of macro-effects such as local longitudinal stratifications.

The manufacturing method used and the composition of HEAs $(FeCo)_{82}B_{13}Si_5$ and $(FeCo)_{82}B_{12}Si_4P_2$ make it possible to obtain soft magnetic ribbons with an amorphous structure, however, the latter, combined with the formation of longitudinal stratifications (discontinuities), causes both extremely low ductility and strength, since brittle and rapidly advancing fracture with linear deformation does not allow to achieve a high level of deforming stresses.

On the other hand, a sufficiently high modulus of elasticity of ~81.5 GPa indicates that the improvement of spinning technology (selection of optimal technological parameters) and, possibly, a different quantitative combination of components, by eliminating the factors of occurrence of (macro)inhomogeneities (stratifications, thicknesses, etc.) can achieve higher strength indicators with satisfactory plasticity.

**Conflict of interests**

The authors declare that there is no conflict of interest.

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