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EFFECT OF DEFORMATION TEMPERATURE ON THE MICROSTRUCTURE OF HARD MAGNETIC FeCr22Co15 ALLOY SUBJECTED TO TENSION COMBINED WITH TORSION DEFORMATION MODES

WPŁYW TEMPERATURY ODKSZTAŁCENIA NA MIKROSTRUKTURĘ MAGNETYCZNIE TWARDEGO STOPU FeCr22Co15 PODDANEGO ROZCIĄGANIU WRAZ ZE SKRĘCANIEM

The paper presents the results of microstructure evolution studies of hard magnetic FeCr22Co15 alloy deformed until destruction by tension and torsion in the temperature range 725-850°C. The temperatures and deformation rates resulted from the condition of superplasticity occurrence in the Fe-Cr-Co alloys. Observations of the longitudinal sections of the deformed samples in the scanning electron microscope showed the formation of a weak gradient microstructure with the highest grain refinement in the surface layer of the material. Increasing the deformation temperature from 725 to 850°C increased the homogeneity of the deformation along the tensile axis of the sample. It also brought about the increase of grain size and slight increase of the thickness of fine grains in the surface layer. The precipitation of the intermetallic σ-phase was also observed with its maximum amount in the zones of the highest deformation.
Keywords: Hard magnetic alloy, high temperature deformation, gradient microstructure, interface segregation

Praca przedstawia wyniki badań ewolucji mikrostruktury magnetycznie twardego stopu FeCr22Co15 poddanego odkształceniu poprzez rozciąganie i skręcanie próbek do ich zerwania w przedziale temperatur 725-850°C. Temperatury i prędkości odkształcenia odpowiadały warunkom nadplastyczności badanego stopu. Obserwacja mikrostruktury na przekroju podłużnym próbek w skaningowym mikroskopie elektronowym wykazała tworzenie się mikrostruktury o słabym charakterze gradientowym z minimalnym rozmiarem ziaren w warstwie wierzchniej materiału. Zwiększenie temperatury odkształcenia od 725 do 850°C spowodowało polepszenie jednorodności odkształconej mikrostruktury wzdłuż osi rozciągania próbek oraz zwiększenie rozmiaru ziaren fazy α. Stwierdzono również, że grubość warstwy wierzchniej o drobnym ziarnie w niewielkim stopniu zależy od temperatury odkształcenia. Ponadto stwierdzono obecność fazy międzymetalicznej σ (Fe-Cr), której największą ilość zaobserwowano w warstwie wierzchniej materiału.

1. Introduction

Magnetically hard Fe−Cr-Co-based alloys are distinguished by their good ductility, excellent magnetic properties and low cost [1-3]. Their superior magnetic properties are obtained by a magnetic treatment and multistage tempering, which results in the spinodal decomposition of the solid solution into isomorphous, ordered and coherent phases, i.e., the α_1 ferromagnetic and α_2 paramagnetic ones [4, 5]. The formation of such structures, in which each precipitate of the α_1 phase is a single magnetic domain, provides superior magnetic properties. However, internal stress fields, which originate from the formation of coherent boundaries between the α_1 precipitates and α_2 matrix cause a reduction in plasticity and strength of the material, which limits its possible applications. In order to improve its mechanical properties, the FeCr22Co15 alloy was deformed by tensile combined with torsion before the magnetic treatment. In the case of the FeCr30Co8 alloy investigated earlier, such a mode of plastic deformation results in the formation of a gradient microstructure with the maximum grain refinement in the surface layer [6, 7]. The gradient microstructure with the axial symmetry of cylindrical samples is an excellent solution for the case of magnets which rotate at high speed and need to have good mechanical properties on the surface as well as good magnetic properties inside the material.

The aim of the paper was to present the results of the microstructure investigation of the FeCr22Co15 alloy deformed until destruction in the range of temperatures 725-850°C.

In the future, in order to achieve stronger gradient microstructure, the deformation of the alloy will be performed under different conditions.

2. Materials and research methods

FeCr22Co15 alloy composed of 23.0 wt.% Cr, 15.8 wt.% Co, 0.9 wt.% Ti, 0.9 wt.% V, balance Fe, was cast and rolled.

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In order to obtain a solid solution α, the alloy was subjected to homogenization and water-quenched from 1240°C. Next, it was subjected to plastic deformation in a modernized "Instron" machine, which allowed deformation at single or simultaneous actions of axial loading and torsion under superplasticity conditions [8]. Cylindrical samples, 8 mm in diameter and 44 mm length, were deformed simultaneously by tensile with torsion, until their breaking at 725, 750, 800 and 850°C. The deformation temperatures were chosen in order to obtain phases from the $\alpha + \gamma$ range. The tension was applied to the upper part of the sample and the torsion to its bottom part (Fig. 1b). The rate of torsion was 0.27 turns/min ($5{,}6 \cdot 10^{-4}$ s^{-1}). The temperatures as well as deformation rates corresponded to the superplasticity conditions of the alloy. The samples were broken off after 3% elongation combined with 11 rotations at 725°C, after 7% elongation together with 12 rotations at 750°C, after 9% elongation with 16 rotations at 800°C, and after 11% elongation and 18 rotations at 850°C. The data of true strains of tensile and torsion of the FeCr22Co15 alloy deformed at different temperatures are given in Table 1.

TABLE 1

The data of elongation by tension, number of rotations and strain of FeCr22Co15 alloy deformed at different temperatures

ε ln	T, [°C]			
	725	750	800	850
Tension	0.02	0.07	0.09	0.11
Torsion	1.6	1.8	2.1	2.2

The tensile strain was calculated basing on the following formula (1) [9]:

$$\varepsilon = ln(l/l_0) \tag{1}$$

where: l_0 [mm] is the sample length before deformation and l [mm] is the sample length after deformation.

The strain in torsion was calculated based on the formula (2) [9]:

$$\varepsilon = ln(1 + (\varphi R/l)^2)^{1/2} \tag{2}$$

where: φ[rad] is the angle of torsion deformation and R [mm] is the radius of the deformed sample.

The microstructure was examined by means of the scanning electron microscope (SEM) XL 30 ESEM, Philips and the transmission electron microscope (TEM) Tecnai G^2 F20 (200 kV). The maps of orientations were measured with use of the EBSD method for the analysis of grain boundaries.

3. Results and Discussion

The example of initial microstructure is presented in Fig. 1a. The grain size of α phase was about 700 μm.

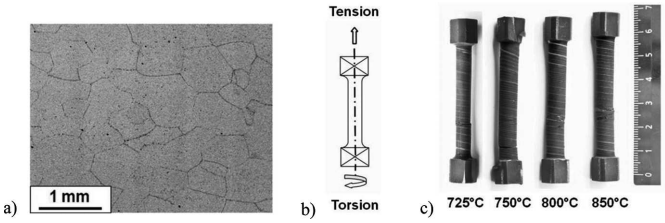

Fig. 1. Initial microstructure of the α-solid solution of the FeCr22Co15 alloy after homogenization at 1240°C (a). The scheme of deformation of the FeCr22Co15 alloy by tension applied to the upper part of the sample and torsion applied to the bottom part of the sample (b). The samples of the FeCr22Co15 alloy after deformation by tension and torsion at 725, 750, 800 and 850°C (c)

Before the deformation a scratch was grooved along the side of each sample. It became spiral after the torsion. Based on the observation of the spiral mark it could be established that at lower temperatures (725, 750°C) the deformation was inhomogeneous with the highest strain from the side of torsion, while at higher temperatures (800, 850°C) the deformation along the longitudinal axis was homogenous (Fig. 1c). Increasing the temperature from 725 to 850°C resulted in an increase of the number of rotations which led to the breaking of the samples: 11, 12, 16 and 18 at 725, 750, 800 and 850°C respectively. It confirmed that the plasticity of material increased with the increase of the temperature of deformation.

The observation of the microstructure in the longitudinal sections of deformed samples showed a weak microstructure gradient with the minimum grain size at the surface layer (Fig. 2). It seemed that in the first stage of deformation, the refinement of microstructure took place only in the surface layer of material, as observed in [7, 8]. With further increase of deformation the microstructure refinement took place deeper into the volume of material, reaching almost the longitudinal axis of the sample. A further increase of deformation destroyed the samples. A thin area of less refined material extended along the tensile axis. The increase of temperature from 725 to 850°C slightly increased the thickness of the surface layer from 3.2 to 3.5 mm (for the total thickness of 7.5 mm). The grains of α phase in the surface layer were generally globular, while those inside the material (near to the longitudinal axis) were larger, elongated and inclined at the angle of approximately 45° to the sample axis.

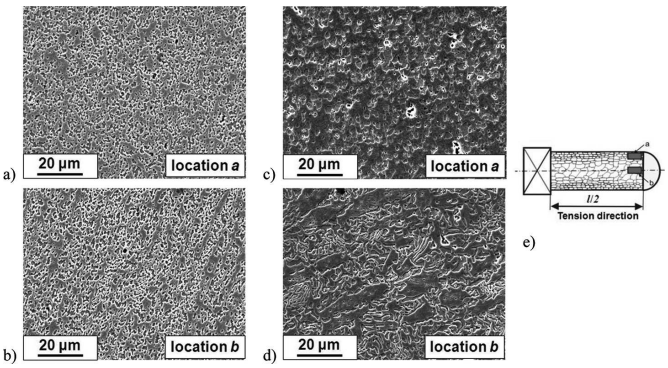

Fig. 2. The microstructure of the FeCr22Co15 alloy after deformation at 725 (a, b) and 850°C (c, d). The microstructures were taken at the defined sample locations as on the scheme (e), SEM

According to the EBSD measurements, the precipitations of the intermetallic σ phase (Fe-Cr) were observed in the deformed alloy. Previous studies [10] showed that intensive deformation stimulated the precipitation of σ phase by the activation of diffusion processes. Therefore, more σ phase precipitates were observed at the surface of the sample (Fig. 3, location a) than in the volume of material (Fig. 3, location b). The fraction of the σ phase precipitations could reach up to 42% depending on the deformation temperature (Table 2). A small amount of γ phase (2÷4.5%) was also observed (Table 2). The amount of γ phase was probably related to the presence of titanium and vanadium in the alloy, which stabilized the α phase, and consequently reduced the amount of γ phase to a lower value than it was expected on the base of the equilibrium phase diagram.

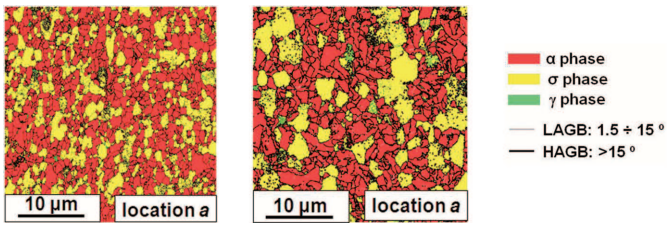

Fig. 3. Maps of phase distribution obtained at surface of samples deformed at 725 (a) and 850°C (b), SEM/EBSD

The detailed analysis of the low angle grain boundaries (LAGB) by the EBSD method showed, that the deformation resulted in the formation of the subgrain microstructure of α phase. It should be noted that the development of the subgrain microstructure at the surface was more intensive than in the volume of the material (except for the deformation at 725°C). Increasing the temperature from 725 to 850°C resulted in an increase of the fraction of LAGBs at the surface layer and slightly decreased their fraction in the volume of material (Table 2).

TABLE 2

The fraction of phases, low and high angle grain boundaries (LAGB, HAGB) in the samples of FeCr22Co15 alloy deformed at different temperatures

T [°C]	The place of EBSD measuring	The fraction of phases [%]			The fraction of boundaries [%]	
		α	σ	γ	LAGB	HAGB
725	location *a*	59.0	38.7	2.3	52.5	47.5
	location *b*	60.0	35.5	4.5	58.0	42.0
750	location *a*	59.0	38.0	3.0	59.6	40.4
	location *b*	65.0	32.2	2.8	58.3	41.7
800	location *a*	55.6	41.5	2.9	63.0	37.0
	location *b*	57.0	39.2	3.8	56.0	44.0
850	location *a*	63.8	33.4	2.8	61.0	39.0
	location *b*	65.9	31.0	3.1	54.0	46.0

The TEM observation of surface layer microstructure of the deformed samples showed that decrease of deformation temperature from 850 to 725°C resulted in decrease of the grain size of α and σ phases from about 2.0 to 0.8 μm. The examination of thin foils also confirmed the presence of the subgrain microstructure in the α phase. At lower temperatures of deformation the high density of dislocations tangles were observed in the α phase grains (Fig. 4a). At higher temperatures the high density of dislocations were also observed, but the dislocations showed a tendency to form distinct low-angle boundaries of the elongated subgrains (Fig. 4b). In that way, the subgrain microstructure was more intensively developed at the higher deformation temperatures.

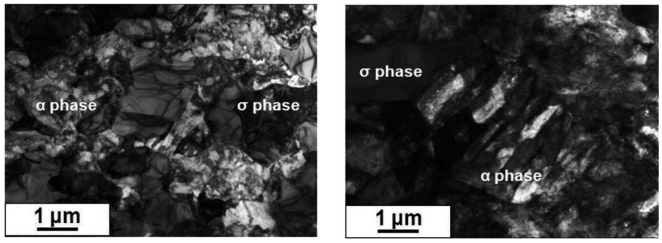

Fig. 4. The microstructure and point diffraction from the surface layer of deformed samples at 725 (a) and 850°C (b), TEM. The grains of σ phase are dark and without dislocations. The grains of α phase are white and reveal a high density of dislocations

4. Conclusions

Severe plastic deformation of the FeCr22Co15 alloy carried out until its destruction resulted in the strong grain microstructure refinement almost on the entire cross-section of the samples. A weak gradient microstructure with an axial symmetry appeared at the minimum grain size (0.8-2.0μm) in the surface layer of the material. Increasing the deformation temperature from 725 to 850°C weakly affected the thickness of the surface layer and increased the homogeneity of the deformation along the tensile axis of the sample.

The deformation also caused the formation of subgrain microstructure which was more intensive in the surface layer than inside the material. Tangles of dislocations were observed in the α phase grains at lower temperatures of deformation. At higher temperatures the dislocations tended to form the distinct low-angle boundaries of elongated subgrains.

The deformation also stimulated the precipitation of intermetallic σ phase. The highest amount of σ phase was observed at deformation temperature 800°C.

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