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STRUCTURE OF 25KH15K HARD MAGNETIC ALLOY AFTER SEVERE PLASTIC DEFORMATION BY UPSETTING AND TORSION

STRUKTURY TWARDEGO MAGNETYCZNIE STOPU 25KH15K PO INTENSYWNYM ODKSZTAŁCENIU PRZEZ SPĘCZANIE I SKRĘCANIE

The structural evolution of the hard magnetic alloy 25Kh15K of ($\alpha + \gamma$) state, subjected to upsetting and subsequent torsion deformation at elevated temperatures, was studied mainly by means of the Orientation Mapping (OM) technique in the transmission and scanning electron microscope (TEM and SEM). Microscopic observations and measured orientation maps show, that the transformation of the coarse plate structure into the globular one occurred in all sample sections. However, the formation of sub-microcrystalline (SMC) layers took place only in the zones of the highest deformation, near the moving anvil. The thickness of SMC layer does not show correlation with deformation temperature. There are some parts of the σ phase as well. It is possible, that a part of the α phase has been transformed into the intermetallic sigma phase. The structure of of the highest deformation zone consisted of uniform grains of α, γ and σ phases, all sized 200-500 nm. Most of the grain and interphase boundaries were characterized by high disorientation angles.

Keywords: hard magnetic alloy, sub-microcrystalline structure, severe plastic deformation, orientation mapping, Electron Back Scattered Diffraction patterns (EBSD), Convergent Beam Electron Diffraction patterns (CBED)

Zmiany struktury w twardym magnetycznie stopie 25Kh15K ($\alpha + \gamma$), który został odkształcony przez spęczanie i następujące po nim skręcanie przy podwyższonej temperaturze, były badane w oparciu o techniki mapowania orientacji w transmisyjnym i skaningowym mikroskopie elektronowym. Obserwacje mikroskopowe oraz analiza zmierzonych map orientacji wskazuje, że transformacja pierwotnej grubo płytkowej struktury w strukturę globularną następuje we wszystkich obszarach odkształcanej próbki. Struktura o submikronowym ziarnie tworzy się jednakże tylko w strefie największej deformacji w pobliżu ruchomego kowadła. Grubość warstwy o submikronowym ziarnie nie zależy w istotny sposób od temperatury odkształcenia. W warstwie tej obok cząstek faz α i γ obserwowano cząstki fazy σ. Jest możliwe, że część fazy α uległa transformacji do międzymetalicznej fazy σ. Struktura strefy największego odkształcenia jest złożona z równoosiowych ziarn o rozmiarach 200-500 nm wszystkich trzech faz. Większość granic międzyfazowych charakteryzują duże kąty dezorientacji.

1. Introduction

The 25Kh15K alloy belongs to hard magnetic materials of the Fe-Cr-Co system, characterized by good magnetic properties and manufacturability [1]. However, the formation of a high-coercivity state during spinodal decomposition leads to a modulated structure, consisting of coherent, ordered precipitates of the α_1 phase in the α_2 matrix, which results in decreased strength and plasticity. On the other hand, it is known that the formation of nano- or sub-microcrystalline (SMC) structures results in the improving mechanical properties of many materials [2, 3]. In particular, severe plastic deformation (SPD) by torsion under quasi-hydrostatic pressure forms a structure with the grain size of 100-200 nm in ductile and hardenable metals, and 10-20 nm in intermetallics and metal-metalloid alloys [4, 5]. It was already shown [6], that SPD by high pressure torsion on Bridgman anvils at room temperature of the 25Kh15K alloy in the state of high coercivity results in improving the alloy

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strength and plasticity. Bridgman anvils technique, however, has one disadvantage: the produced samples have small thickness (less then 1 mm). Thus, the aim of this ongoing work consists in analysing the structure evolution of the hard magnetic 25Kh15K alloy during more complex SPD, consisting of upsetting and torsion, which enables one to produce bulk samples [7]. Depending on the necessary resolution the techniques of OM in SEM or OM in TEM were used for analysis of microstructure.

2. Research material and methods

The cast alloy ingot (Table 1) was water-quenched from 1200°C, to obtain a single-phase α solid solution.

TABLE 1

Alloy 25Kh15K, chemical composition (% by weight)

Cr	Co	Ti	V	Si	Al	Nb	Fe
25	15	1	1	0,4	1	1	Remainder

Four cylindrical samples – 12 mm in diameter and 10 mm high – were cut from the ingot and subjected to SPD by complex two-step loading at constant temperature. The temperatures of deformation (700, 750, 800, 850°C) were chosen from the range of the two-phase (α + γ) domain. The SPD was realized in two steps: in the first step, (Fig. 1a) the samples were subjected to upsetting with the strain rate of $4 \cdot 10^{-4}$ s^{-1}. In the second step (Fig. 1b), the torsion of the lower anvil with the strain rate of $4 \cdot 10^{-2}$ s^{-1} was applied to the sample.

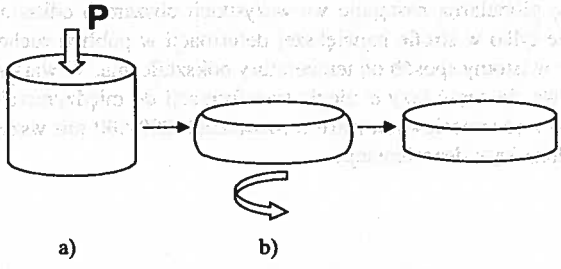


Fig. 1. Schema of deformation by upsetting and subsequent torsion

The level of upsetting and torsional strain was estimated with the equation (1) proposed by Degtyarev [8].

$$e = e_1 + e_2 = \ln(h_0/h_{iR}) + \ln(1 + (\varphi \cdot R/h_{iR})^2)^{1/2}, \quad (1)$$

where: h_0 is the initial height of the sample, h_{iR} is its height after processing at a distance R from the center, φ is the angle of rotation of the mobile anvil.

The strain of pure compression reached $e_1 = 1.4$. The contribution of torsion to the total amount of strain at a distance R/2 from the center is estimated to reach $e_2 = 4.5$.

The microstructure of the samples was examined in JXA 6400 SEM (Jeol). Thin foils were examined by CM 20 (Philips) TEM, operated at 200 kV.

Electron Back Scatter Diffraction (EBSD) analysis was realized by XL 30 ESEM (Philips) using the program Channel 5. Convergent Beam Electron Diffraction (CEBD) analysis was realized in TEM CM 20 (Philips) at 200 kV, by means of the *EP* program [9].

The X-ray analysis was carried out at the Roentgen Difractometer PW 1830 (Philips).

3. Results and discussion

The 25Kh15K alloy, after annealing in the temperature range of 700-850°C, represents a two-phase structure with γ lamellae in coarse α grains. An example of non-deformed structure is given in Fig. 2.

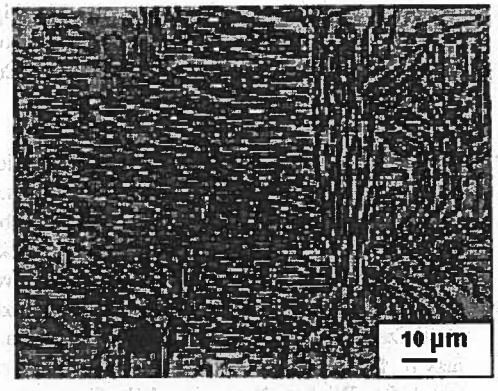


Fig. 2. The microstructure of non-deformed 25Kh15K alloy of ($\alpha + \gamma$) state, after annealing at 850°C

The application of upsetting and consequent torsion causes transformation of the lamellar structure into a granular one at all temperatures covered by the examination. It is necessary to note, that the structure of the samples was not uniform (Fig. 3a) due to the deformation technique. The lowest deformation occurred in their top parts, close to the immobile anvil (Fig. 3b). The highest deformation occurred near the mobile anvil, in the bottom parts of the samples (Fig. 3d).

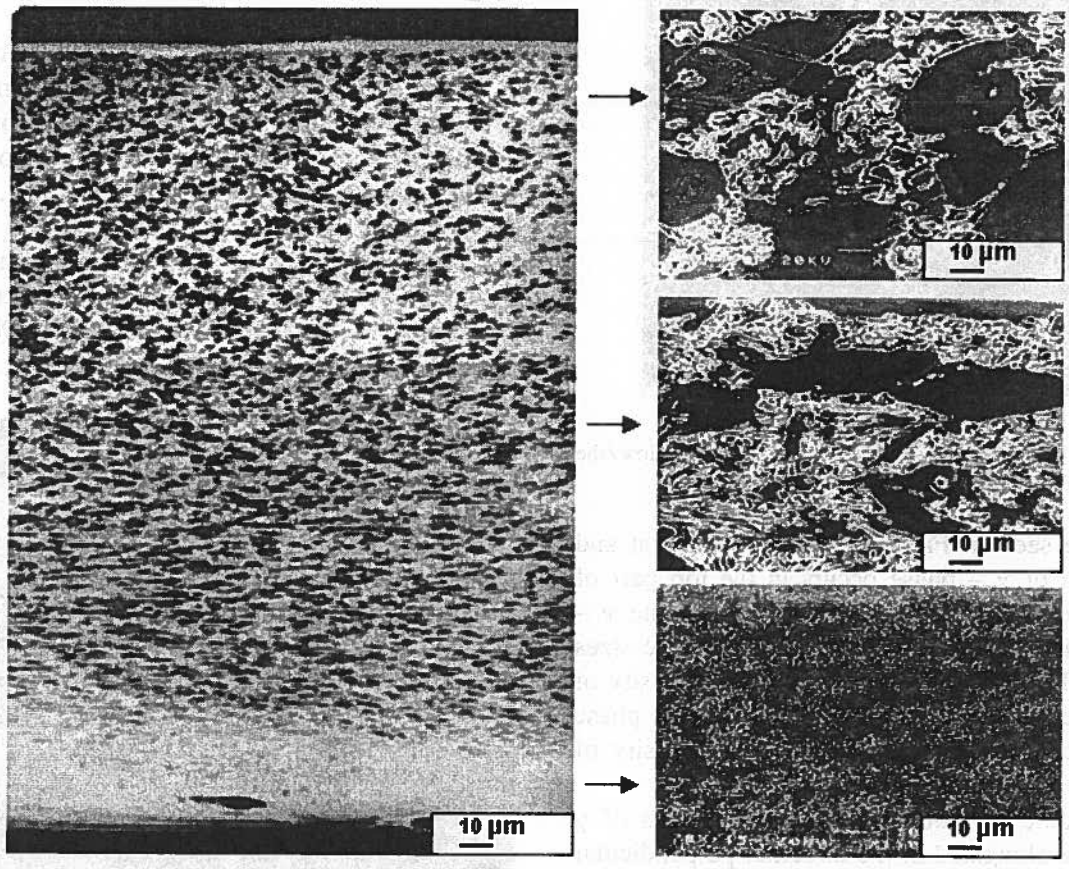
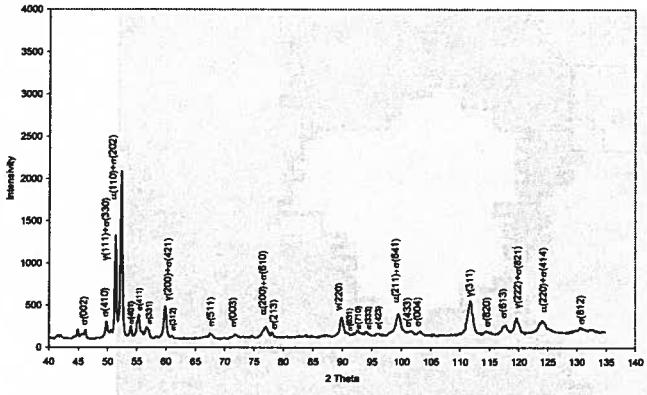


Fig. 3. Panoramic image of the cross-section of the sample deformed at 700°C (a) and detailed images of microstructure of the top (b), middle (c) and bottom (d) parts of the sample, SEM

a)



b)

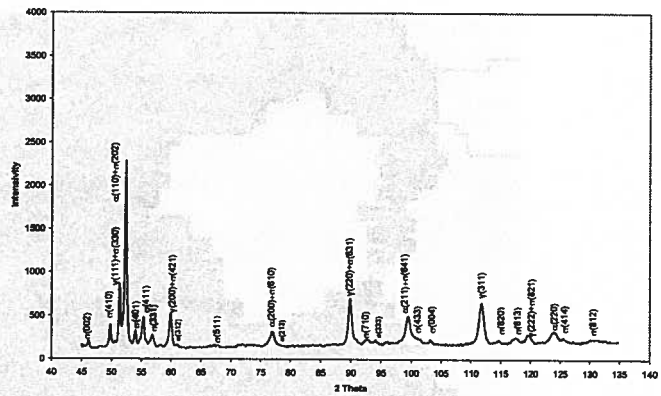


Fig. 4. X-ray analysis of the sample deformed at 700°C: a) the top part; b) the bottom part

The X-ray diffraction analysis of top (Fig. 4a) and bottom (Fig. 4b) parts of all samples shows that structure after deformation consists of three phases – α, γ and σ.

However, a slight increase of intensity of σ phase picks (4 1 0) and (4 1 1) in the bottom part of the samples can be observed. EBSD/SEM examination shows also the same phases. Fig. 5 shows, by way of example, a topography of phases measured in the top part of the sample; the estimated shares of α, γ and σ phases deformed at 700°C are here equal to ~20, ~50 and ~30%, respectively. It is possible, that the intermetallic σ phase is formed as a result of partial transformation of the α phase.

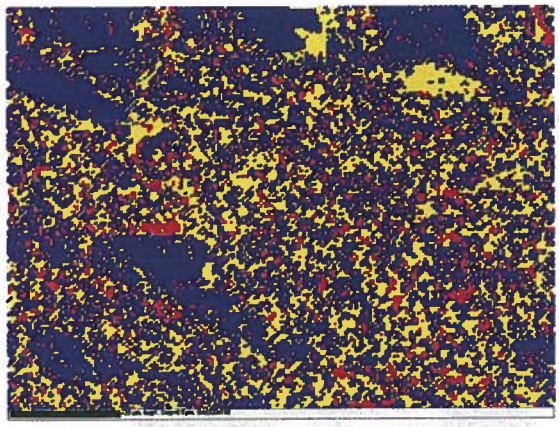


Fig. 5. The phase's topography; α – red, γ – blue, σ – yellow / the top part of the sample, deformed at 700°C, EBSD/SEM

As can be seen in Fig. 3b, the fragmentation and spheroidization of γ – phase occurs in the top part of the sample, deformed at 700°C. The grains of the γ – phase are rounded, only slightly elongated, of the sizes of 40-50 μm. They are characterized by high density of twins. More detailed TEM study shows that the α phase has partially cellular structure with a high density of dislocation.

In the middle part of the sample, the grains of γ phase are more elongated in the direction perpendicular to upsetting, and have longitudinal and transverse sizes of about 60 and 10 μm, respectively. Some fragmentation of γ – grains is also observed. The volume of cellular structured α-grains is here much more increased in comparison with the top part of the sample. The σ-grains have globular form, which is characteristic for σ phase generated as a result of deformation.

The most significant refinement of the structure occurs in the zones of the highest deformation, near the moving anvil (Fig. 3d). The foil, prepared from the bottom part of this sample, revealed sub-microcrystalline structure with equiaxial globular grains of α- , γ- and σ-phases with the average size of 200-500 nm. The structures of the samples processed at higher temperatures were similar to that observed at 700°C. For more detailed study of the bottom part of the samples CBED/TEM technique was used. By way of example, Fig. 6 shows

Fig. 6. The microstructure of the bottom part of the sample deformed at 850°C, TEM

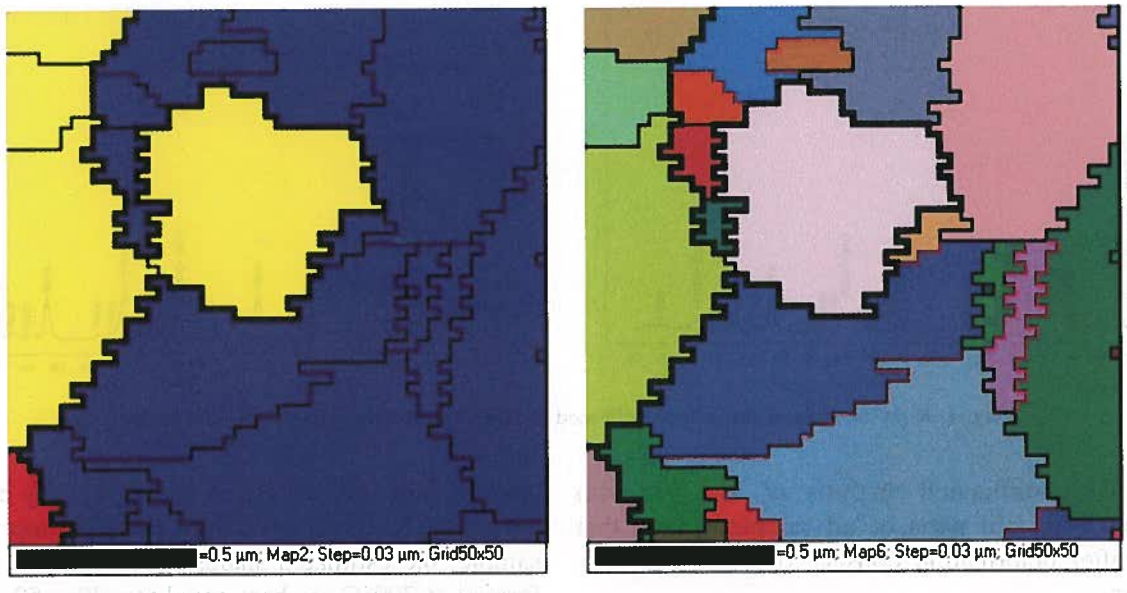


Fig. 7. The phase's topography; α – red, γ – blue, σ – yellow (a) and orientation map of the same place (b) / the bottom part of the sample deformed at 850°C, CBED/TEM

the fine structure bottom parts of the sample deformed at 850°C. All grains have here equiaxial globular forms with sizes of 200-500 nm.

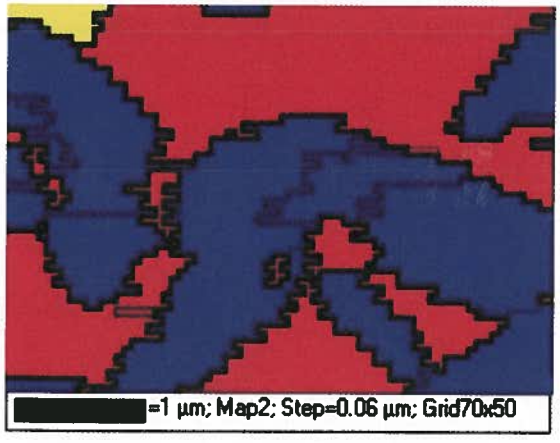


Fig. 8. Orientation map of the top part of the sample, deformed by 850°C, CBED/TEM

The topography of phases (Fig. 7a) shows that the three phases are also present. However, as it follows from measurements of four maps, in the structure of the bottom part the γ- and σ-grains are dominating. The orientation map (Fig. 7b) shows that most of the grains and interphase boundaries are characterized by high disorientation angles. The phase's topography of top part is shown in Fig. 8 for comparison with the bottom part. The grains are larger (see also the EBSD/SEM map Fig. 5); it seems also that the amount of σ-grains is lower.

The thickness of the layer (h_{smc}) with sub-microcrystalline structure in the bottom part of the sample varies from 570 μm at the temperature of 700°C to 700 μm at 850°C (table 2). The volume fraction (V_{smc}) of sub-microcrystalline structure increases with the rise of the loading temperature.

TABLE 2

The thickness of SMC layer of 25Kh15K alloy after complex loading at different temperatures

T_{def}, °C	h_{smc}, μm	h_{sample}, μm	V_{smc}, %
700	570	3800	15
750	700	3800	19
800	700	3000	23
850	700	2700	26

The measuring of microhardness [10] shows, that the value of microhardness is not uniform in all samples. The maximum value of microhardness was observed at the bottom of all the samples, where sub-microcrystalline structure is formed.

4. Conclusions

- Complex two-step loading of 25Ch15K alloy, including upsetting and torsion, provides transformation of a coarse lamellar structure into granular sub-microcrystalline structure for all examined temperatures. The maximum structure refinement occurs in the layer located close to the mobile anvil leading to grains with the average size of 200-500 nm.
- The thickness of SMC layer slightly depends on temperature.
- The process may be used for the surface treatment (hardening) of bulk specimens.

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