A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
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THE EVOLUTION OF THE MICROSTRUCTURE OF HARD MAGNETIC FeCr30Co8 ALLOY SUBJECTED TO PLASTIC DEFORMATION BY A COMPLEX LOAD

ROZWÓJ MIKROSTRUKTURY TWARDYCH MAGNETYCZNIE STOPÓW FeCr30Co8 PODDANYCH ODKSZTAŁCENIU W ZMIENNYM STANIE OBCIĄŻEŃ

The structural evolution of hard magnetic FeCr30Co8 alloy in the α state after deformation by upsetting and subsequent torsion was studied. The temperatures (750, 800, 850, 900 °C) and deformation rates corresponded to the condition of superplasticity of Fe-Cr-Co alloys. A gradient microstructure was formed in the sample sections, parallel to the direction of upsetting, because the torsion deformation was applied only to the bottom parts of samples. Particular analysis of microstructure by SEM/EBSD method showed that dynamic recovery with formation of subgrain microstructure took place during deformation. The deformation is also conducive to the precipitation of intermetallic σ phase in the temperature range from 750 to 850 °C. The maximum refinement of microstructure and the maximum precipitation of σ phase are observed at the temperature of deformation of 800 °C (the minimal sizes of α and σ grains are 5 and 2 μm, respectively). The refinement of the microstructure and the precipitation of σ phase resulted in an increase of hardness of the material.

Keywords: hard magnetic alloy, high temperature deformation, gradient microstructure, EBSD/SEM, interface segregation

Badano zmiany mikrostruktury jednofazowego stopu α FeCr30Co8, który został odkształcony przez spęczanie i następujące po nim skręcanie. Zastosowane temperatury (750, 800, 850 i 900 °C) oraz prędkości odkształcenia odpowiadały warunkom nadplastyczności stopów układu Fe-Cr-Co. Ze względu na to, że skręcanie dotyczyło tylko dolnej części próbek, w ich poprzecznych przekrojach powstała mikrostruktura o charakterze gradientowym. Jej szczegółowa analiza metodą SEM/EBSD wykazała, że w czasie odkształcenia w materiale zachodzi zdrowienie dynamiczne i tworzy się struktura podziarnowa. Intensywne odkształcenie w zakresie temperatur 750 – 850 °C sprzyjało także wydzielaniu się międzymetalicznej fazy σ. Maksymalne rozdrobnienie mikrostruktury oraz maksymalne wydzielanie się fazy σ obserwowano w temperaturze odkształcenia 800 °C (minimalny rozmiar ziarn faz α i σ wynosi odpowiednio około 5 i 2 μm). Rozdrobnienie mikrostruktury i wydzielenie fazy σ powoduje wzrost twardości materiału.

1. Introduction

Fe – Cr – Co based alloys belong to the deformable magnetic materials of the precipitation-hardening class [1]. Due to their good ductility, excellent magnetic properties and low cost, they are used for the production of permanent magnets of various sizes and shapes, such as wire, tube, bar, strip magnets, etc [2, 3]. The high coercive state is obtained in the process of decomposing a solid α solution into isomorphous α_1 and α_2 phases, the precipitates of which are ordered and coherent. Formation of such a structure, however, results in a reduction in material plasticity and strength [1]. It is known that intensive deformation improves mechanical properties of materials [4, 5]. For example, the intensive deformation of FeCr25Co15 alloy by complex load method (upset followed by torsion) allows to increase the plasticity and strength of deformed samples in comparison with the high coercive state [6]. Besides, a gradient microstructure with a layer of submicron grains was formed in the deformed sample sections parallel to the upsetting direction. It should be emphasized that the formation of such microstructure can be advantageous in magnets, which rotate with high speed and need to have good mechanical properties on the surface as well as good magnetic properties inside the material.

FeCr30Co8 alloy is a new alloy and its advantages include a low price as well as the possibility of satura-

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tion of α solid state from lower temperature and relatively low temperature of magnetic treatment. This paper presents the results of microstructure investigations and measurements of hardness of the hard magnetic FeCr30Co8 alloy after upset followed by torsion.

2. Materials and research methods

The chemical composition of examined alloy is presented in Table 1. The cast alloy was subjected to homogenization at 750 °C to obtain a solid α solution. Cylindrical samples, 10 mm in diameter and 10 mm high, were cut off from the ingot. Severe plastic deformation was achieved in an appropriately modernized "Instron" machine, which ensured the conditions for superplastic deformation. In the research presented in this paper, the deformation was achieved in two separate stages: upsetting and then torsion applied to the lower part of samples (Fig. 1).

TABLE

Chemical composition of FeCr30Co8 alloy

Cr	Co	Ti	V	Si	Fe
30	8	1	0.3	0.4	Remainder

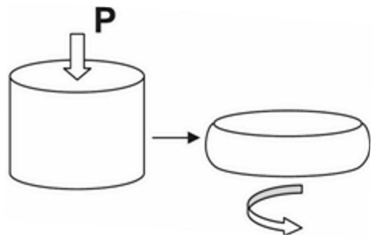

Fig. 1. Scheme of deformation by upset, followed by torsion. P refers to the deformation load

The temperatures 750, 800, 850 and 900 °C were chosen for the deformation of the α phase. Samples were subjected to upsetting at the rate of 0.34 mm/min to obtain the deformation of 40 % and followed by torsion (0.25 turns/min) up to 10 rotations. The torsion deformation was applied only to bottom parts of the samples. The friction force between lower sample part and bottom anvil provided the plastic deformation. The above temperatures and deformation rates correspond to the superplasticity conditions of the examined alloy.

The degree of upsetting was calculated based on the following formula (1):

$$\varepsilon = ln(h_0/h_{iR}), \quad (1)$$

where: h_0 is the sample thickness before deformation and h_{ik} is the sample thickness after deformation at distance R. The degree of upsetting reached 0.43 – 0.58 (depends on the temperature deformation).

The microstructure was examined by means of scanning (Jeol JXA 6400 and FEI XL 30 ESEM) and transmission electron microscopy (Philips CM 20). The EBSD analysis was performed in the FEI SEM XL 30 ESEM. The convergent beam electron diffraction (CBED) analysis was carried out with the Philips TEM CM 20. X-ray diffraction analysis was done with the Philips PW1710 diffractometer using Cu $K\alpha$ radiation. The microhardness was measured with a CSM device at the load of 1 N. The hardness measurements were carried out on sample cross-sections along the upsetting direction, at half-distance from the rotation axis.

3. Results and discussion

Microstructure

The investigations of microstructure of alloy FeCr30Co8 after annealing at the temperatures of 750, 800, 850 and 900 °C for 30 minutes, carried out by means of the methods of optical microscope and X-ray diffraction analysis, showed that the initial state before deformation was the α solid solution. The example of microstructure after annealing at 800 °C and quenching in the water is showed in Fig. 2. The grain sizes of α phase are about 40, 50, 70 and 300 μm for the temperatures of 750, 800, 850 and 900 °C, respectively. It could be noted, that the σ phase according to its stability equilibrium range can occur at the annealing temperatures of 750 and 800 °C, but the process of its precipitation is very long-lasting (it can require the several days of annealing) [7].

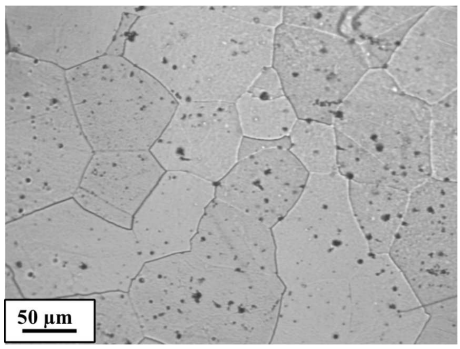

Fig. 2. Initial microstructure of the α solid solution after annealing at 800 °C

The curves of deformation are presented in Fig. 3. These curves are responding only to axis stress during the upsetting deformation. At the moment of transition to the torsion stage, the upsetting stopped and axis stress decreased sharply. It can be observed that the transition

of the material to the plastic flow stage occurs faster when temperature increases. For example, the plastic flow stage begins at the logarithmic degree of deformation of 0.1 and 0.05 at the deformation temperature of 750 and 900 °C, respectively. It can also be seen, that the stresses corresponding to 750 and 800 °C are twice higher than the ones at 850 and 900 °C. This can be explained by the operation of different mechanisms of deformation at those temperatures. The maximum stress occurs at 800 °C and reaches about 45 MPa.

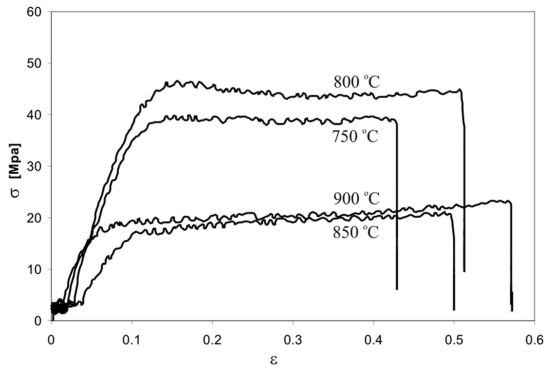

Fig. 3. Compression curves of samples deformed at 750, 800, 850 and 900 °C

The microstructure investigations of deformed alloy by SEM showed that the resultant microstructure was of gradient character, with the largest grain refinement in the lower part of the sample, where the deformation was the greatest. Figure 4a shows the panoramic image of the cross section of the sample deformed at 750 °C and detailed images of microstructure of the upper, middle and bottom parts of the sample (Fig. 4b-d). It is clearly visible in Fig. 4b that the subgrain microstructure is formed during deformation as a result of the process of dynamic recovery. The precipitation of σ phase with grain size of about 5 μm is also observed in the bottom part of this sample. The sample deformed at 800 ° has larger microstructure refinement and the precipitation of σ phase is observed in the entire cross section, with the maximum amount in the bottom part of the sample (Fig. 4e-g). The precipitations of σ phase are smaller at the deformation temperature of 850 °C (Fig. 5a-c) and they are absent in the sample deformed at 900 °C (Fig. 5d-f). The X-ray analysis of the bottom part of the sample deformed at 800 °C is showed in Fig. 6. It is possible to claim that the deformation activates the precipitations of σ phase, because the maximum precipitation of σ phase is observed in the places of intensive deformation. It may be that the maximum precipitations of σ phase in the sample deformed at 800 °C, as compared with the sample deformed at 750 °C, is caused not only by the deformation of microstructure, but also by stronger thermal activation of diffusion processes. The decrease of the σ phase precipitations at 850 °C and its absence at 900 °C are due to the fact, that the σ phase (according to phase equilibrium diagram) is no longer stable at these temperatures.

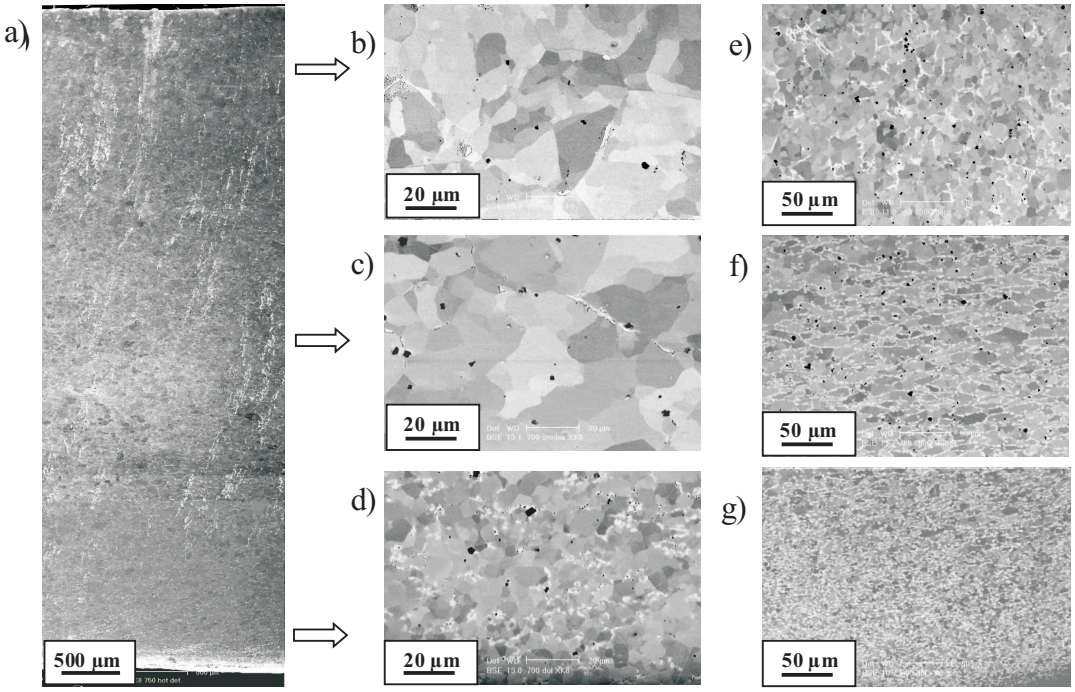

Fig. 4. (a) The microstructure in the entire cross-section of the sample deformed at 750 °C, SEM; (b) the upper sample part; (c) the middle sample part; (d) the lower sample part. (e), (f), (g) – the upper, middle and lower parts of the sample deformed at 800 °C

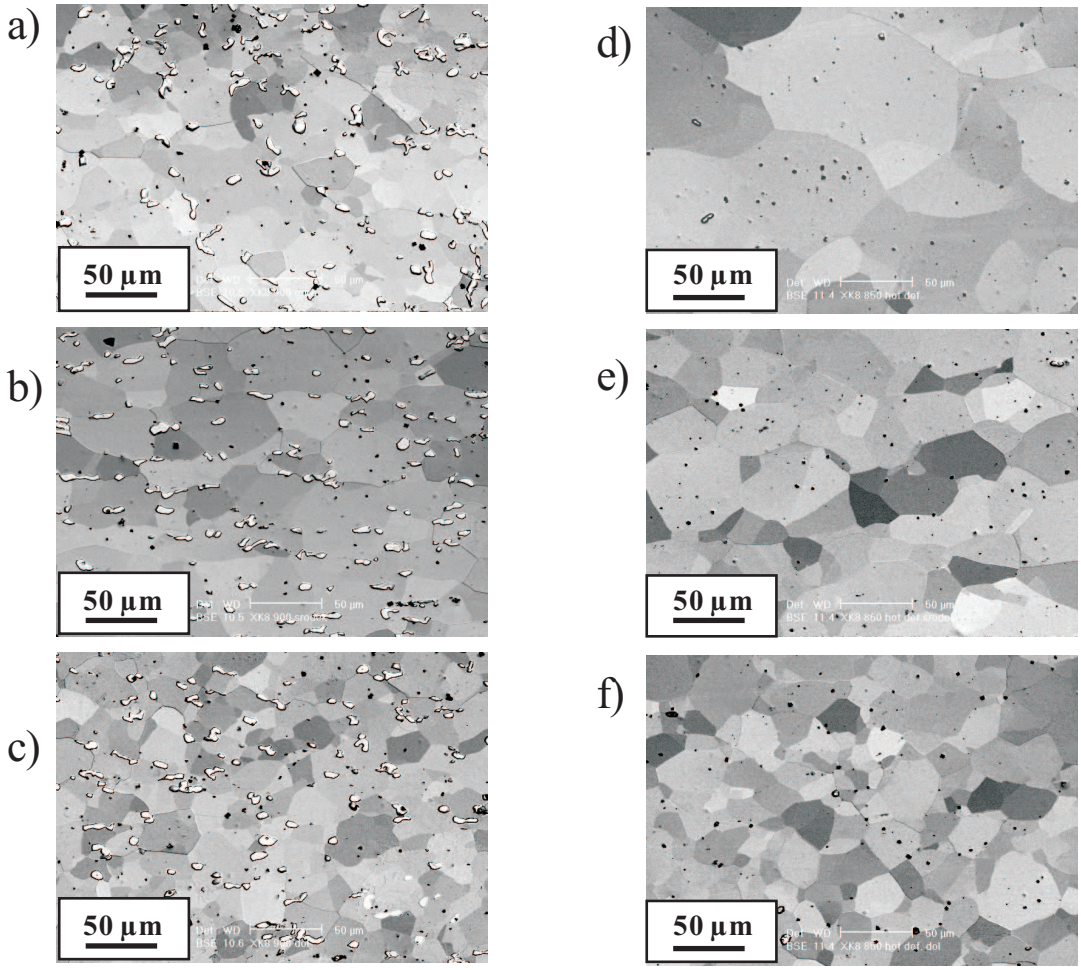

Fig. 5. (a), (b), (c) – the upper, middle and lower parts of the sample deformed at 850 °C (d), (e), (f) – the upper, middle and lower parts of the sample deformed at 900 °, SEM

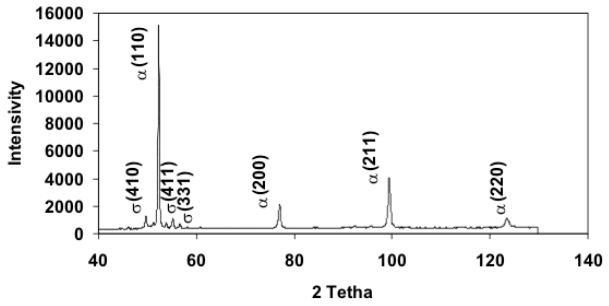

Fig. 6. X-ray phase analysis for the bottom part of the sample deformed at 800 °C

It is possible, that the precipitation of σ phase promotes the refinement of microstructure. The σ phase precipitates on the grains boundary of α phase block the movement of dislocations and therefore the transmission of deformation from one grain to another one is more difficult. Then, the density of dislocations increases inside of α grains, which results in the refinement of grains. The sizes of α grains in the bottom part of the sample deformed at 800 °C are of about 5 µm, while the thickness of microcrystalline layer is of about 260 µm.

The analysis of low angle grain boundaries (LAGB) in individual grains by the EBSD method showed that angles of disorientation of subgrains in the upper parts of samples deformed at 750, 900 °C are smaller than one degree and do not exceed four degrees in the samples deformed at 800, 850 °C. Therefore, the subgrain microstructure is weakly developed in the upper parts of the samples. The increase of disorientation angles up to 8 – 11 ° is observed in the bottom parts of the samples. The disorientation angle analysis of individual grains for the upper and bottom parts of the sample deformed at 700 °C is showed in Fig. 7 as the example.

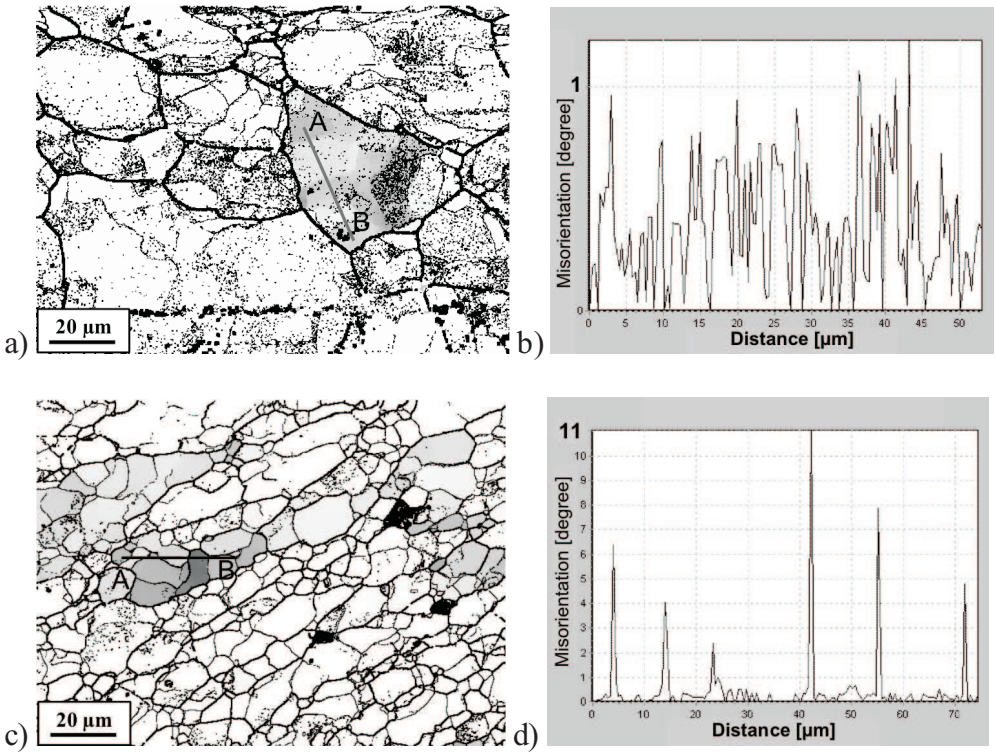

Fig. 7. Topographies of grain boundaries (a, c) and disorientation angle distributions (b, d) along defined A-B lines in individual grains for the upper (a, b) and bottom (c, d) parts of the sample deformed at 700 °C

The comparison of disorientation angle distributions in the top and bottom parts of samples shows that the share of LAGBs with disorientation angles of 8 – 12 ° increases in the bottom parts and the share of high angle grain boundaries (HAGB) increases as well. Therefore, one can observe intensive development of subgrain microstructure as well as the grain refinement in the bottom parts of the samples, particularly at the temperature of deformation of 750 – 850 °C. The share of LAGBs decreases in the top parts of the samples, when the deformation temperature increases. It can be connected with the decrease of defect density at higher temperatures. However, this effect is not observed in the bottom parts of the samples, where the share of LAGBs does not change with the increase of the deformation temperature. In general, the share of LAGBs is higher than the share of HAGBs for all deformation temperatures.

The microstructure analysis of thin foils from the bottom part of the sample deformed at 800 °C (Fig. 8a) showed that the σ phase generally precipitates at triple points (joints), which can be the source of nucleus of a new σ phase. The dislocation of α grains accumulates into dislocation walls and forms the low grain boundary (Fig. 8b). The minimum size of α grains is of about 2 µm.

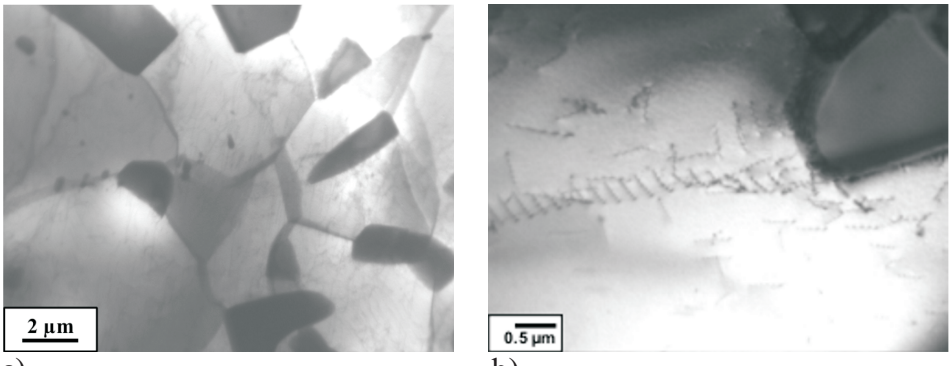

Fig. 8. The microstructure of the bottom part of the sample deformed at 800 °C, TEM (a,b)

Hardness measurement.

The hardness measurement of deformed samples shows that the maximum changes of hardness are observed in the sample deformed at 800 °C (Fig. 9). A considerable increase of hardness in its bottom part is caused by the microstructure refinement and the presence of hard σ phase in the material.

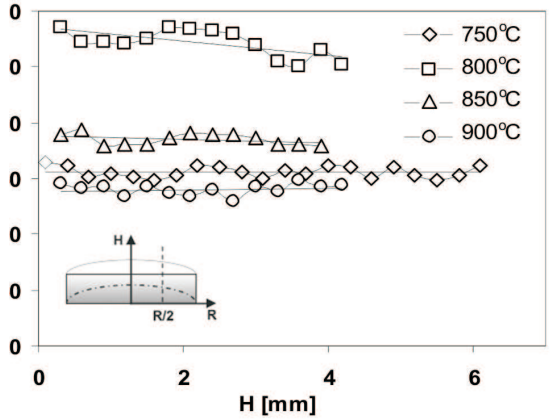

Fig. 9. Hardness measurement diagram and hardness graphs; all measurements carried out on a cross-section at the distance of half a radius from the sample axis

4. Conclusions

1. The deformation of FeCr30Co8 alloy by the complex load method in the conditions of superplasticity results in the formation of gradient microstructure in sample cross sections, with the minimum grains sizes in the bottom part of samples.
2. Dynamic recovery accompanied by the formation of subgrain microstructure developed during deformation.
3. The deformation activates the precipitations of σ phase in the temperature range from 750 to 850 °C. The maximum precipitations of σ phase are observed at 800 °C.
4. The precipitation of σ phase promotes the refinement of α grains.
5. The process may be used for the surface treatment (hardening) of bulk specimens.

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REFERENCES

[1] A. V. Korznikov, Structure and Mechanical Properties of Metals and Alloys Subjected to Severe Plastic Deformation, Thesis, IPMS Ufa, Russia (2000).
[2] X. Y. Sun, C. Y. Xu, L. Zhen, R. S. Gao, G. G. Xu, J. Magn. Magn. Mater. **283**, 231-237 (2004).
[3] Z. Ahmad, A. ul Hag, S. W. Husain, T. Abbas, Physica B **321**, 96-103 (2002).
[4] H. Gleiter, Nanostructured materials: basic concepts and microstructure, Acta Mater. **48**, 1-29 (2000).
[5] R. Z. Valiev, I. V. Aleksandrov, R. K. Islamgaleev, Prog. Mater. Sci. **46**, 103-189 (2000).
[6] A. V. Korneva, M. Bieda, G. F. Korznikova, K. Sztwiertnia, International Journal of Materials Research **9**, 991-998 (2008).
[7] K. Yano, K. Abiko, Phys. Stat. Sol. A **160**, 449 (1997).

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