ISSN 1027-5495. Functional Materials, 26, No.2 (2019), p. 233-237.
doi:https://doi.org/10.15407/fm26.02.233

Effect of hydrostatic extrusion on structural and magnetic properties of polycrystalline Dy

N.A.Chernyak, V.I.Sokolenko, A.V.Mats

NSC "Kharkiv Institute of Physics & Technology",
1 Akademicheskaya St., 61108 Kharkiv, Ukraine

Received January 15, 2019

Electron microscopy and X-ray diffraction methods have been used to investigate special features of polycrystalline Dy transformation (under severe plastic deformation at hydrostatic extrusion conditions) into a submicrocrystalline aggregate with a strongly pronounced texture. The deformation texture emergence correlates with the process of subdivision microcrystallites, reducing the size of coherent scattering regions. The field dependence of magnetization, a saturation magnetization value of completely grain-oriented Dy extrudates coincide with those for a Dy single crystal with imposition of the field along the direction one of $b\langle 10\bar{1}0\rangle$ axis. The extruded Dy may be successfully used for fabricating bulk magnetic field concentrators.

Keywords: Dy extrudates, severe plastic deformation, submicrocrystalline structure, favorable texture, high magnetic properties.

Методы электронной микроскопии и рентгеновской дифракции использованы для исследования особенностей превращения поликристаллического Dy (при интенсивной пластической деформации в условиях гидростатической экструзии) в субмикрокристаллический агрегат с сильно выраженной текстурой. Формирование текстуры деформации коррелирует с процессом разбиения микрокристаллитов, уменьшения размеров областей когерентного рассеяния. Полевая зависимость намагниченности, величина намагниченности насыщения полностью текстурированных экструдатов Dy совпадают с магнитными свойствами для монокристалла Dy с наложением поля вдоль направления одной из $b\langle 10\bar{1}0\rangle$ осей. Гидроэкструдированный Dy может быть эффективно применен для производства массивных концентраторов магнитного поля.

Формування деформаційної структури у полікристалічному Dy під час інтенсивної пластичної деформації в умовах гідроекструзії. *М.О.Черняк, В.І.Соколенко, О.В.Мац.*

Методи електронної мікроскопії та рентгенівської дифракції були використані для дослідження особливостей перетворення полікристалічного Dy (при інтенсивній пластичній деформації в умовах гідростатичної екструзії) у субмікрокристалічний агрегат з сильно вираженою текстурою. Формування текстури деформації корелює з процесом розбиття мікрокристалітів, зменшення розмірів областей когерентного розсіювання. Польова залежність намагніченості, величина намагніченості насичення повністю текстурованих екструдатів Dy збігаються з магнітними властивостями для монокристала Dy з накладенням поля вздовж напрямку однієї з $b\langle 10\bar{1}0\rangle$ осей. Гідроекструдований Dy може бути ефективно застосований для виробництва масивних концентраторів магнітного поля.

1. Introduction

Ferromagnetic metals having a high saturation magnetization value form the basis of nearly all magnetic materials employed as magnetic flux concentrators or specially-shaped pole pieces. If the need arises to minimize overall dimensions of mechanical articles, it appears most efficient to use rare-earth metals (Dy, Ho, Er, Tb), which have high saturation induction values as compared to ferromagnetics of the Fe group [1].

It is known that the polycrystalline Dy was used to fabricate a magnetic field concentrator for enhancement the field in the bore of the hybrid magnet [2]. The anisotropy of mechanical properties of Dy (the highest plasticity in the basal plane) allowed one to produce (by cold rolling to foils with recrystallization anneals) a structure with enhanced magnetization along the rolling direction [3]. Feasibility of using Dy foils for fabricating magnetic field concentrator was studied in works [4, 5].

The advisability of forming a favorable deformation texture by cold pressing of items with the use of high-pressure fluid has been exemplified by polycrystalline low-temperature ferromagnetic Dy samples [6]. The parameter optimization of the method, which involved plastic deformation by hydroforming of workpieces transferred to a high-plasticity state, has made it possible to obtain bulky section-homogeneous concentrators during one of pass with assigned high-level magnetic properties that ensure stable service performance with regard to multiple temperature and magnetic cycling [7, 8]. It is this fact that has provided a way of developing a variety of devices with strong uniform magnetic fields, applicable for experimental studies into critical parameters of superconductors, properties of atomic hydrogen [9, 10]. However, the specifics of controlling the structure transformation with the given method of external action are still not clearly understood, and the needed information on the mechanisms of texture formation is missing. In view of this, the aim of the present work has been to investigate the structural and magnetic properties evolution of polycrystalline Dy under severe plastic deformation at hydrostatic extrusion conditions, followed by annealing.

2. Experimental

In the present experiments, the impurity content (Ho, Ni, Co, Zr, Tb) of Dy was no more than 0.1 %. The optimal process of hydrostatic extrusion was realized at pressures ranging from 5 to 8 kbar, depending on the workpiece diameter [8]. The samples for the magnetization measurements ($d \approx 17.25 \div 0.02$ mm in diameter and $l \approx 45.0$ mm in length) were cut from Dy rods using precision machining. After machining, the samples, were annealed in a vacuum ($T = 450...500°C$ for $t = 2 \div 3$ h).

The flat disks, cut by electrosparking in cross direction relative to the samples axis, were the subjects of electron-microscopic and X-ray diffraction studies. For final reduction in thickness, the samples were subjected to ion etching in vacuum.

The concentrator magnetization measurement procedure, with the error of method being less than 1 %, has been described in detail in [8]. The transmission microscopy was carried out using the electron microscope JEM-200CX. The X-ray diffraction studies were performed with the use of the diffractometer DRON-1.5 in the filtered (Ni β-filter) K_α 1,2-emission of the Cu anode. The dimensions of coherent-scattering regions (CSR) were estimated by the Selyakov-Scherrer equation.

3. Results and discussion

The structural changes observed in the polycrystalline Dy subjected to different modes of treatment are shown in Fig. 1. In the initial state, the material is characterized by the microstructure that comprises blocks of size 0.1 μm to 1 μm, with chaotic distribution of dislocations between the boundaries, the dislocation density being $\rho \approx 3 \cdot 10^9$ cm^{-2} (Fig. 1a). At the degree of strain $\varepsilon = 0.32$, the substructure is formed, which may be assigned by its morphology to the cellular type (Fig. 1b). The distance between the boundaries d varies between 0.1 and 0.3 μm, giving rise to azimuthal disorientations ω of the neighboring microscopic volumes equal to 0.5...1°. The dislocation density in the cell bodies amounts to $\rho \approx 2 \cdot 10^{11}$ cm^{-2}. It should be noted that broken boundaries are occasionally observed. These are typical disclination-type defects [11].

With the development of large plastic deformations in the range from $\varepsilon = 0.5$ to 1.0 the morphological features of fragmentation can be observed. Thus, at $\varepsilon = 0.916$, the angles of disorientation $\omega \approx 3°$ become typical, and fragments of transverse size $d \approx 0.1$ μm predominate (Fig. 1c). The increase in the degree of strain up to $\varepsilon = 1.61$ results in a severe fragmentation. The formed fragments of typical sizes between 0.04...0.2 μm are characterized by the dislocation density reduction in their central parts down to $\rho \approx (1-3) \cdot 10^{10}$ cm^{-2}, and also, by the increase in the azimuthal disorientations up to $\omega \approx 9°$ (Fig. 1d).

The X-ray studies have demonstrated that after deformation all the specimens

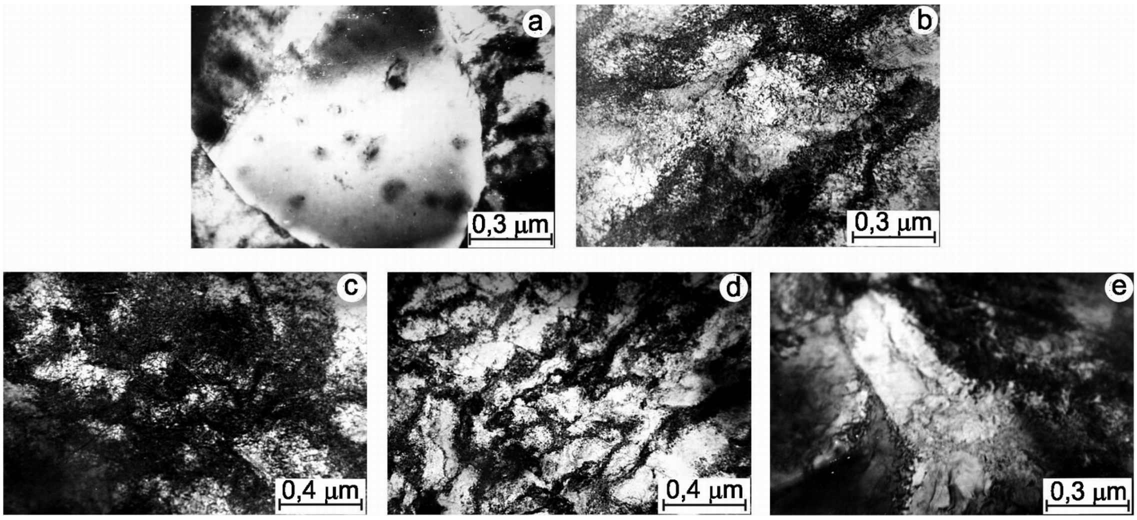

Fig. 1. Polycrystalline dysprosium structure in the initial state (a) and after hydrostatic extrusion: (b) $\varepsilon = 0.32$, (c) $\varepsilon = 0.916$, (d) $\varepsilon = 1.61$, (e) $\varepsilon = 0.916$ + annealing.

showed the occurrence of texture with the basal hexagonal-lattice planes arranged parallel to the extrusion direction. The examination of the fine crystalline structure has revealed the tendency to a significant CSR size reduction with an increasing degree of strain (Fig. 2).

Among the typical morphological features of the formed defect structure we mention the nonequilibrium interfragment boundaries. These substructural elements act as powerful sources of extended inhomogeneous stress fields. For relaxation of this stresses, which may adversely affect the performance characteristics, the mode of low-temperature (pre-recrystallization) annealing has been used, at which the general configuration of the structure undergoes no noticeable changes. Figure 1e shows the specimen structure created by combination of hydraulic pressing and a subsequent hot treatment ($T = 450°\mathrm{C}$, $t \approx 2.5$ h). It can be seen from the figure that the fragment sizes remain practically unchanged, whereas their boundary outlines become more clearly-defined. Without causing recrystallization, the annealing intensifies the motion of dislocations and their arrival at the boundaries, enhances the diffusion processes at the sites of high stress gradients, especially in near boundary regions. It is essential that in this case the energy losses due to hysteresis get reduced [6, 8].

To judge more precisely about the type of the formed deformation texture, we have measured the limiting values of concentrator magnetization in saturating longitudinal fields up to $H \approx 140$ kOe at 4.2 K. Figure 3 shows the measured magnetization of both the concentrator and the Dy single crystal, with imposition of the field along the $b\langle 10\bar{1}0\rangle$ axis. Good agreement between the $M(H)$ curves in the fields $H \geq 30$ kOe indicates that the deformation texture has formed in the magnetic flux concentrator, which can be identified as the $b\langle 10\bar{1}0\rangle$ type corresponding to the arrangement of a crystallographic direction $\langle 10\bar{1}0\rangle$ parallel to the longitudinal axis of the product.

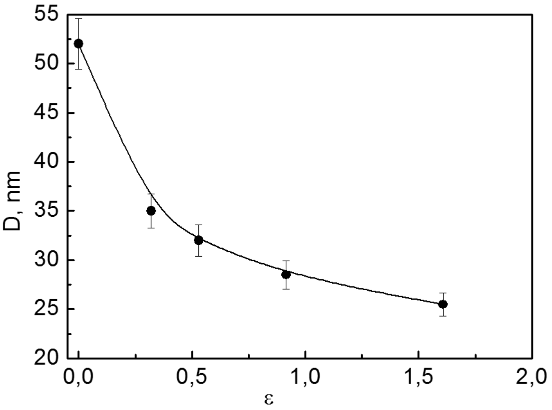

Fig. 2. Dimensional change of the CSR versus degree of strain.

Based on the measured data, we have constructed the plot, which features the regularities of texture development in polycrystalline Dy with the increasing degree of reduction (Fig. 4). The qualitative analysis of the experimental results leads to the conclusion that the texture formation correlates with the process of microcrystallites subdivision. In particular, a insignificant decrease in the size of the CSR in the inter-

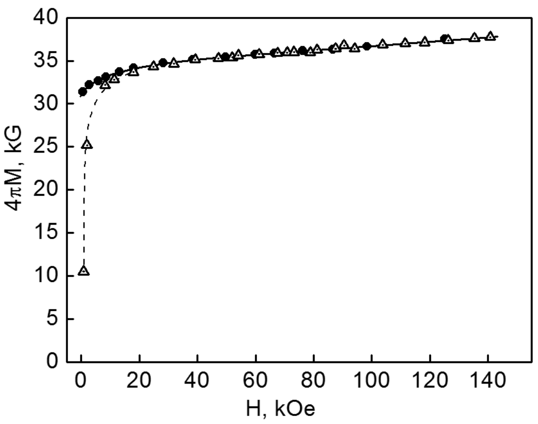

Fig. 3. Dy magnetizations at 4.2 K: textured polycrystals (Δ, $\varepsilon = 0.916$ + annealing), and a single crystal (•, [12]).

val $0.8 \le \varepsilon \le 1.6$ (Fig. 2) corresponds to the region where the maximum value of the magnetization is reached (Fig. 4).

Thus, the set of crystallographic orientations of submicrocrystallites causes the preferred texture of the Dy extrudates. The formation of preferred orientation specifies the physical anisotropy of completely grain-oriented Dy samples ($\varepsilon \ge 0.916$). The obtained magnetic flux concentrator shows appreciable electric conduction anisotropy by virtue of its structure and the orientation order of basal planes intercrossing in the same direction $10\bar{1}0$ [8, 13]. The absence of prismatic disorientation in Dy extrudates after $\varepsilon \ge 0.9$ has provided the possibility of creating the magnetic fields of extremely high homogeneity. This fact was confirmed by observation of magnetoresistance quantum oscillations of the Be single crystal, which are resolved only if the inhomogeneity of the magnetic field is less than $\Delta H/H \sim 5\cdot10^{-5}$ [8, 14].

We now consider some aspects of structure formation in the context of general ideas about large plastic deformations and self-organization of dissipative structures [11, 15]. It was established previously that the hydrostatic pressure level should substantially exceed the threshold value of Dy brittle-ductile transition [8]. The application of excessive load causes the increase in the intrinsic energy and interacting forces of dislocations, the accumulation of their density fluctuations. It is natural to assume that at the given conditions of loading, the material structure reaches the state being far from thermodynamic equilibrium. On reaching the threshold pressure value at plastic flow, the translational and rotation modes of plasticity are initiated. The main variations of defect structure observed after deformation are caused due to an increase in the density of plastic deformation carriers, the joint development of translational and rotational modes.

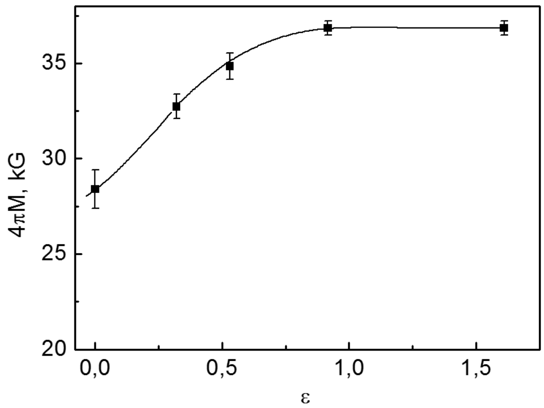

Fig. 4. Deformation effects on magnetization of dysprosium concentrators.

Perhaps, in a general way the process of texture formation one can to describe within the framework of model the visco-plastic self-consistent polycrystal model with disclination approach [16]. However, it is very hard to compare our results with texture predictions of this combined scheme. We note the following substantial matter. The development of structure fragmentation during plastic deformations is ensured by an increase in the density of partial disclinations. It is reasonable to suppose that providing the self-organization conditions makes possible the realization of the spatial dissipative structure arising in the partial disclinations system. The dynamical stability of spatially ordered structure is supported due to the supplied mechanical energy, provide the favorable texture formation at the sample scale level. At the same time an efficient channel of external force energy dissipation is in operation to prevent microcrack nucleation.

The obtained data and their analysis permit us to conclude that the method of formation of highly-textured polycrystalline Dy showing unique properties should be assigned to the class of technologies, based on the efficiency of dissipative structure self-organization processes to control structure formation [17]. The extruded products will be useful to prepare magnetic flux concentrators, the dimensions of which were varied as applied to the specified task.

4. Conclusions

It has been found that under severe plastic deformation at hydrostatic extrusion conditions, polycrystalline Dy transforms into a submicrocrystalline aggregate with a strongly pronounced texture characterized by high magnetization and performance properties.

The deformation texture formation correlates with the process of subdivision microcrystallites.

Substructure formation in polycrystalline Dy under large deformation is considered as a process of self-organization of dissipative structure arising in system of partial disclinations.

The strongly nonequilibrium state of the polycrystalline Dy structure is favorable for increasing the metal ductility resource through the straining scheme used which prevent microcrack nucleation.

References

1. K.N.R.Taylor, M.I.Darby, Physics of Earth Solids, Chapman and Hall LTD, London (1972).
2. D.Bird, S.Bole, I.Dixon et al., *Physica B*, **294–295**, 639 (2001).
3. M.Wayne, W.Swift, M.Mathur, *IEEE Transactions on Magnetics*, **10**, 308 (1974).
4. R.Agustsson, P.Frigola, A.Murokh et al., in: Proc. of Particle Accelerator Conf., New York, NY, USA (2011), p.1256.
5. R.Agustsson, Y.C.Chen, T.Grandsaert II et al., in: Proc. of IPAC, New Orleans, Louisiana, USA (2012), p.756.
6. B.G.Lazarev, A.A.Galkin, A.S.Bulatov et al., *Fiz. Metall. i Metalloved.*, **57**, 298 (1984).
7. B.G.Lazarev, L.S.Lazareva, N.A.Chernyak et al., *Cryogenics, ICMC Supplement*, **32**, 217 (1992).
8. B.G.Lazarev, L.S.Lazareva, N.A.Chernyak et al., *Phys. Met. Metallogr.*, **86**, 255 (1998).
9. B.G.Lazarev, O.V.Cherny, L.S Lazareva et al., *Cryogenics, ICMC Supplement*, **32**, 593 (1992).
10. E.Tjukanov, A.Ya.Katunin, A.I.Safonov et al., *Physica B*, **178**, 129 (1992).
11. A.E.Romanov, V.I.Vladimirov, Dislocations in Solids, ed. by F.R.N.Nabarro, Elsevier, Amsterdam, 9, 191 (1992).
12. J.J.Rhyne, S.Foner, E.J.McNiff, R.Doclo, *J. Appl. Phys.*, **39**, 892 (1968).
13. B.G.Lazarev, L.S.Lazareva, N.A.Chernyak et al., *Fiz. Metall. i Metalloved.*, No.5, 103 (1990).
14. B.G.Lazarev, N.A.Chernyak, L.S.Lazareva, in: Book Abstr. of the 17th All-Union Conf. on Physics of Magnetic Phenomena, Donetsk, Ukraine (1985), p.135.
15. I.Prigogine, I.Stengers, Order Out of Chaos, N.Y., Bantam (1984).
16. A.A.Nazarov, N.A.Enikeev, A.E.Romanov et al., *Acta Materialia*, **54**, 985 (2006).
17. D.Walgraef, Spatio-Temporal Pattern Formation, Springer, N.Y. (1996).