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Journal of Alloys and Compounds 230 (1995) 42–45

Journal of
ALLOYS
AND COMPOUNDS

Upset forged Nd–Fe–B–Cu magnets

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Received 24 February 1995; in final form 15 May 1995

Abstract

Cylindrical ingots of the alloy Nd₁₇Fe_{76.5}B₅Cu_{1.5} were upset forged in a crank press at an average strain rate of 0.8 s⁻¹ at temperatures of 750, 850 and 950°C. Microstructural development and magnetic properties are treated in terms of the material flow during the semi-solid deformation of the alloy as a function of the forging temperature. The results indicate increasing microstructural inhomogeneity due to the compaction flow at higher forging temperatures.

Keywords: Rare earth magnets; Hot deformed magnets; Upset forging; Semi-solid deformation

1. Introduction

An alternative processing route for sintered high-energy-product Nd–Fe–B magnets is the hot deformation of the cast alloy. Texture development is achieved by hot pressing, upset forging, hot rolling or extrusion, resulting in the magnetic alignment of the hard magnetic Nd₂Fe₁₄B phase. Essential processing parameters are: alloy composition, deformation temperature and strain rate. High deformations (around 80%) are necessary to obtain acceptable magnetic properties. The addition of Cu to the basic ternary alloy is also essential for good deformability and coercivity [1–3].

Since the hot deformation is possible only at temperatures where these alloys are in the semi-solid state, because of the coexistence of the solid Nd₂Fe₁₄B and the liquid Nd-rich phases, the problem of the liquid expulsion and/or redistribution in the volume of the material arises, resulting in inhomogeneity of the microstructure and magnetic properties of the magnet. Encasing the ingot in iron tubes or sheaths limits this expulsion [2], but the role of the strain rate and liquid phase volume fraction may be more important in obtaining a homogeneous microstructure in the volume of the material deformed in the semi-solid state [4].

2. Material and experiments

The Nd₁₇Fe_{76.5}B₅Cu_{1.5} alloy was induction melted from pure constituents under an argon atmosphere and solidified in iron molds to achieve a fine microstructure. Pieces of ingot were sealed in steel tubes by welding before they underwent upset forging in a crank press at 750, 850 and 950°C with a height reduction of 85%. The strain rate profile is not constant during the deformation cycle in a crank press so the average strain rate $\dot{\epsilon}_{av}$ was calculated as $\dot{\epsilon}_{av} = \epsilon/t_c$, where ϵ is the total deformation of the forged magnet and t_c is the time of the working cycle of the crank press. The calculated value of the average strain rate was 0.8 s⁻¹ for our experiments. The deformed ingots were oil quenched after forging in order to fix the as-forged microstructure. A post-deformation heat treatment at 1000°C for 5 h and 520°C for 2 h was applied to enhance the coercivity.

Quantitative metallography was employed to evaluate the influence of the forging temperature on the final microstructure in terms of the texture development, grain size, phase volume fractions, contiguity and microstructural homogeneity in the volume of the magnet. The magnetic alignment degree was expressed as the medium angle of the spread of the *c* axis of Nd₂Fe₁₄B grains from the direction of the height

reduction. For the latter measurements, the method described in [5] was applied using Kerr micrographs.

A vibrating sample magnetometer was employed for measuring the remanence J_r and the coercivity H_{cj} of the samples, parallel and perpendicular to the forging direction.

The Curie and the eutectic temperatures of the alloy were measured by a differential scanning calorimeter (DSC).

3. Results and discussion

The cylindrical ingots of the as-cast alloy showed the following microstructure: in the surface region a fine grained chilled zone, followed by a columnar region, as shown in Fig. 1; in the center of the ingot a restricted area of randomly oriented grains, shown in Fig. 2. After a heat treatment at 1000°C for 5 h and 520°C for 2 h this undeformed ingot had a remanence of 0.39 T and a coercivity of 143.2 kA m⁻¹ measured in the direction perpendicular to the direction of heat extraction. The Curie temperature was 309°C and the eutectic temperature 496°C. The latter is identical to the eutectic temperature in the ternary Nd–Fe–Cu system [6].

The microstructure of the as-forged rapidly under-cooled samples revealed the extensive fragmentation of the hard magnetic phase by a cleavage mechanism, Fig. 3. After heat treatment at 1000°C for 5 h and 520°C for 2 h the sharp edges of the fragments became rounded and the microstructure showed a high degree of texture development, as demonstrated by the Kerr micrographs in Fig. 4. Owing to the different volume fractions of the liquid phase at 750, 850 and 950°C, the conditions of texture formation were different. The results of the quantitative metallography showed that the homogeneity of the magnets, characterized by the differences in the microstructure in the volume of the magnet, deteriorated with the increasing forging temperature, see Table 1.

When these differences are interpreted in terms of the semi-solid metal forming, it can be deduced that forging the alloy at 750°C results in continuous flow and a homogeneous microstructure. Increasing the forging temperature increases the volume fraction of the liquid phase, so changing the conditions of the material flow. In the sample forged at 850°C significant differences already occur; partial expulsion of the intergranular Nd-rich phase to the peripheral areas of the deformed body occurs, resulting in a higher volume fraction of the intergranular phases in this region. A higher volume fraction of the Nd-rich phase means better separation of the grains of the hard magnetic phase and therefore lower contiguity here. The average grain size decreases slightly in the central area

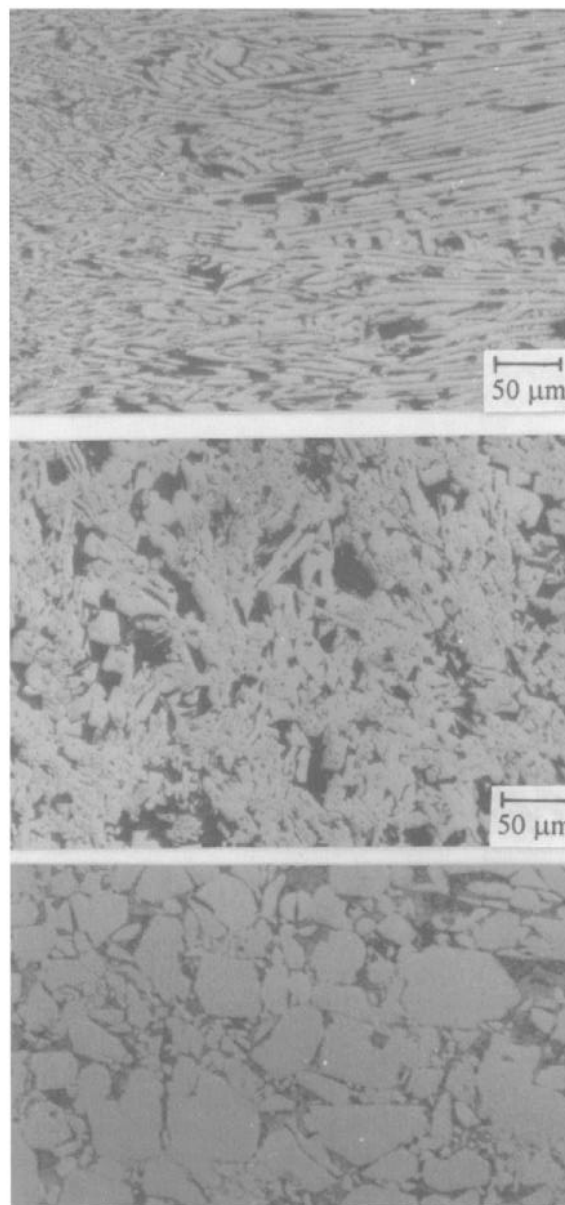


Fig. 1. Microstructure of the superficial region of the as-cast alloy (chilled and columnar zones).

Fig. 2. Microstructure of the central region of the as-cast alloy.

Fig. 3. As-forged microstructure of the magnet forged from 750°C.

with the increasing forging temperature due to the more efficient fragmentation having less intergranular liquid. However, the grains in the extremities tend to grow. The reason may be that the hard magnetic grains surrounded by the more abundant intergranular liquid can grow more freely by the Ostwald ripening mechanism. As for the microstructural alignment, it increases with the increasing forging temperature in the center (smaller medium angles) and decreases in the peripheral region (greater medium angles).

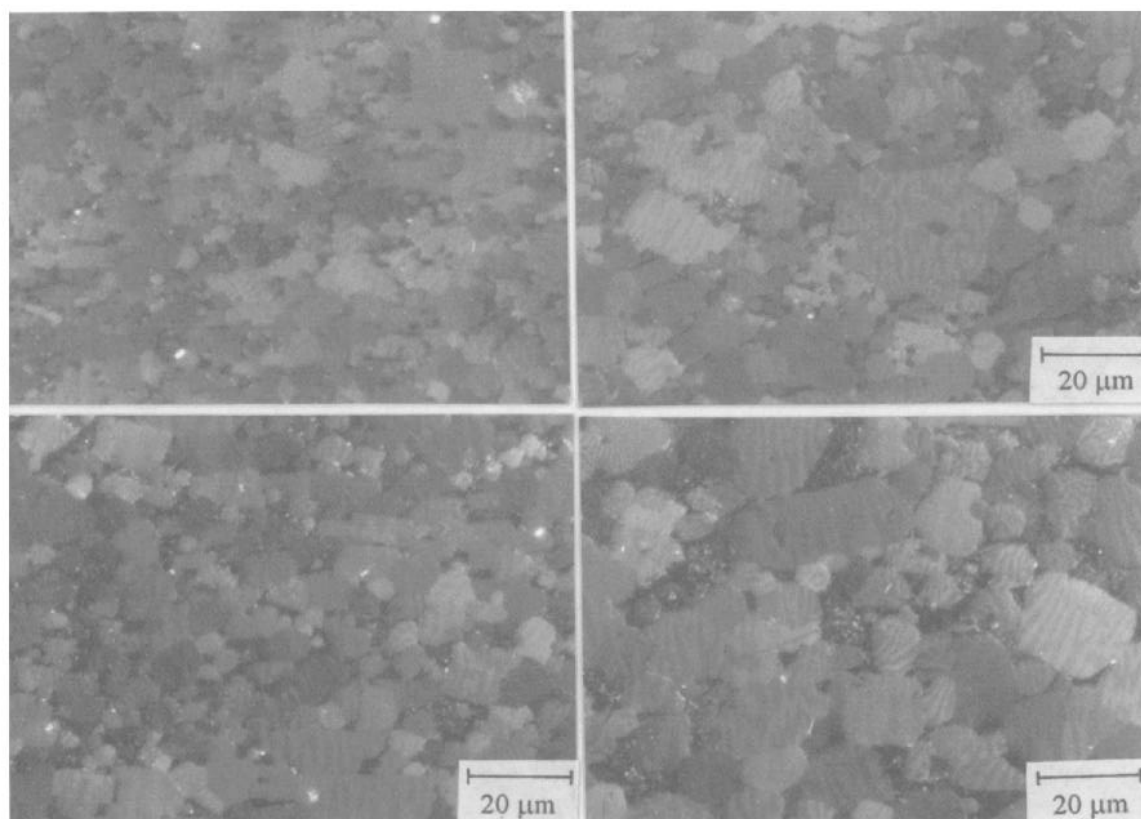


Fig. 4. Kerr micrographs of the heat treated magnets: (a) forged from 750°C, central region; (b) forged from 750°C, peripheral region; (c) forged from 950°C, central region; (d) forged from 950°C, peripheral region.

Table 1
Microstructural parameters of upset forged magnets

Forging temperature (°C)	750		850		950	
	Center	Extremity	Center	Extremity	Center	Extremity
Intergranular fraction (%)	15.2	15.4	13.2	16.4	10.2	17.6
Contiguity	0.41	0.39	0.43	0.37	0.51	0.23
Average grain size (μm)	7.7	8.5	7.4	10.2	7.1	10.0
Microstructural alignment (°)	15.2	15.5	14.7	26.1	12.8	26.7

Another method of expressing the degree of alignment is based on the anisotropy ratio defined as follows: $J_r / (J_{r\parallel} + J_{r\perp}) \times 100$, where $J_{r\parallel}$ and $J_{r\perp}$ are the remanences measured parallel and perpendicular to the forging direction respectively. Fig. 5 shows the demagnetization curves of the magnets cut from the central region and traced in these two directions. These curves and the calculated values of the anisotropy ratio, Table 2, also showed that the alignment increases with increasing forging temperature in the central region.

Comparing now the microstructural characteristics of the central region of the magnets (Table 1) with the magnetic properties measured for material in this region (Table 2) it will be obvious that the remanence increase with increasing forging temperature is caused

by both the increasing volume fraction of the hard magnetic phase and better alignment degree. The decrease of the coercivity occurs in spite of the increasing grain refinement, but the expulsion of the intergranular Nd-rich phase results in a poorer separation of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ grains and this effect seems to have a major impact. Compared with hot pressed [2] and hot rolled [7] NdFeB magnets these coercivities are still low.

The better squareness of the demagnetization curve of the magnet forged at 750°C shows that its energy product is almost on the same level as the magnet forged at 850°C, which had a higher remanence.

The upset forging of NdFeB magnets in a crank press enables high strain rates during the semi-solid deformation but at higher volume fractions of the

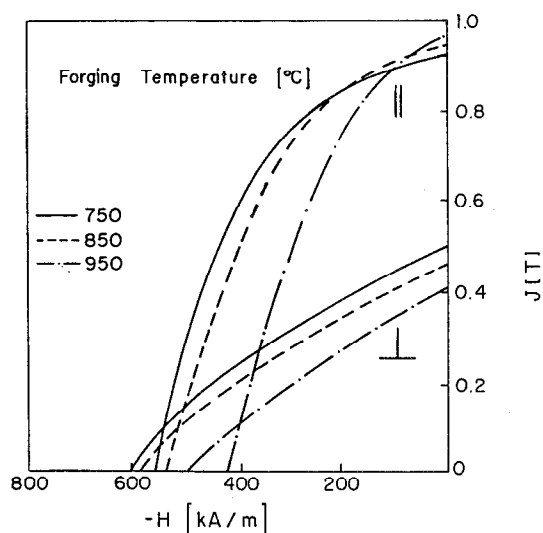


Fig. 5. Demagnetization curves of the forged magnets cut from the central region and traced parallel (||) and perpendicular (⊥) to the forging direction.

Table 2

Magnetic properties of the upset forged magnets measured in the central region

Forging temperature (°C)	750	850	950
Remanence J_r (T)	0.92	0.96	0.97
Coercivity (kA m)	559	528	421
Maximum energy product BH_{max} (kJ m ⁻³)	123	125	106
Remanence ratio (%)	69	71	74

liquid phase, i.e. at a higher forging temperature it results in a compaction flow mode characterized by the expulsion of the Nd-rich liquid. This results in lower coercivity; however the remanence increases. Hot working in conditions of homogeneous flow, where no expulsion of the liquid phase occurs, may result in homogeneous properties in the volume through the homogeneous microstructure. For different composi-

tions, by optimizing the processing parameters these conditions of deformation can be achieved.

4. Conclusions

Microstructural characteristics and magnetic properties of upset forged Nd₁₇Fe_{76.5}B₅Cu_{1.5} magnets were related and interpreted in terms of the material flow during deformation in the semi-solid state.

It was found that in the Nd₁₇Fe_{76.5}B₅Cu_{1.5} alloy homogeneous flow occurs when forged at 750°C, resulting in a homogeneous microstructure. Upon increasing the forging temperature (850 and 950°C) at the same strain rate ($\dot{\epsilon}=0.8\text{ s}^{-1}$) a transition to compaction flow with partial expulsion of the intergranular Nd-rich phase was observed. This resulted in different microstructures in the central and peripheral regions. The compaction flow caused higher remanence in the central region of the magnet but lowered the coercivity due the expulsion of the Nd-rich phase.

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