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Effect of Hot-compaction Temperature on the Magnetic Properties of Anisotropic Nanocrystalline Magnets

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The effect of the hot-compaction temperature on the microstructure and magnetic properties of anisotropic nanocrystalline magnets was investigated. The hot-compaction temperature was found to impact both the magnetic properties and the microstructure of die-upset magnets. The remanence of the isotropic precursor increases slightly with the improved hot-compaction temperature, and the grains start to grow on the flake boundary at higher hot-compaction temperatures. After hot deformation, it was found that the change in the magnetic properties was the inverse of that observed with the hot-compaction temperature. Microstructural investigation showed that die-upset magnets inherit the microstructural characteristics of their precursor. For the die-upset magnets, hot pressed at low temperature, scarcely any abnormal grain growth on the flake boundary can be seen. For those hot pressed at higher temperatures, however, layers with large equiaxed grains could be observed, which accounted for the poor alignment during the hot deformation, and thus the poor magnetic properties.

Keywords : Nd-Fe-B magnets, hot deformation, magnetic properties

1. Introduction

Hot pressed Nd-Fe-B magnets have been commercially available for a number of years. These fully dense magnets offer certain advantages over the more widely available sintered forms. Their nano-scale melt-spun microstructure has great environmental stability, together with the ability to be formed into intricate near-net shapes with isotropic or highly anisotropic magnetic properties. One particular application is backward extruded ring magnets or motor devices [1, 2]. Compared with their sintered forms, the back extruded rings show many advantages, such as high magnetic properties, small diameters and small wall thicknesses, *etc*.

The fabrication of anisotropic nanocrystalline magnets includes two steps: hot-compaction and hot deformation. Previous work mainly focused on the hot deformation process, especially the relationships between flow stress, stain rate, temperature and grain size, and so on [3-5]. The correlation between the hot-compaction temperature and the magnetic properties of the hot deformed magnets are seldom studied. Grünberger found the visible growth of regions with equiaxed coarse grains which started from flake boundaries (FB) during the course of the compaction and deformation process [5]. Volkov *et al.* found that quasi-periodic non-aligned "extended defect layers" were associated with the original ribbon interfaces, which were considered to play an important role in the high coercivity mechanism [6]. The textures and microstructures are closely connected to the domain structure and magnetic properties [7]. It is still unclear, however, how these equiaxed layers have come into being, and what kind of role they play on the magnetic properties. Therefore, we studied the change in the microstructure of the hot-deformed magnets by altering the hot-compaction temperature, in order to try to give a better understanding of these microstructual inhomogenities.

2. Experiment

The magnetic alloys used in this study with composition $Nd_{13.5}Fe_{80}Ga_{0.5}B_6$ were prepared by induction melting. Melt spinning was then used to make ribbons using a wheel speed 20-40 m/s. The melt-spun powders were hot-compacted at different temperatures 550 °C, 600 °C, 650

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°C, 700 °C, under 100-200 MPa in a vacuum. Then the isotropic precursors were hot deformed in an additional processing step under an argon atmosphere at 800 °C with a strain rate of 8×10^{-3} s^{-1} to 70% height reduction. The anisotropic specimens are referred to as A, B, C and D, respectively, corresponding to the isotropic precursors hot pressed from low temperature to high temperature.

The magnetic properties of the specimens were measured using hysteresis loops at room temperature after external magnetization in a pulsed field of 5 T. The microfractures were investigated with the help of a JMS-6400 scanning electron microscope.

3. Results and Discussion

The magnetic properties of hot-compacted specimens are shown in Fig. 1. It shows that both the remanence B_r and the maximum energy products $(BH)_m$ increase with increasing hot-compaction temperature. The coercivity H_{cj} reached its maximum at 650 °C. The magnetic properties of the anisotropic specimens are shown in Fig. 2. For the anisotropic specimens, A, B, C and D, both the remanence and the maximum energy products dropped, i.e. the inverse of the trend displayed by their precursors.

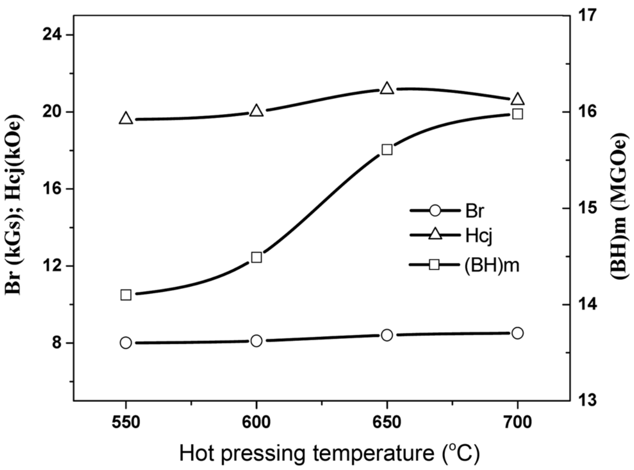

Fig. 1. Magnetic properties of hot-compacted specimens.

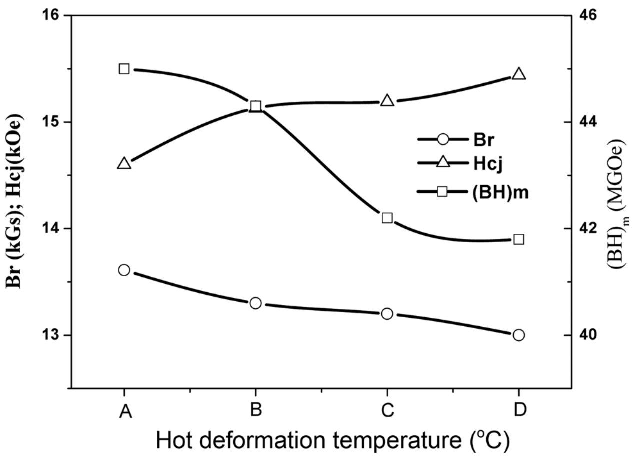

Fig. 2. Magnetic properties of die-upset specimens.

SEM images of the fracture surface of the isotropic precursor, hot pressed at 550 °C, are shown in Fig. 3(a). These showed that the grain size on the surface region of the flakes was small and homogeneous, due to the low hot-pressing temperature. For the SEM images of the isotropic precursor hot pressed at 700 °C, shown in Fig. 3(b), one particular phenomenon is observed, that the grains on one side of the flakes grow dramatically, which is considered to be related to the melt spinning of the flakes. During the cooling of the flakes, the flakes on the wheel side have a higher cooling rate, and the grain sizes are smaller. During consolidation of the flakes, those grains on the wheel side with smaller diameters are easier to grow.

SEM images of fracture surface for specimens A and D are shown in Fig. 4(a) and Fig. 4(b), respectively. For specimen D, two kinds of grains can be detected: elongated plate-like grains within the flakes, and equiaxed coarse grains between the flakes (with grain sizes of the order of 1 μm). For specimen A, the grains have small sizes and show an entirely plate-like morphology. The microstructures of the anisotropic die-upset specimens are closely connected with that of the isotropic hot-pressed

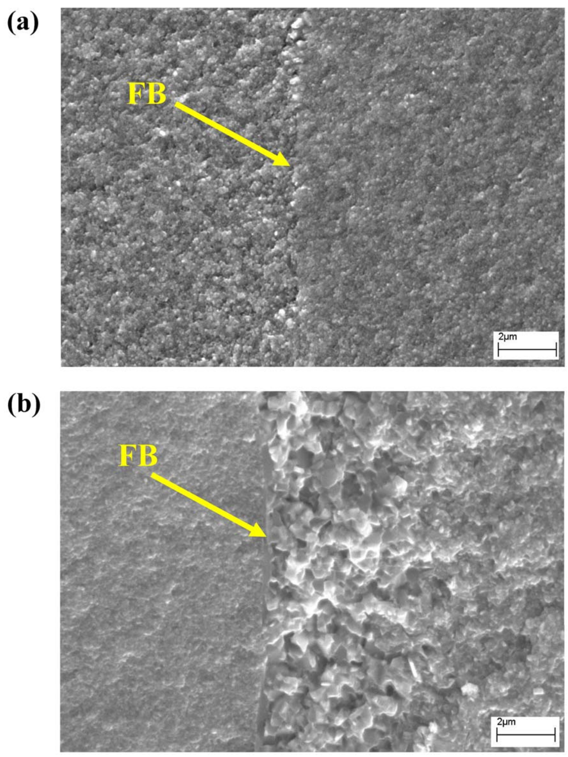

Fig. 3. (Color online) The fracture surface SEM images of isotropic precursor, hot pressed (a) at 550 °C, (b) at 700 °C.

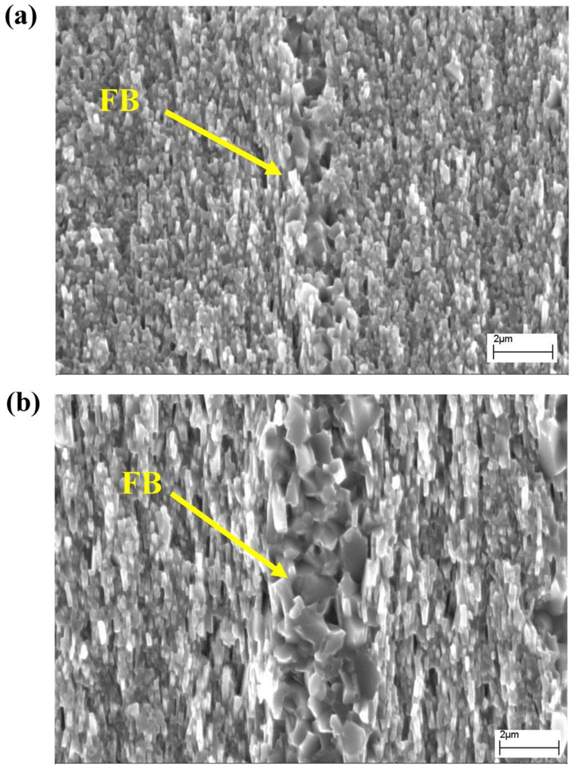

Fig. 4. (Color online) SEM images of the fracture surface of (a) specimen A, and (b) specimen D.

precursors. For the isotropic precursors, hot pressed at 700 °C, the grains are large in size on FB, which may lead to the equiaxed coarse grains in the anisotropic die-upset specimens.

The hot workability in terms of hot deformation was studied in the die-upset experiments. Fig. 5 shows the deformation behavior during die-upsetting for specimens A and D. Specimen A was characterized by a relatively low deformation stress of 30 MPa at the initial deformation stage. Specimen D, on the other hand, showed a relatively high deformation stress of 40 MPa. The large equiaxial grains on FB in specimen D may account for the high deformation stress during die-upsetting.

The different grain morphologies in die-upset magnets show different deformation mechanisms in the two regions. Previous studies showed that deformation and formation-of-texture processes can be explained by the solution-precipitation creep model, a combination of plastic deformation, grain boundary migration, and grain boundary sliding [8]. The plate-like grains are formed due to the anisotropy of grain growth during die-upsetting. The development of large equiaxial grains on FB during hot deformation, however, showed no anisotropic growth, instead perhaps showing mainly grain rotation by grain boundary sliding. It shows that grains in the specimens

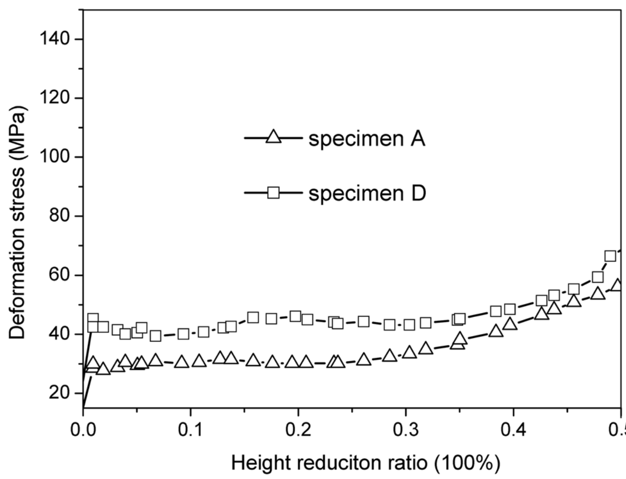

Fig. 5. Height reduction rate as a function of deformation stress for specimens A and D.

with different sizes show various deformation mechanisms. For the grains with small sizes, the deformation is controlled by the anisotropy of grain growth, driven by the chemical potentials of the atomic species in the liquid phase or at the surface of the crystallites [9]. For the grains with large sizes, the deformation is dominated by grain boundary sliding.

4. Conclusions

The hot-compaction temperature was found to impact the magnetic properties of die-upset magnets. The remanence of the isotropic precursor increases slightly with the improved hot-compaction temperature, and the grains start to grow on FB at the higher hot-compaction temperature. After hot deformation, the magnetic properties of the anisotropic magnets are closely connected with the hot-compaction temperature. For the magnets hot pressed at a higher temperature large equiaxial grains can be detected, which accounts for the low alignment and high deformation stress during the hot deformation, and thus the low magnetic properties.

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