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Magnetic microstructure and magnetic properties of spark plasma sintered NdFeB magnets

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ABSTRACT

Nanocrystalline NdFeB magnets were prepared by spark plasma sintering (SPS) technique using melt-spun ribbons as starting materials. A distinct two-zone structure with coarse grain zone and fine grain zone was formed in the SPSed magnets. Multi-domain particle in coarse grain zone and exchange interaction domain for fine grain zone were observed. Intergranular non-magnetic phase was favorable to improve the coercivity due to the enhancement of domain wall pinning effects and increased exchange-decouple. The remanent polarization of 0.83 T, coercivity of 1516 kA/m, and maximum energy product of 118 kJ/m^3 are obtained for an isotropic magnet.

1. Introduction

NdFeB magnets are critical components for numerous devices ranging from electric motors to disk drives to traction motors to wind generators [1–4]. Nanocrystalline NdFeB magnets have attracted much attention not only because of their good magnetic properties but also their exceptional fracture toughness and thermal stability comparing to the conventional microcrystalline sintered magnets. As a rapid sintering technique, the advantages of the spark plasma sintering (SPS) technique make it suitable for the preparation of nanocrystalline NdFeB magnets [5]. For nanocrystalline magnets, previous studies have focused on the microstructure, magnetic properties, and corrosion resistance [6–11]. The studies on the magnetic microstructure and their relation to the magnetic properties are still lacking. The knowledge of magnetic structure is not only of fundamental interest, but also of technological significance. The understanding the magnetic microstructure is an indispensable step toward the realization of high performance magnets. In this work, a detailed understanding in the magnetic microstructure and their relation to the magnetic properties is investigated in details.

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2. Experimental

Commercial melt spun ribbons with nominal compositions of $Nd_{13.5}Co_{6.7}Ga_{0.5}Fe_{73.5}B_{5.6}$ were put into the cylindrical graphite die for SPS in vacuum using the facility of SPS-825 (Sojitz Machinery Co.). The unscreened initial ribbons (< 400 μm) and < 45 μm size powder were used, respectively. The SPS sintering temperature T_{sps}, Pressure P_{sps} and holding time t_{sps} are in the ranges of 600–800 °C, 30–50 MPa and 0–5 min, respectively. Microstructure for the magnets was characterized by scanning electronic microscope (FEI Quanta FEG 250). TEM specimens were prepared by mechanical polishing of thin sections of the material followed by ion milling. Magnetic domain ns microstructure was investigated by TEM (JEOL2100F). Magnetic properties were tested by physical properties measurement system (PPMS-9, Quantum Design, USA) equipped with a 9 T vibrating sample magnetometer (VSM). Magnetic domains were imaged by means of atomic force microscopy with a magnetic force microscopy (MFM) (Cypher, Asylum Research).

3. Results and discussion

Fig. 1 shows the microstructure of SPSed magnets prepared using unscreened powders (< 400 μm). Two distinguished zones with different grain sizes which form layers perpendicular to the

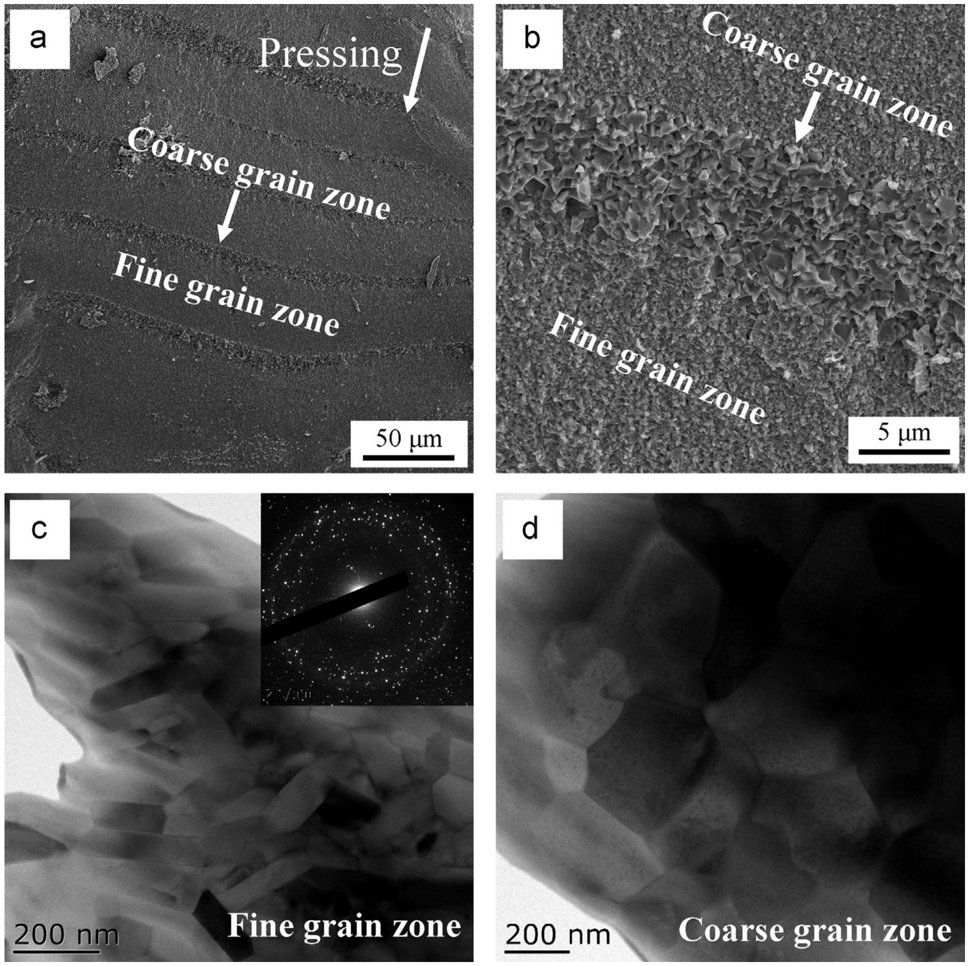

Fig. 1. Microstructure of SPSed NdFeB magnets, SEM images (a) and (b), TEM images (c) and (d), Inset shows the SAED pattern.

pressing direction are noticed. These two zones are referred as coarse grain zones and fine grain zones. The formation of coarse grain zone and fine grain zone are attributed to the sintering mechanism of SPS. Local high temperature zone was generated by the pulsed energy existed between the particle contacting surfaces. The temperature at the center of the particle was dramatically lower than that in the particle boundaries. Song et al. reported that the temperature in the particle contacting surface is nearly 3000 K higher than that at the particle center during SPS [12]. The huge difference in the temperature between the particle contacting surface and the center is the main reason of two distinguished zones formation.

From the TEM images of coarse grain zone and fine grain zone, demonstrated in the Fig. 1(c) and (d), respectively, it is found that the coarse grain zones consist of equiaxed grains, while the fine grain zones are mainly composed of elongated grains with various aspect ratios. This suggests their anisotropic growth due to the non-equilibrium process of SPS. Unfortunately, due to the small size, their crystallographic orientation was varied at this stage. As a whole, the spark plasma sintered magnet is isotropic. A selected area diffraction pattern can also evidence the isotropic structure of SPSed magnets, shown in the Fig. 1(c) inset. The optimum magnetic properties with J_r=0.83 T, $_jH_c$=1516 kA/m and $(BH)_{max}$ =118 kJ/m^3 are obtained for a nanocrystalline SPSed magnet at T_{sps}= 700 °C, P_{sps}=50 MPa and t_{SPS}=5 min, demonstrated in the Fig. 2(a).

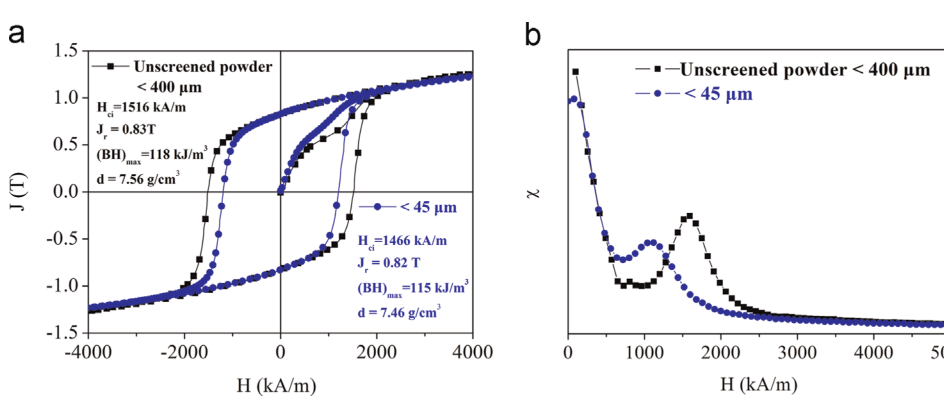

Fig. 2. Magnetic hysteresis curves (a) and differential susceptibility curves (b) for SPSed NdFeB magnets prepared using various particle size powders.