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Magnetization reversal in textured NdFeB–Fe composites observed by domain imaging

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ABSTRACT

Textured composite samples consisting of $Nd_{13.6}Fe_{73.6}Ga_{0.6}Co_{6.6}B_{5.6}$ (MQU-F^{TM}) and micron-sized Fe particles with weight ratios from 100:0 to 70:30 have been prepared by hot deformation. Microstructure studies revealed a layered structure of both phases with the layer normal parallel to the pressing direction. Magnetic measurements showed single-phase hysteresis curves for all samples when measured along the pressing direction, which is also the easy axis of magnetization. Coercivity decreased drastically from 1.32 T for pure NdFeB samples to 0.154 T for a sample with 30 wt% Fe. Magneto-optical Kerr microscopy with a digitally enhanced imaging technique has been used to examine the evolution of magnetic domains in the hard and soft phase during demagnetizing a sample consisting of 70 wt% NdFeB and 30 wt% Fe. It is shown that demagnetization takes place via domain rearrangements within the soft phase, which lead to and support the nucleation of reversed interaction domains at phase boundaries. Also nucleation of interaction domains within the hard magnetic phase could be revealed.

1. Introduction

Anisotropic NdFeB–Fe composite magnets attract a lot of interest due to their theoretical potential of exhibiting higher energy products than those of currently existing magnets [1,2] and with this possibly presenting the next generation of permanent magnets. The hard magnetic phase provides high uniaxial magnetocrystalline anisotropy and coercivity whereas the magnetically soft component contributes to high saturation magnetization.

It was commonly believed that only when both components are coupled through exchange interactions the magnet as a whole system behaves in a unitary manner. Gabay et al. [3] showed in 2006 that magnetic exchange interaction can lead to magnetic single-phase behavior in composite magnets consisting of soft and hard magnetic phases. By mixing micron-sized Fe powder with rare-earth rich NdFeB melt spun ribbons and taking the route of hot plastic deformation (hot pressing+die upsetting) they produced anisotropic composite samples that behave in a magnetically uniform manner along the easy axis of magnetization though the dimension of the soft phase is much greater than the effective exchange length of NdFeB (which is in the range of 8 nm). Hot plastic deformation of consolidated melt-spun R–Fe–B alloys is one of the common techniques used to fabricate anisotropic magnets [4]. The easy axis of magnetization, which is in the case of NdFeB consistent with the *c*-axis, is parallel to the pressing direction. Gabay et al. [5] stated that the coupling effect between the two magnetic phases is due to magnetostatic interactions, which could also be supported by simple numerical simulations [5]. The single-phase behavior of these composites during demagnetization makes them interesting for detailed studies of the magnetic microstructure and its behavior in a magnetic field.

For hot-deformed samples made of mere melt-spun NdFeB ribbons the existence of interaction domains [6] has been observed many times before [7–15]. Domain structures of this kind occur due to the magnetic coupling between grains whose sizes are about equal or smaller than the expected single-domain grain size [6]. For NdFeB this value is in the range of about 300 nm [16]. Though the phenomenon of interaction domains was recognized 50 years ago, fundamental questions are still unanswered such as e.g. the behavior of interaction domains in magnetic fields. Volkov and Zhu [11] have studied the local domain structure evolution in a die-upset magnet at different points of the magnetization loop by Lorentz microscopy. However the limited sample thickness of less than 100 nm for observations by TEM and the known sensitivity of domain structures to sample thickness [17] question the applicability of these observations to bulk magnets.

The current paper presents microstructure, magnetic property and magnetic domain investigations of hot-deformed composite samples consisting of NdFeB and Fe. For studies of magnetic

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domains during magnetization reversal we performed in-situ Kerr microscopy experiments on a sample with 70 wt% NdFeB and 30 wt% Fe. Images of the domain configuration obtained at different points of the demagnetization curve provide information about the reversal processes and give insight on the interaction between the two magnetic phases in those samples.

2. Experimental

For the preparation of composite magnets we used NdFeB melt spun ribbons of Magnequench International Inc. with a nominal composition of $Nd_{13.6}Fe_{73.6}Ga_{0.6}Co_{6.6}B_{5.6}$ (MQU-F^{TM}) and Fe powder of particle size <105 μm (-140+325 mesh). Both components were thoroughly mixed in varying compositions from a NdFeB to Fe weight ratio of 100:0 to 70:30. The powder blends were then hot pressed in vacuum at 725 °C under a pressure of 150 MPa followed by hot plastic deformation in Ar atmosphere at 750 °C with a strain rate of $2\times10^{-3}\,s^{-1}$. The deformation degree φ was 1.3 ($\varphi=\ln(h_0/h_{end})$, with h_0 as the starting height of the sample and h_{end} the height after deformation), which results in a height reduction of about 70%. For magnetic measurements small cuboids were cut from the middle of the samples and magnetized with a 5 T field pulse along the measuring direction. Demagnetization curves parallel and perpendicular to the pressing direction were then obtained with a Permeagraph® C300 (Magnet-Physik). Magnetic properties have also been characterized using a vibrating sample magnetometer (cryogenic). For microstructure and magnetic domain investigations the sample surface was polished with the observation plane parallel to the pressing direction. The microstructure and morphology were studied with scanning electron microscopy (SEM, LEO 1530 Gemini). For phase distribution information backscattered electron contrast (BSE) was used, whereas for all other micrographs

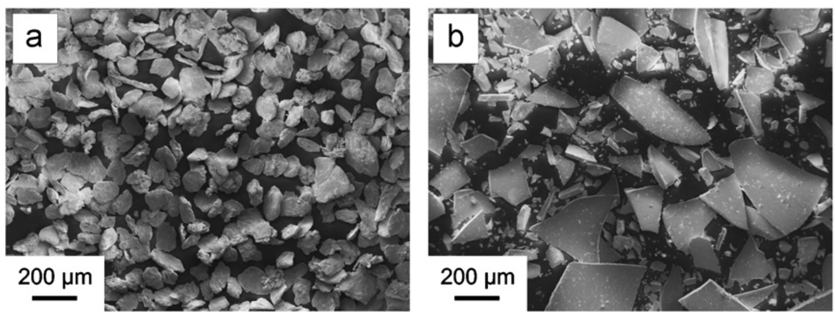

Fig. 1. SE images of starting materials: (a) Fe and (b) NdFeB.

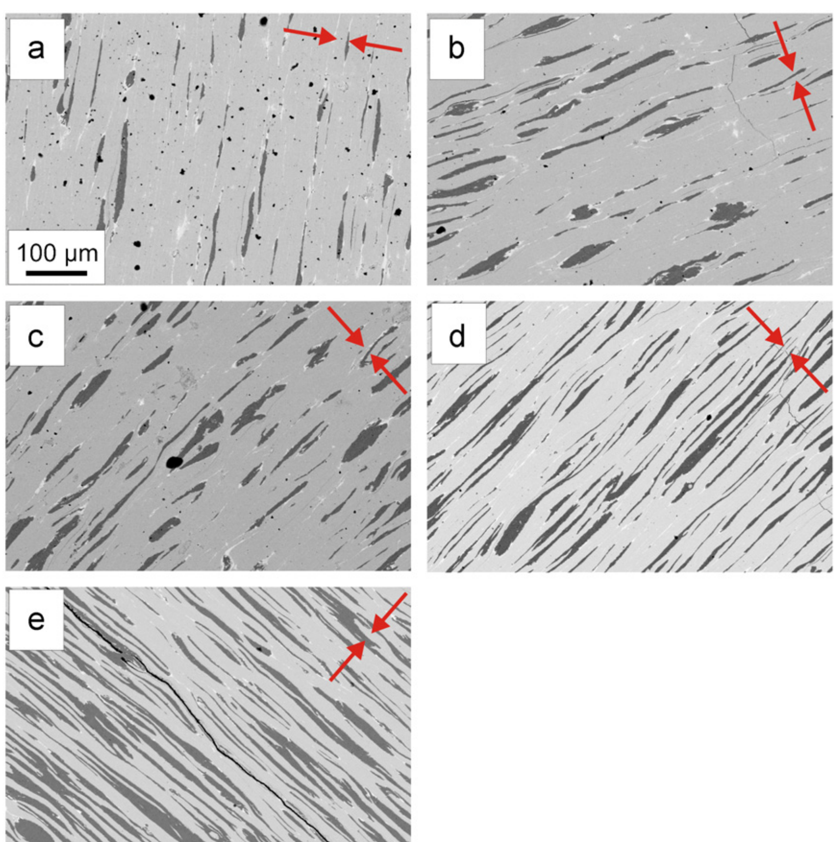

Fig. 2. BSE images of polished composite samples with Fe content of (a) 5 wt%, (b) 10 wt%, (c) 15 wt%, (d) 20 wt% and (e) 30 wt%.