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Tutorial

Grain size quantification by optical microscopy, electron backscatter diffraction, and magnetic force microscopy

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ABSTRACT

Quantification of microstructure, especially grain size, in polycrystalline materials is a vital aspect to understand the structure-property relationships in these materials. In this paper, representative characterization techniques for determining the grain size, including optical microscopy (OM), electron backscatter diffraction (EBSD) in the scanning electron microscopy (SEM), and atomic force microscopy/magnetic force microscopy (AFM/MFM), are thoroughly evaluated in comparison, illustrated by rare-earth sintered Nd-Fe-B permanent magnets. Potential applications and additional information achieved by using aforementioned characterization techniques have been discussed and summarized.

1. Introduction

The microstructure of materials is central to understand the relation between microstructural quantities and material properties. In polycrystalline materials, grain size, as one of the most paramount microstructural features, plays a vital role in governing mechanical, optical, electrical, and magnetic properties of materials. Proposed by Hall (Hall, 1951) and Petch (Petch, 1953), the relation between yield strength and average grain size of materials is formulated as $\sigma = \sigma_0 + kd^{-1/2}$, where σ_0 represents the lattice friction stress required to move individual dislocations, k is the Hall-Petch slope which is material-dependent, and d is the average grain size of materials (Lehto et al., 2014). In addition, reduced strength with broadening the grain size distribution for a fixed averaged grain size was reported by Berbenni by computer simulation (Berbenni et al., 2007). In optical materials, it has been found that the emission decay times decrease significantly as the grain size of Sr_2CeO_4 nanocrystalline materials increases (Stefanski et al., 2015). In (Ba,Ca)(Zr,Ti)O_3 ferroelectric ceramics, the transition temperature increases and maximum dielectric constant decreases with the decreased grain size (Mudinepalli et al., 2014). Another example is Nd-Fe-B permanent magnets, which have been extensively studied owing to their superiority of the magnetic properties, including intrinsic coercivity, remanence, $(BH)_{max}$ (Croat et al., 1984; Sagawa et al., 1984a,1984b; Cahn, 1985). Low intrinsic coercivity value (H_{ci}) which is only ~20-30% of the theoretical value, severely restricts the applications of Nd-Fe-B magnets in high temperatures of ~150 °C due to the massive thermal demagnetization (Sagawa et al., 1984a, 1984b; Sinnema et al., 1984). It has been argued that the coercivity and temperature coefficients of Nd-Fe-B magnets can be improved via tuning the microstructure/microchemistry of Nd-Fe-B magnets, such as refining grain size (Li et al., 2009; Ramesh et al., 1988). In detail, the relations between the intrinsic coercivity and average grain size are as follows: $H_{ci} = a\text{-}blnD$ (Ramesh et al., 1988), or $H_{ci} = a\text{-}blnD^2$ (Li et al., 2009), where D is the average grain size of the matrix grains. Therefore, accurate measurements of grain size are indispensable to underpin the development of models to predict the mechanical, optical, electrical, and magnetic properties of materials.

Multiple microscopy techniques have been applied in particle/grain size analysis (Allen, 2013; Chen et al., 2017, 2016; Fairbrother et al., 2014; Gutierrez-Urrutia et al., 2010; Hoo et al., 2008; Humphreys, 2004; Kabla et al., 2014; Lim et al., 2014; M., 1954; Mingard et al., 2009; Trimby et al., 2014; Wong et al., 2015). Ellison reported the errors in the particle-size distribution of silica dust suspended in liquid by using optical microscopy (Allen, 2013; Ellison, 1954). In recent years, scanning electron microscopy-electron backscatter diffraction (SEM-EBSD) has been integrated for grain size measurement in Fe-Mn-C

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Table 1
The nominal composition of the sintered Nd-Fe-B magnet.

	PrNd	Fe	B	Co	Cu	Al	Ga	Nb	Zr
wt.%	34.5	62.46	0.98	0.6	0.18	0.6	0.08	0.5	0.1

TWIP steel (Gutierrez-Urrutia et al., 2010), aluminum (Humphreys, 2004), WC/Co cemented carbide hard metals (Mingard et al., 2009), and etc. Furthermore, SEM-transmission Kikuchi diffraction (TKD) with better spatial resolution than EBSD has been successfully applied in the grain size quantification and microstructural observation in MgB_2 superconductors (Wong et al., 2015), and HPT-deformed duplex stainless steel (Trimby et al., 2014). In addition, A. Fairbrother et al. (Fairbrother et al., 2014) reported the grain size distribution of ZnS thin films by integrating high resolution transmission electron microscopy (TEM). Except for the optical and electron microscopy, it should be noted that X-ray diffraction (XRD) and micro/nano-computerized tomography (CT) have also been integrated into grain size quantification in various materials, such as NiTi thin films (Kabla et al., 2014) and the anode electrode of lithium-ion battery (Lim et al., 2014). Furthermore, Hoo et al. (Hoo et al., 2008) characterized particle size distributions of polystyrene nanoparticles by using scanning probe microscopy (SPM). As one of the branches of SPM, magnetic force microscopy (MFM) is a powerful tool to characterize the magnetic domain structure and grain size of magnetic materials simultaneously, which provides access to investigating the relation between the grain size and magnetic domain structure of magnetic materials.

However, selecting the most appropriate characterization technique is still an obstacle for accurate particle/grain size analysis due to the lack of evaluation and comparison of these techniques. The aim of this work is to compare and evaluate various characterization techniques for grain size quantification, illustrated by rare-earth sintered Nd-Fe-B permanent magnets. The merit of choosing sintered Nd-Fe-B magnet is that magnetic domain structure can also be revealed with grain size quantification by AFM/MFM simultaneously.

2. Experimental procedure

The nominal composition of the Nd-Fe-B magnet is listed in Table 1. The as-pressed Nd-Fe-B magnets were obtained after induction melting, strip casting, hydrogen decrepitation, jet milling and isostatic pressing. The as-pressed Nd-Fe-B magnets were sintered at 1085 °C for ~4 h, followed by first/second step post-sinter annealing at 900 °C and 620 °C for ~2 h and ~4 h, respectively. Sintered Nd-Fe-B permanent magnets were cut into small disks with a height of ~1.6 mm and a diameter of ~10 mm by wire-electrode cutting.

All the samples for OM, SEM-EBSD, and AFM/MFM were mounted in epoxy and then mechanically polished using 220-grit, 500-grit, 1200-grit SiC papers, and standard colloidal silica suspension successively. For OM, well-polished samples were etched using a solution of 2% nital for 0.5–1 s to reveal the grain boundaries (GBs) and then optical images of 10 times magnification and 20 times magnification were captured with an Olympus BX61 microscope (SIS). Finally, the images obtained were further processed in Adobe Photoshop and MagniSci for determining the average grain size of sintered Nd-Fe-B magnets.

SEM-EBSD mapping was investigated in a Zeiss Ultra Plus field-emission scanning electron microscope equipped with an Oxford Instruments Aztec and Nordlys-nano EBSD detector. The Kikuchi patterns were recorded with an acceleration voltage of 25 kV, an aperture size of 120 μm, and a 'high current' mode. The time for mapping the selected area with a step size of 0.5 μm and a grid size of 489 × 388 pixels was ~4 h and results were processed by the standard EBSD processing software. In addition, energy dispersive X-ray spectroscopy (EDS) mapping was also obtained for revealing the elemental distribution simultaneously.

Atomic Force Microscope (AFM) measurements were conducted with a Bruker Dimension Icon (Santa Barbara, CA, USA), utilizing Magnetic Force Microscopy (MFM). Before performing MFM experiments, normal AFM experiments in tapping mode were carried out on the mechanically polished sample, to reveal the actual surface topography. The tip-sample interaction was measured as the variations of the oscillation frequency of the probe. The probes employed for imaging surface topography were non-magnetic high lateral resolution Otespa probes (Bruker) with normal spring constants of ~42 N/m, the tip radius of ~7 nm and the resonance frequency of 300 kHz.

MFM is a secondary AFM imaging mode that uses a two-pass technique. The initial pass measures the surface topography using tapping

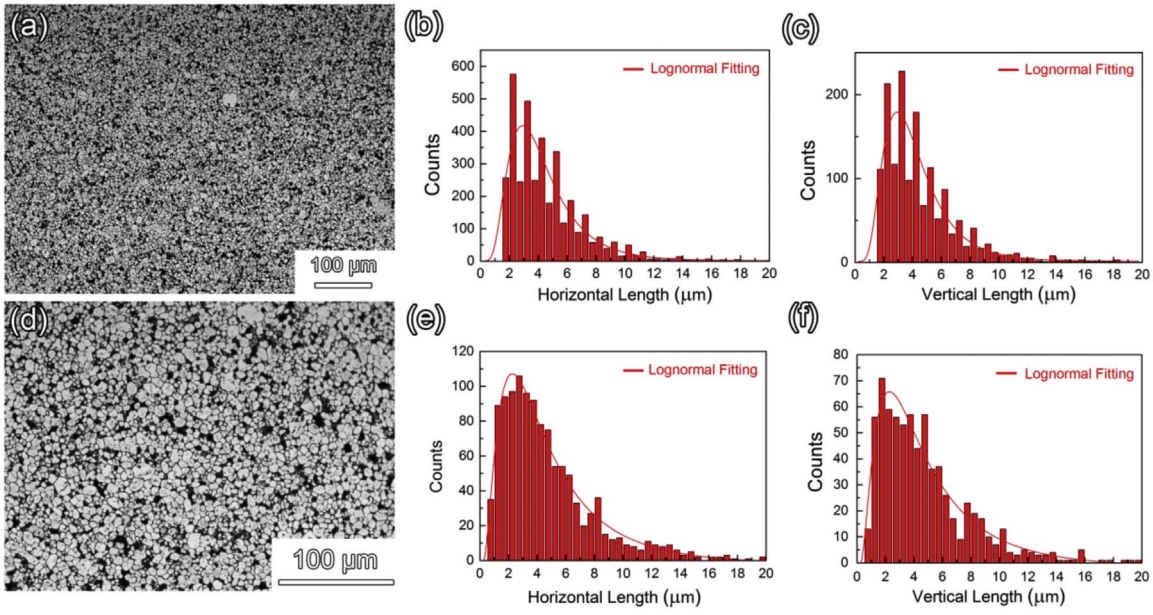

Fig. 1. (a) Optical image (10 times magnification) (b) The counts as a function of the grain size (horizontal length) in (a). (c) The counts as a function of the grain size (vertical length) in (a). (d) Optical image (20 times magnification) (e) The counts as a function of the grain size (horizontal length) in (d). (f) The counts as a function of the grain size (vertical length) in (d).