High performance nanostructured Nd–Fe–B fine powder prepared by melt spinning and jet milling

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Isotropic Nd–Fe–B nanocrystalline fine powders with particle size in the range of 1–10 μm have been developed using melt spinning and jet milling. The processing steps primarily consist of melt spinning Nd–Fe–B alloy to obtain ribbons with 25–50 μm thickness, crushing the ribbon to obtain coarse powder with average particle size of about 200 μm, and jet milling the coarse powder to obtain fine powder with average particle size of about 5–6 μm. The effects of jet milling conditions on powder particle size, microstructure, and magnetic properties were systematically studied. For a magnet alloy nominally composed of $Nd_{11.9}(Fe_{0.93}Co_{0.07})_{82.6}B_{5.5}$, a particulate yield of $D_{10}=2$ μm, $D_{50}=6$ μm, and $D_{90}=11$ μm and magnetic properties of $B_r=8.82$ kG, $H_{ci}=9.5$ kOe, and $(BH)_{max}=15.3$ MGOe have been achieved in melt-spun and jet milled fine powders. The combined advantage of small particle size and high magnetic performance will make the Nd–Fe–B fine powder an attractive candidate for applications such as magnetic fluids, inks, micromachines, and flexible sheets. © *2010 American Institute of Physics*. [doi:10.1063/1.3348544]

I. INTRODUCTION

Magnetic fine powders with particle size in the range of 1–10 μm have found many applications in sensors, transducers, magnetic fluids, inks, miniature electromechanical systems (MEMS), flexible sheets, etc.[1] For decades, ferrites have been the primary commercially available magnetic fine powder products. Although ferrite has a lower cost compared to rare earth magnetic materials, its magnetic performance is very low, only at about 1/5–1/3 of rare earth magnets.[2] As such, the ferrite powders are not desirable for applications, which require high magnetic performance. Also, there is an increasing demand for high performance magnetic fine powders for applications in magnetic fluids, inks, MEMS, and flexible sheets.[3] Thus, the purpose of the present work is to develop high performance Nd–Fe–B magnetic fine powders to better serve the industry needs.

In this work, high performance Nd–Fe–B nanocrystalline fine powders with particle size in the range of 1–10 μm have been developed using melt spinning and jet milling. The processing steps primarily consist of melt spinning Nd–Fe–B alloy to obtain ribbons with 25–50 μm thickness, crushing the ribbon to obtain coarse powder with average particle size of about 200 μm, and jet milling the coarse powder to obtain fine powder with average particle size of about 5–6 μm. The underlying technical principles are (1) the use of melt spinning to develop nanoscaled microstructure and therefore providing magnetic hardening in the ribbon and (2) the use of jet milling to obtain micron-sized fine powders with narrow PSD while retaining the nanoscaled microstructure within each individual particle. In this paper, we report our studies on the effects of jet milling conditions on fine powder particle size, microstructure, and magnetic properties in a single-phase type Nd–Fe–B melt-spun alloy.

II. EXPERIMENTAL

A commercially available MQP-B+ (registered trademark of Magnequench International) melt-spun powder product was used as precursor for a jet milling process to produce fine powders. The starting magnet powder has a nominal composition of $Nd_{11.9}(Fe_{0.93}Co_{0.07})_{82.6}B_{5.5}$ and is produced by melt spinning the Nd–Fe–Co–B alloy to obtain ribbons with 25–50 μm thickness, crushing the as-quenched ribbons with a twin-roller, sieving with a 40 mesh sieve to obtain coarse powder with average particle size of about 200 μm, and annealing the crushed powders at 600–900 °C for 1–30 min to optimize powder microstructure and magnetic performance. The starting magnet powder has grain size of about 20–30 nm and hard magnetic properties of $B_r=8.95–9.15$ kG, $H_{ci}=9.0–10.5$ kOe, and $(BH)_{max}=15.8–16.8$ MGOe.[4]

The jet milling was carried out with a QLM-100T jet mill manufactured by JiLin Care Mechanical and Electrical Equipment Factory Ltd. Corp. and was operated under nitrogen atmosphere with a gas pressure of 0.2–0.4 MPa. The rotating speed of the classifier was set at a wide range between 1020 and 4500 rpm to study its effect on particle size of milled powders. (Thereafter, the classifier speed is referred as milling speed in this paper.) A small amount of oxygen (less than 1%) was introduced into the milling chamber to passivate the as-milled powder surface for the purpose of safe and easy handling.

The particle size distribution (PSD) of the powders was measured with a Microtrac S3500 Laser Particle Size Analyzer. A scanning electron microscope (SEM) was used to examine powder size and surface morphology. A Panalytical x-ray diffractometer (XRD) was used to detect the phases

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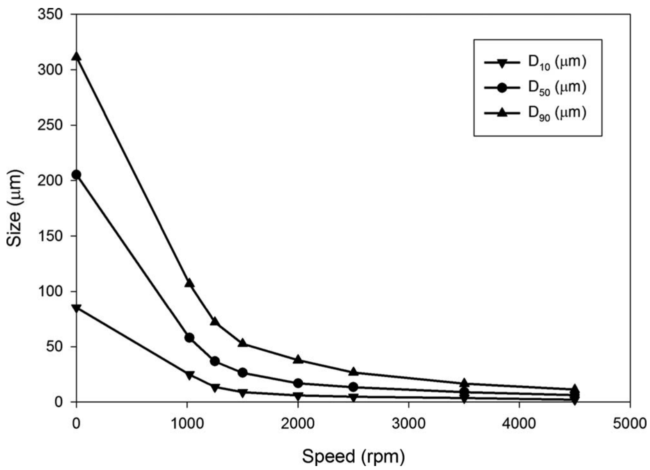

FIG. 1. Powder particle size of jet milled magnet powder as a function of milling speed.

present and the grain size. The grain size was calculated from the XRD patterns using Scherrer's formula. Magnetic properties were measured with a Lakeshore vibrating sample magnetometer (VSM).

III. RESULTS AND DISCUSSION

The jet milling process was systematically studied in this work. It was found that the classifier rotating speed (milling speed) is the most critical parameter in controlling the PSD of jet milled powder. Figure 1 summarizes the effect of milling speed on PSD of jet milled magnet powder. As shown, powder particle size decreases rapidly with increased the milling speed from 0 up to 1500 rpm, then decreases slowly as the speed increases from 2000 to 3500 rpm, and gradually approached stable when milling speed reaches 4500 rpm (the maximum speed for the jet mill). Further, not only the powder average particle sizes (defined as D_{50}) were decreased upon jet milling, the powder PSD (defined as the difference between D_{10} and D_{90}) also became much sharper, indicating that the coarse powder particles become more uniform when being milled into fine powders. To further illustrate this, Figs. 2 and 3 compare SEM images and PSD data of magnet powder before and after jet milling, respectively. As can be seen, the prior-to-jet-milling magnet powders have flakelike morphology and a broad range of particle sizes with $D_{10} = 85\ \mu m$, $D_{50} = 205\ \mu m$, and $D_{90} = 311\ \mu m$. After jet milling at 4500 rpm, the flakelike powders become very fine powders with $D_{10} = 2\ \mu m$, $D_{50} = 6\ \mu m$, and $D_{90} = 11\ \mu m$.

Figure 4 shows the demagnetization curves of magnet powder before and after jet milling. As shown, although the unmilled starting MQP-B+ coarse particle sample has the highest magnetic properties of $B_r = 9.02$ kG, $H_{ci} = 9.6$ kOe, and $(BH)_{max} = 16.0$ MGOe, the milled particles retained a very high percentage of their magnetic performance after the jet milling step, with only a slight decrease in the magnetic performance as the speed increases. For example, for the sample milled at 4500 rpm, even though the particle size of the milled particles was very small, the milled particles still

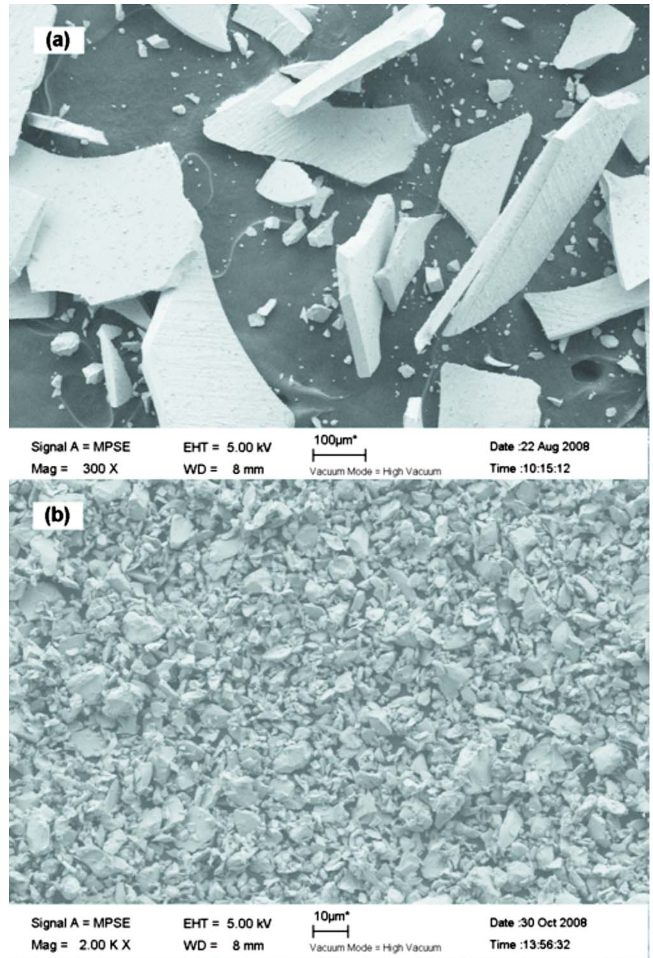

FIG. 2. (Color online) SEM of magnet powder (a) before jet milling and (b) after jet milled at 4500 rpm.

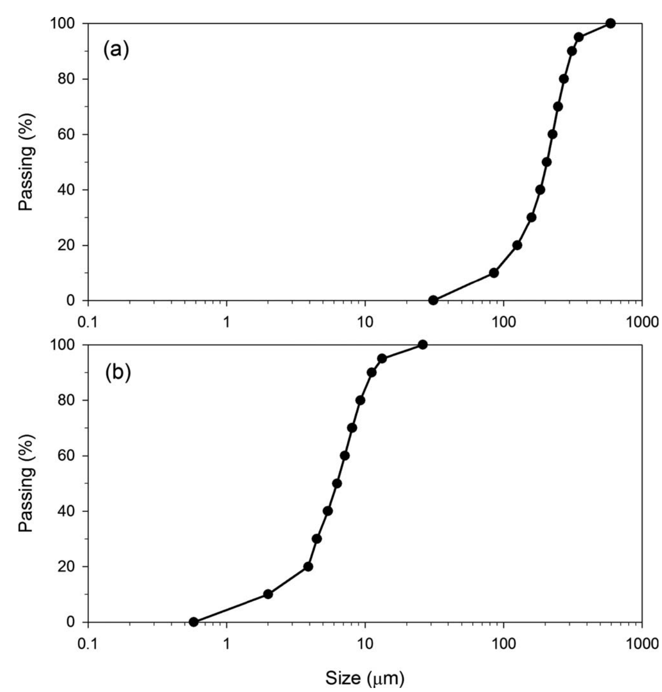

FIG. 3. PSD data of magnet powder (a) before jet milling and (b) after jet milled at 4500 rpm.

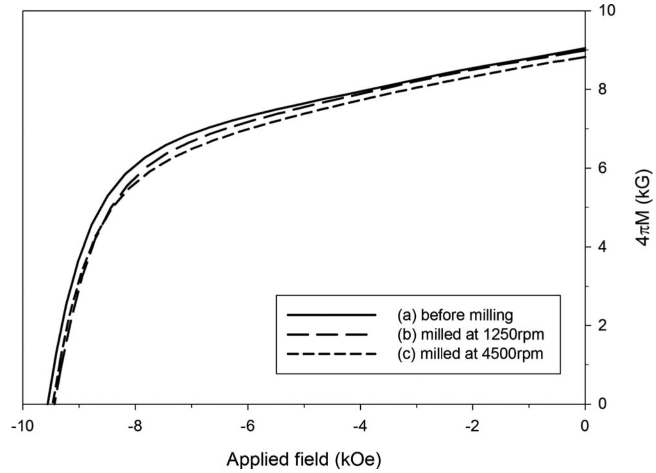

FIG. 4. VSM demagnetization curves of magnet powder (a) before jet milling, (b) jet milled at 1250 rpm, and (c) jet milled at 4500 rpm.

had magnetic properties of B_r=8.82 kG, H_{ci}=9.5 kOe, and $(BH)_{max}$=15.3 MGOe. This corresponds to a minimal loss of 2% in B_r, 1% in H_{ci}, and 4% in $(BH)_{max}$.

Figure 5 shows the XRD data of magnet powder before and after jet milling. It was found that all the diffraction peaks in each sample could be indexed with the $Nd_2Fe_{14}B$ structure and that there was no extra peak in the XRD data upon jet milling. This indicates that jet milling process did not cause any extra phase occurrence or any phase decomposition of the starting magnet material. It was further found by using the Scherrer's formula that the grain size of the $Nd_2Fe_{14}B$ matrix is about 20 nm in all samples. This confirmed that the nanoscale grain size developed in melt spinning was well retained upon jet milling, which is the main reason for the good magnetic properties in jet milled powders.

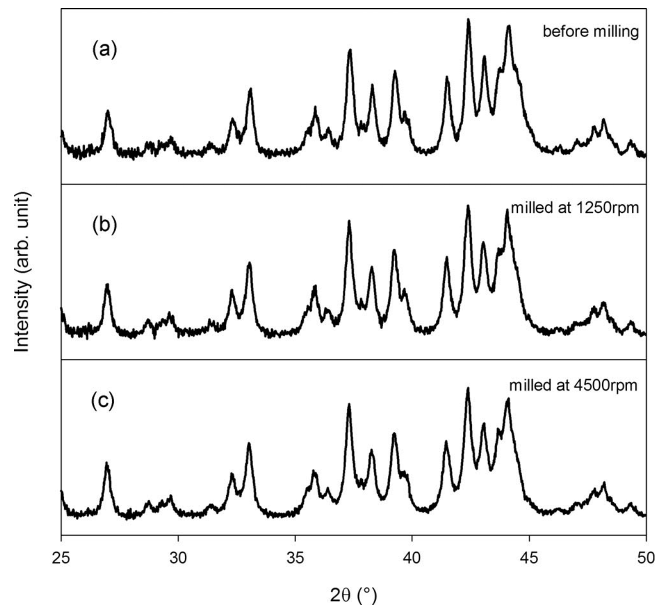

FIG. 5. XRD pattern of magnet powder (a) before jet milling, (b) jet milled at 1250 rpm, and (c) jet milled at 4500 rpm.

IV. CONCLUSIONS

In summary, our present work has demonstrated that melt spinning and jet milling are an appropriate technique route to produce high performance Nd–Fe–B fine powders. For a magnet alloy flake particulate with a nominal composition of $Nd_{11.9}(Fe_{0.93}Co_{0.07})_{82.6}B_{5.5}$, a postmilling particulate yield of D_{10}=2 μm, D_{50}=6 μm, and D_{90}=11 μm and magnetic properties of B_r=8.82 kG, H_{ci}=9.5 kOe, and $(BH)_{max}$=15.3 MGOe were achieved in the resulting fine powders. The combined advantage of small particle size and high magnetic performance can make the Nd–Fe–B fine powder an attractive candidate for applications such as magnetic fluids, inks, MEMS, and flexible sheets.

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[2]See: http://www.tdk.co.jp/tefe02/ferrite.htm.
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[4]See: http://www.magnequench.com.