Characterization of Melt-Spun NdFeB Magnets Prepared by Explosive Compaction

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Explosive compaction processing is applied to prepare NdFeB magnets with melt-spun powders. It is found that shock consolidation processing could result in the magnetic properties, compressed strength and density of NdFeB bulk better than that of the conventional resin-bonded magnet. Scanning electron microscope (SEM) observation shows the melted areas and a mass of the micro-cracks in the close-packed particles resulted from the shock wave. The small Nd2Fe14B grains less than 100 nm, existing inside the powder particle without appreciable change of the original grain size, are revealed using the field emission scanning electron microscope (FE-SEM). The corresponding magnetic structure consisting of the very fine magnetic domains is also revealed with magnetic force microscopy (MFM). The results indicate that not only the microstructure but also the magnetic domain structure of the original powder are kept after explosive compaction. Although there are many micro-cracks in particles, the excellent magnetic properties are still obtained because the magnetic domain structure is so fine that seems not to be affected by the micro-cracks. The compression strength is also 40% higher than that of the polymer-bonded NdFeB magnet for its increased density.

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1. Introduction

The high magnetic properties make NdFeB permanent magnets of significant and growing commercial importance for many applications in recent two decades. Polymer bonding processing by blending the silane-treated NdFeB powders with the polymeric binder is the main fabrication method for the melt-spun powders in this field. Inevitably, the non-ferromagnetic binder, typically ~20 vol%, would make the magnetic properties reduced. As an alternative consolidation technique to produce the bulk materials from the powders, explosive compaction involves a variety of metal powders processes.1) Some studies referring to this field were reported such as the explosive compaction processing of Fe-Al, Ti-Ni, Sm-Fe-N, Nd-Fe-B solids.2–6) The most promising results were achieved by S. Ando et al.7) They produced sample 10 mm in diameter and 8 mm thick using amorphous NdFeB powders, with density more than 95%, the remanence of 8.5 kGs, the intrinsic coercivity of 772 kA/m, and the energy product of 105.1 kJ/m3. The present paper studies the explosive compacted NdFeB magnet made from melt-spun powders. The magnetic properties and compress strength as well as density are measured. SEM, FE-SEM and MFM are employed to illustrate their microstructure and the magnetic-domain structure. By the comparison to that of the conventional polymer-bonded NdFeB, the effects of explosive compaction processing on the magnetic properties and compress strength of this NdFeB compact are discussed.

2. Experimental Procedure

The explosive compaction configuration is shown in Fig. 1. The explosive used is the mixture of TNT (40 mass%) and hexogen (also referred to as RDX, 60 mass%), with a denotation velocity of 6.5 km/s and a maximum pressure of 13 Gpa on the steel tube.

Fig. 1 Experimental set-up of the explosive compaction.

The melt-spun NdFeB powders (MQLP-B, average particle size: 75 μm), characterized by the remanence of 8.12 kGs, the intrinsic coercivity of 780 kA/m, and the energy product of 104.3 kJ/m3, are firstly packed to a bulk densities about 67% of the corresponding theoretical density (ρth = 7550 kg/m3) and then explosively consolidated in the seamless steel tubes, labeled as sample A. The powders are also processed for the polymer-bonded magnet B by the conventional methods as reference sample. Magnetic properties of the magnets are measured using NIM2000HF hysteresigraph test machine. Compressed strength is measured on a universal mechanical test machine (WE-300A) with GB7314-87 procedure. The observation of microstruc-
ture is performed on a PHILIPS XL-30 scanning electron microscope (SEM) and a LEO 1550VP field emission scanning electron microscope (FE-SEM). The magnetic force images of the samples along the direction of the compaction are obtained using a Nanoscope IIIa-D3000 MFM microscope with the tip of magnetic force etched silicon probe (MESP) magnetized upward. Density is measured by drainage method.

3. Results and Discussion

For the primary study of the characteristics of compact magnet, the researches about the compaction process parameters such as the shock wave speed and shock pressure are not enough in our present work. The density of the as-compacted sample A is about 88% of the theoretical NdFeB, somewhat lower than the result in Ref. 7).

Table 1 lists the magnetic properties and compressed strength as well as density of the samples. The properties of original powders and the results in Ref. 7) are also included as reference. It is shown that the magnetic properties of the original powders are maintained after explosive compaction, but the remanence $B_r$ improvement of the original powders presented in Ref. 7) has not been observed in our results, which should be owing to the relatively lower density and could be ameliorated by optimizing the compression parameters. It is worthy of notice that the properties of as-compacted sample A are quite superior, about 30% improvement of $(BH)_{max}$ and 40% increase of the compression strength, to that of the polymer-bonded magnet B.

Figure 2 shows the SEM micrographs of the large flaky melt-spun NdFeB particles of sample A and B. Fig. 2(a) reveals a Mach stem hole in the central part of sample A, about 40 µm in diameter, but no serious defects formed by the tensile stresses during compaction are found. By the comparison of Fig. 2(b) and Fig. 2(c), it is obvious that the more compact structure of sample A than that of sample B is obtained by the shock wave of the high speed during explosive compaction.

Figure 3 shows two features in sample A at higher magnification. In Fig. 3(a), several flaky particles are bonded as a whole through the melted zone at the particle boundaries, and this kind of melted zone is observed in many other areas of the compact. Figure 3(b) presents the alveolate microstructure and some micro-cracks resulted from the severe shock compaction at the edge of the melted area. Since the drastic temperature increment and the very high pressure in explosive compaction process, the friction welding has taken place on the boundaries of the powders, leading to the melted areas. Considering the inter-particle colliding under the co-action of the multiple convergent waves and the reflection waves, the fragmentation of the grains occurs rather than the compressive deformation of powders in the fragile NdFeB material. Therefore, the alveolate microstructure and micro-cracks are produced at the edge of the melted area. So powder size is an important factor. The proper choices of particle scale and grain size will increase the melted zone and reduce the defect density of the compacted magnet.

Figure 4 is the FE-SEM image of the melted area at higher magnification. The nano-scale grains of the Nd$_2$Fe$_{14}$B phase

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>$B_r$ (T)</th>
<th>$\mu H_s$ (kA/m)</th>
<th>$\mu H_c$ (kA/m)</th>
<th>$(BH)_{max}$ (kJ/m$^3$)</th>
<th>$\sigma_c$ (MPa)</th>
<th>$\rho$ (g/cm$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>explosive compacted sample A</td>
<td>0.80/0.8</td>
<td>489</td>
<td>773/780</td>
<td>100/104.3</td>
<td>110.61</td>
<td>6.65</td>
</tr>
<tr>
<td>polymer-bonded sample B</td>
<td>0.63/0.8</td>
<td>412</td>
<td>696/780</td>
<td>67.5/104.3</td>
<td>65.86</td>
<td>5.87</td>
</tr>
<tr>
<td>explosive compacted sample in Ref. 7)</td>
<td>0.85/0.8</td>
<td>772/796</td>
<td>105.1/971</td>
<td>7.35</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Fig. 2 SEM micrograph of sample A and B. (a) sample A ($\times$200), (b) sample A ($\times$600), (c) sample B ($\times$600).
are revealed without the appreciable growth of the original grains even in the area that suffers the most drastic shock force.

Figure 5 is the MFM images of sample A. In Fig. 5(a), the topographic features appear in the left image and the fine magnetic-domain structure is demonstrated in the right. Fig. 5(b) is the rescanned image of Fig. 5(a) but at the higher magnification. It seems that domain structure of sample A is very similar to that of NdFeB nano-material reported in Ref. 8). Although some cracks appear in the topographic image, the domain structure is not disturbed and is still continuous despite of a little obscurity at the corresponding places. Figure 5(c) is the cross section analysis, showing the fine structure ordered 58 nm by the markers.

Figure 6 shows the magnetic force image of sample B, topographic microstructure in the left image and magnetic-domain structure in the right. For the influence of the resin binder, the quality of polishing preparation is not very good and some scratch marks appear in the left image. The same fine domain pattern as that of sample A can be distinguished in the right part.

From the MFM observation, it is shown that the micro-cracks almost have no effect on the magnetic domains structure as shown in Fig. 5, in which the magnetic domains are regular even at the particle boundaries. It owes to the very fine scale of magnetic domains in the small grains that keep uninjured. So the unchanged magnetic domain structure, the higher density and the absence of nonmagnetic binder make sample A have the superior magnetic properties. The nanometer scale discrimination shown in Fig. 5(c) also indicates that explosive compaction is a particularly promising technology to fabricate the nano-structured or amorphous NdFeB bulk magnet.

4. Conclusions

(1) Owing to the high pressure and high strain rates during the passage of shock wave, explosive compaction processing makes NdFeB particles more compact, and substantially improves the magnetic properties and the compressive strength of NdFeB bulk magnet comparing to the conventional resin bonding method. It appears to be a promising method to prepare the nano-structured bulk magnet from the melt-spun powders or amorphous powders.

(2) The fine grain size and the magnetic domain structure of the original powder are maintained after explosive compaction with the features of the melted area and the alveolate microstructure resulted from the shock wave at the edge of the melted area.

(3) The micro-cracks existing in the close-packed particles have no negative effects on the magnetic properties of the magnet since the magnetic domain structure is so fine that would not be disturbed by the discontinuities of such scale as micro-crack in the powders.

Acknowledgments

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Fig. 5  MFM image of sample A with the tip lift height of 50 nm. (a) Scan size of 20$\mu$m × 20$\mu$m, (b) Scan size of 5$\mu$m × 5$\mu$m, (c) Scan size of 5$\mu$m × 5$\mu$m.
REFERENCES


Fig. 6 MFM image of sample B, scan size of 5 μm × 5 μm, tip lift height 50 nm.