Microstructure of Explosively Compacted Nd-Fe-B Magnets

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The microstructure of explosively compacted Nd-Fe-B alloys has been investigated by means of transmission electron microscopy and X-ray diffraction. It is shown that there are three kinds of phases, i.e. the matrix phase Nd$_2$Fe$_{14}$B, O-rich phases and Nd-rich phases in explosively compacted Nd-Fe-B magnets. The matrix Nd$_2$Fe$_{14}$B phase is tetragonal, with lattice parameters of $a = 0.88$ nm and $c = 1.22$ nm. The O-rich phase was present at the two-grain boundary and three-grain junctions, with the fcc structure, and the lattice parameter is $a = 0.559$ nm. The O-rich phase belongs to the compound Nd-Fe-O, and the contents of O, Nd and Fe are 45–60 at% ; 20–40 at% and 10–21 at%, respectively. Also a few dislocations in the boundary phase have been observed.

1. Introduction

It is well known that permanent magnets based on the Nd-Fe-B system are normally prepared by the method of hot pressing.\cite{1-5} Besides, the explosive compacting process is one of the effective methods to produce magnets for special applications.\cite{6} Explosively compacted magnet has advantages such as rapid consolidation, simple technics, isotopic magnetic properties, etc.\cite{6,7} It is obvious that the hard magnetic properties of the magnets depend strongly on their microstructure, including distribution, composition, and magnetic properties of minority phases presented within magnets.\cite{1-5,7,11-14} Up to now, only a few investigations aimed at explaining the structural features and magnetic properties observed in explosively compacted magnets.\cite{6,10} However, further clarification of the microstructure, compositions and phase boundary is needed. Thus, the objective of this paper is to investigate some of aspects by X-ray diffraction and the transmission electron microscopy (TEM).

2. Experimental Procedure

Nd-Fe-B magnets was prepared by the explosive compacting process.\cite{10} The explosive compaction configuration is shown in Fig. 1. The mixture of the TNT (40 mass%) and the hexogen (also referred to as RDX, 60 mass%) was used in the explosive compacting, and with a denotation velocity of 6.5 km/s and a maximum pressure of 13 GPa on the steel tube.

The jet-milled powders (with an average particle size of 3.5 μm) are firstly packed to the bulk densities about 67% of the corresponding theoretical density ($\rho_{th} = 3900$ kg/m$^3$) and then explosively consolidated in the seamless steel tubes. The chemical composition of the specimen was analyzed to be Nd-13 mass%, Fe-79 mass%, B-3.6 mass%, Co-1.5 mass%, Nb-1.5 mass%, Pr-1.4 mass%. The magnetic properties of the magnet after the heat treatment are as follows: $B_r = 0.5$ kG, $J_H = 694$ kA/m, and $(BH)_{m} = 38.9$ kJ/m$^3$.

Thin film samples are prepared mechanically as thin as 50 μm, followed by ion milling for electron transparency. The crystalline structure was determined by X-ray diffractometer with Cu-K$\alpha$ radiation. The samples were observed using HITACH H-800 transmission electron microscopy (TEM) operated at 200 kV. Energy dispersive X-ray spectroscopy (EDX) of the magnet has been done with the probe size of 20 nm.

3. Results and Discussion

For the primary study of the characteristics of the 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. Table 1 lists the magnetic properties and compressed strength as well as density of the samples. It is shown that magnetic properties and compressed strength of the sample is improved by after annealing.
Figure 2 shows an X-ray diffraction pattern obtained from explosively compacted Nd-Fe-B magnets. The peaks observed correspond to the Nd$_2$Fe$_{14}$B phase. This confirms that the material is crystalline and contains predominantly the hard Nd$_2$Fe$_{14}$B phase. The amount of other possible phases is too little to be identified from this pattern.

In explosively compacted Nd-Fe-B magnets, there co-exist three kinds of phases. Figure 3 and Fig. 4 show the microstructure and the selected area diffraction (SAD) patterns of the explosively compacted Nd-Fe-B magnet. The composition analysis corresponding to the large-grain A in Fig. 3(a) is given in Fig. 5(a). According to the X-ray diffraction and the composition analysis, the large-grain A are the matrix phases, that the contents of Fe and Nd are 70–75 at% and 20–25 at%. According to the selected area diffraction (SAD) patterns (see Fig. 3(a)), this phase is identified as tetragonal (Tetra.) structure, which can be indexed as [001] Tetra, with the lattice parameters of $a = 0.884$ nm and $c = 1.224$ nm.

Figure 5(b) shows the composition of the three-grain junction (B) and two-grain boundary (C) in Fig. 3. Based on the composition analysis, the contents of O, Nd and Fe are 45–60 at%, 20–40 at% and 10–15 at%, respectively. It is deduced that the three-grain junction and two-grain boundary belongs to the compound Nd-Fe-O, and since the content of O is higher than that of Nd in the three-grain junction and two-grain boundary, it can be called as O-rich phase.

Figures 3(b) and (c) shows the microdiffraction and SAD patterns at the three-grain junction and two-grain boundary. Based on the selected area diffraction (SAD) patterns, the O-rich phase was identified to be face-cubic-centered (fcc) structure that is indexed as [011]fcc, and lattice parameter is
\( a = 0.559 \text{ nm} \), which is similar to the results of sintered magnet. However, the width of the grain boundary in explosively compacted samples is 30–300 nm in width about 10 times larger than the boundary width of the sintered sample.\(^8\) Also the content of \( O \) increased about 10 times larger than that of sintered sample. It is one of the reasons why the coercivity and energy product of explosively compacted magnets are lower than that of sintered magnets.

Besides the three-grain junction and two-grain boundary phases, there is some typical microstructure of boundary containing the different block-shaped phases (see Fig. 4). According to the composition analysis (see Fig. 5(c)), the different block-shaped phases belongs to compound Nd-O, and the contents of Nd and O are 80–85 at% and 10–15 at%, respectively. Figure 4 is the cases in which differently shaped Nd-rich phases exists as an independent grains at the grain boundary or in the matrix grain. Based on the selected area diffraction (SAD) patterns (see Fig. 4), the Nd-rich phase was identified as a hexagonal close packed (hcp) structure. It is determined that the lattice parameters are \( a = 0.395 \text{ nm} \) and \( c = 0.628 \text{ nm} \). Figure 6 shows a few dislocations in the boundary phase. Since mechanical properties of the grain boundary is weaker than matrix phase’s,\(^{15} \) in the process of explosive compacting or TEM sample fabricating, dislocation was formed in grain boundary. It is necessary to study further for clarify the cuase of dislocation.

Analytical TEM observation shows the Nd-rich phase with different shapes and compositions. It can be easily seen that the fourth element is oxygen which enters the alloy in the process of the explosive compaction. At the beginning of the explosively compaction, the temperature of the powders’ surface increases sharply, which accelerates the oxidation reaction at the surface of the powders. So the oxide is formed as the block-shaped phase during the subsequent consolidating process between the matrix phases in Nd-Fe-B explosively compacted alloys.

In order to understand the microstructure and the coercivity mechanism of explosive compacting Nd-Fe-B magnets, it is necessary to investigate the behaviors of the formation of the Nd-rich phase further.

4. Conclusions

There are three kinds of phases present in explosively compacted Nd-Fe-B magnets. The matrix \( \text{Nd}_2\text{Fe}_{14}\text{B} \) phase is tetragonal, with the lattice parameters of \( a = 0.88 \text{ nm} \) and \( c = 1.22 \text{ nm} \). The O-rich phase is present at the two-grain boundary and three-grain junctions, with the fcc structure, and the lattice parameter is \( a = 0.559 \text{ nm} \). A large number of different block-shaped Nd-rich phases in the Nd-Fe-B magnets were considered to be the Nd-O compounds with the hcp structure and the lattice parameters of \( a = 0.395 \text{ nm} \) and \( c = 0.628 \text{ nm} \). In the O-rich phase, the contents of O, Nd and Fe are 45–60 at%, 20–40 at% and 10–21 at%, respectively. The Nd-rich phase is considered to be Nd-O compound with the content of 80–85 at%Nd and 10–15 at%O.

Acknowledgements

The authors are grateful to professor Shan Aidang, Zhang Lanting, and Jiang Chuanhai for their helpful discussions. This work is supported by National Nature Science Foundation under Grant No. 50071035 and Shanghai Nature Science Foundation under Grant No. 02ZE14054.

REFERENCES


Fig. 6 Dislocations of the boundary phase in explosively compacted Nd-Fe-B magnets.