EB09

Interface Nanostructure of Cu-added Nd-Fe-B Sintered Magnets Processed by High Magnetic Fields

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Sintered Nd-Fe-B magnets consist of $Nd_2Fe_{14}B$ main phase with about 5 μm diameter and Nd-rich grain boundary phase. It is well known that the coercivity of sintered Nd-Fe-B magnets is highly dominated by this Nd-rich phase, which is reported [1] to change from discontinuous to continuous arrangement by a low temperature annealing around 500~600°C. We have shown [2,3] that the coercivity of sintered Nd-Fe-B magnets can be further increased by applying a high magnetic field during the annealing. We found that such a coercivity enhancement phenomena occurs only when a small amount of Cu is added, and when the annealing temperature is around 500°C or 550°C. In this paper, we report on the results of interface nanostructure analysis performed in order to investigate the reason why magnetic field effect occurs only in Cu-containing samples at specific annealing temperatures.

A series of sintered Nd-Fe-B samples is identical to that described in ref. [3] with the chemical composition $(Nd_{1-x}Dy_x)_{15}Fe_{79-y-z}B_6Al_yCu_z$. In order to identify any existing phase in sample, DSC measurements were performed by using a SETARAM DSC-111. Small angle neutron scattering (SANS) experiments were performed at JAEA to analyze an average fine structure in the interface.

In Fig. 1, we show the DSC curves of four Dy-free (x=0) samples with and without Al and Cu additives. In addition to a large endothermic peak around 310°C observed in every sample, which is owing to the Curie temperature of the main $Nd_2Fe_{14}B$, there is a marked endothermic reaction just below 500°C only for the Cu-containing ($z \neq 0$) samples. Moreover, we can notice an extra peak around 550°C, which has a significant intensity in samples with both Cu and Al additives. By inspecting a series of binary phase diagrams, we suggest that these temperatures correspond to the eutectic points of Nd-Cu and Al-Cu phases, respectively. These results therefore indicate that some amount of liquid phases coexist in the grain boundary at these temperatures. According to the SANS measurement, we deduced a structure such that a core of main phase is surrounded by a Cu-rich shell with a thickness of 4~8 nm. The DSC and SANS results thus suggest that an existence of the Cu-containing liquid phase is strongly correlated with the coercivity enhancement by the high field annealing.

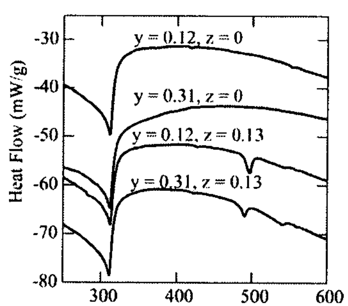

Fig. 1. DSC curves for Dy-free (x=0) samples recorded during heating from RT to 800°C at 5°C /min

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EB10

Mechanical Properties of Sintered NdFeB Magnets

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Since its development in 1983, the production and use of NdFeB magnet has grown rapidly. However, NdFeB magnet is rather brittle. Because of the brittleness, electric-spark machining and grinding are often used during its machining process, and its application is limited in some instances of demanding high fracture resistance, such as high-rpm motors, etc. The mechanical properties of sintered Nd-Fe-B magnet have attracted some researchers' attentions in recent years at the aim of improving its workability and increasing its dynamic applications[1-3].

In this article, the bending strength of three groups of samples with different oriented direction, which were cut off from the same bulk of sintered NdFeB magnet, was measured. The bending macrofractography was carefully observed and scanning electron microscope technique was employed to study microfractography. The anisotropy of mechanical properties and fracture behavior of sintered NdFeB magnets were emphatically investigated. The effects of the minor addition of grain-boundary alloys on the mechanical properties and microstructure of sintered Nd-Fe-B magnets were studied. The effect of Pr and Dy substituted for Nd, and the effect of Nd content on the impact stability of sintered Nd-Fe-B magnets were also studied by falling-weight impact test. In comparison, the impact stability of several kinds of permanent materials also was measured.

It shows that the bending strength and fracture behavior of sintered NdFeB magnets obviously display anisotropy. The fracture mechanism of sintered NdFeB magnets mainly appears intergranular fracture. Cleavage fracture was also observed in the group of $Nd_2Fe_{14}B$ specimens with their orientation direction parallel to tensile stress loaded by test machine. Minor additions of the grain-boundary alloys made the distribution of grain-boundary phase in magnets more uniform, and in this study, sintered NdFeB magnets prepared by dual alloys have a mucher higher bending strength (reached 397MPa) than the magnets prepared by the single alloy. the impact stability of sintered Nd-Fe-B magnets linely increases with increasing Nd contents. The increased Nd-rich phase observed in the microfractograghs, may contribute to the improvement of impact stability by its plastically deforming. The heavy rare-earth element of Dy substitution for Nd helps improve the impact stability of sintered Nd-Fe-B magnets. It is probably caused by the lanthanide contraction along c-axis of $R_2Fe_{14}B$ compounds, that has important influences on the elastic modulus of matrix, and the needed energy of specimen fracture increases with increasing elastic modulus.The light rare-earth element of Pr substitution for Nd deteriorated the impact stability of magnets.

Table I Impact stability comparison between commercial and experimental magnets

Materials	Maximum Energy Product (MGOe)	Impact stability (J 10^{-3})
AlNiCo magnets	5~10	60
$SmCo_5$-type magnets	17~20	32
Sm_2Co_{17}-type magnets	20~30	42
$Nd_2Fe_{14}B$ magnets	30~50	156
Experimental magnets	22~38	370

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