Progress of Sm-Fe-N Anisotropic Magnets

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Abstract: A reduction and diffusion method (R/D) is used to make a mother alloy of Sm-Fe-N anisotropic magnets. Reduction of 0.5wt% of samarium content compared to the conventional powder increases magnetization. Milling condition and surface treatment improve the squareness of demagnetization curve, the aging property and the heat resistance. The maximum energy product of 292 kJ/m³ is obtained with the powder. High coercive force is maintainable even if the powder is exposed for 300h in 80 °C 90%RH. The maximum energy product of 141 kJ/m³ is obtained with an injection molded anisotropic magnet. The aging property estimated by irreversible flux loss is comparable to the conventional MQP-B magnets. The heat resistance temperature ($T_{-5\%}$) at which the initial irreversible flux loss becomes -5% is 125-more than 150 °C for Sm-Fe-N magnets and 150–170 °C for hybrid magnets. The magnetic properties of bonded HDDR Nd-Fe-B magnet were improved by substituting for Nd-Fe-B powder with Sm-Fe-N powder. A new technology to make anisotropic bonded Sm-Fe-N thin cylinder magnets by an injection molding using unsaturated polyester (UP) resin was developed.

Key words: bonded; Sm-Fe-N; anisotropic; magnet; reduction; diffusion; injection

1 Introduction

Bonded rare-earth magnets are indispensable to computer and audio-visual applications. Most of them are bonded Nd-Fe-B isotropic magnets whose maximum energy product is almost 90 kJ/m³ in compression type. However, it is a limit of isotropic magnets. They are made from MQ powder (MQP) that is supplied by Magnequench International.

On the other hand, Sm-Fe-N magnet powder, Nd-Fe-B anisotropic magnet powder produced by HDDR and nanocomposite Nd-Fe-B magnet powder have been developed aiming at high performance. Bonded magnets made from these powders have been proposed to the market.

An Sm-Fe-N magnet is produced by an introduction of nitrogen into an Sm-Fe mother alloy. Sintering is not applicable, because the compound decomposes at high temperature more than $600~^{\circ}$ C. Therefore the application is limited to a bonded magnet at present.

Two types of Sm-Fe-N magnet were reported; one is the anisotropic or isotropic $Sm_2Fe_{17}N_x$ magnet powder with a Th_2Zn_{17} structure^[1-3] and the other is the isotropic $SmFe_7N_x$ powder with a $TbCu_7$ structure^[4,5].

We chose Sm₂Fe₁₇N_x and have started commercial production of bonded magnet up to 95 kJ/m³ from 1999^[6]. In the process, an Sm₂Fe₁₇ mother alloy is made by a reduction and diffusion (R/D) method for the benefit of the cost-performance^[7]. Thereafter we improved the whole process and made the magnetic properties and stability higher.

2 Experimental Procedures

Magnetic properties of powder are measured by a vibrating sample magnetometer (VSM). Because Sm-Fe-N powder developed is anisotropic, the sample is made as follows: the powder is put into a plastic case with paraffin powder, heated up to 80 ℃ and cooled down to room temperature in a magnetic field of 2 T.

Magnetic properties of bonded magnets are measured by a hysteresis loop tracer using an electromagnet. The sample size is $\phi 10 \text{ mm} \times 7 \text{ mm}$. The sample is magnetized by a pulse magnetic field of 7 T before it is set on the pole pieces.

Both irreversible flux loss and initial flux loss are measured by a flux meter. The sample size is $\phi 10$ mm×7 mm, the permeance of which is almost two. The sample is magnetized by a pulse magnetic field of 7 T

beforehand.

Sample name is as follows: S3 means injection molded Sm-Fe-N magnets and S4 means injection molded hybrid Sm-Fe-N and ferrite magnets. 14M, 10M, 5M and 3M means grades of magnet. It means that the maximum energy products of them are 112, 80, 40 and 24 kJ/m³ respectively.

3 Results and Discussion

Fig.1 shows a manufacturing process of the Sm-Fe-N magnet powder. A feature of the process is to make Sm₂Fe₁₇ mother alloy powder by R/D method. Powder of samarium oxide and iron metal and calcium metal granules are mixed together. They are heated at about 1100 °C in an Ar atmosphere for several hours.

The reaction occurs as follows:

 $Sm_2O_3+17Fe+3Ca \rightarrow Sm_2Fe_{17}+3CaO$.

Samarium oxide is reduced by calcium to samarium metal that diffuses into iron metal to form a samarium-iron alloy. The reaction produces an ingot containing both Sm₂Fe₁₇ alloy and calcium oxide. Because the calcium oxide is soluble to the water, Sm₂Fe₁₇ alloy powder is easily obtained by washing the ingot under water.

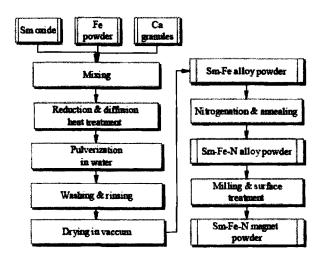


Fig. 1 Manufacturing process of Sm-Fe-N magnet powder

 $\rm Sm_2Fe_{17}N_x$ alloy is made by heating the powder under ammonia and hydrogen gases atmosphere at the range of 450 to 500 °C. The magnet powder is obtained by milling the alloy and by surface treatment of the fine powder.

Magnetic properties of bonded anisotropic mag-

nets depend on those of the magnet powder, the degree of crystal axis alignment and volume ratio of the powder.

Fig.2 shows a relationship between the magnetization and Sm content of Sm₂Fe₁₇N_x. Usually, the samarium content of Sm₂Fe₁₇N_x is richer than that of stoichiometry in order to avoid unreacted iron phases. However excess samarium forms SmFe₃N_x and SmFe₂N_x phases that deteriorate magnetic properties. Thus the samarium content should be close to the stoichiometry. We have succeeded to increase magnetization by reducing 0.5 wt.% of samarium compared to conventional powder through modification of R/D process. On the other hand, the coercive force of $Sm_2Fe_{17}N_x$ powder increases by decreasing nucleation sites such as a soft magnetic phase or crystal defects on the surface of the powder. We also modified both milling and surface treatment process and have succeeded to improve the squareness of the demagnetization curve.

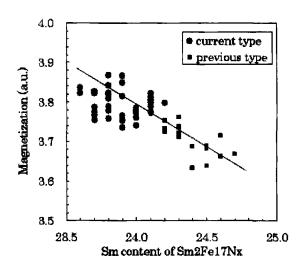


Fig. 2 Relationship between magnetization and Sm content of Sm₂Fe₁₇N_x

Fig.3 shows an example of the magnetization curve of currently mass-produced powder measured by VSM. The maximum energy product is 292 kJ/m³ that is 22% higher than that of conventional powder that has 240 kJ/m³ on the average.

Modified milling and surface treatment process have also improved the stability of the fine powder. The powder is so small in size that the moisture resistance and the handling capability in air were not enough.

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