VACUUM SINTERING BEHAVIOUR OF NdFeB MAGNETS

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Introduction

The dimensional changes that occur during the sintering of NdFeB magnets have been investigated using a specially constructed high temperature vacuum dilatometer. These measurements have been made on samples produced by the conventional powder metallurgical method and on compacts produced using hydrogen decrepitation (HD)[1], as a pre-milling technique.

Other experiments have been carried out on the amount of shrinkage in the a and c directions of sintered compacts produced with a range of aligning fields.

Experimental

Samples of powder were produced by conventional powder processing and by the hydrogen decrepitation route and pressed statically and un-aligned at 1600 kgm^{-2}. The compacts were then sintered under argon at a pressure of 460 mmHg by raising the temperature from 20 to 1100°C at a rate of 2°C/min. in the dilatometer.

As the temperature was increased the HD compact showed a 0.6% contraction between 110 and 350°C, Fig. 1. This contraction is attributed to the loss of hydrogen from the $Nd_2Fe_{14}B$ matrix phase, and compares well with the value calculated from lattice expansion measurements. The conventionally produced compacts showed no sign of this contraction (Fig. 1). Above 700°C a pronounced contraction is again observed in the HD compact, again this shrinkage is not observed in the conventionally milled compact. Hydrogen loss from the Nd-rich intergranular phase between 700 and 900°C, previously confirmed by mass spectrometer measurements[2], appears to be the reason for this shrinkage. As the temperature increases above 900°C both samples show signs of liquid phase sintering, indicated by a considerable contraction, to form the fully dense sintered compact. Figure 1 shows evidence for an increased rate of sintering in the hydrogen containing material.

The effect of aligning field, when pressing a 'green' compact, on the resulting shrinkage during sintering has also provided interesting results. Compacts produced from hydrogen decrepitated and milled material were pressed at 750 kgm^{-2} and sintered at 1080°C. The fields used in the perpendicular aligning press were in the range 0 to 1.23 T. After sintering, the lengths and widths of the magnets were measured. The resulting size variations can be seen in Fig. 2. The width of the sintered magnet, parallel to the alignment direction, was reduced by ~5% as the alignment field was increased from 0 to 1.23 T, and the length increased by ~3.5% with the same increase in field. The variation in the extent of shrinkage appears to be due to increased growth in the a-axis direction during sintering. This is confirmed by subsequent measurements made using a VIDS II image analyser. Fully aligned magnets were mounted, polished, and measurements made on the grain size viewed from the a-axis direction and the c-axis direction. This

confirmed that the 8-9% directional difference in shrinkage, in a fully aligned magnet could be attributed to the increased growth of grains in the a-direction.

Conclusions

The experiments carried out on the shrinkage of magnets during sintering and the shape changes of compacts measured after sintering have provided useful information. Compacts pressed from powder using the HD/milling route exhibit greater shrinkage at lower temperatures i.e. below 900°C when compared to the conventionally produced powder. This has been attributed to the loss of hydrogen in the first stage from the $Nd_2Fe_{14}B$ matrix phase, and the secondary stage betwen 700 and 900°C is loss of hydrogen from the Nd-rich phase. A second important point to note is that sintering begins at lower temperatures and proceeds more rapidly in the hydrogen containing material, this has been shown to produce excessive grain growth in HD produced magnets unless the sintering temperature and time are carefully controlled. This is important if good magnetic properties are to be achieved[3]. The shrinkage measurements made on the sintered compacts produced with varying aligning fields shows how the growth of the sample in the a-direction is greater than that in the c-direction, this provides important information for manufacturers wishing to know the optimum pressed compact size for a required final sintered shape.

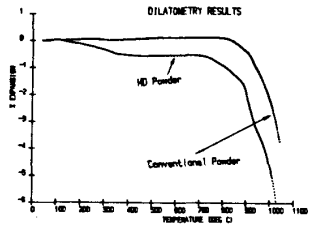

Figure 1

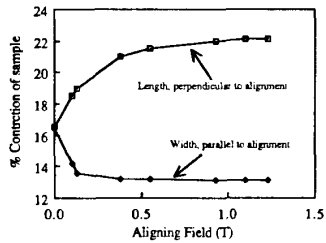

Figure 2

References

/1/ McGuiness, P.J., Harris, I.R., Rozendaal, E., Ormerod, J. and Ward, M.J. J. Mat. Sci. 21, 4107 (1986).

/2/ Harris, I.R., McGuiness, P.J., Jones, D.G.R., Abell, J.S., Physica Scripta, Vol. 9, 435 (1987).

/3/ McGuiness, P.J. and Harris, I.R. Presented at Intermag-MMM (1988).