

# THE EFFECT OF ALLOY ADDITIONS ON THE MAGNETIC PROPERTIES OF RFeB SINTERED MAGNETS

Dr. A. Ahmad\*

## ABSTRACT

The RFeB system has been developed over the last fifteen years to provide permanent magnets of the highest quality for advanced applications. Although these magnets exhibit high saturation and good coercivity, their poor corrosion resistance and high temperature properties has led to considerable research into improving these properties through alloying additions.

The paper describes the production of sintered RFeB based magnets by conventional powder metallurgical techniques and by blending with fine particles of other elements such as Zr. Properties of the magnets made from Jet milled and ball milled powders have been compared. Both the effectiveness of blending as a process and the variation of properties by varying Zr content have been investigated. The effects of heat-treatment on the magnetic properties and microstructure are also investigated.

## INTRODUCTION

Permanent magnets provide a constant magnetic field in an air gap without the continuous expenditure of energy required by an electromagnet. Their uses range from low technology applications such as fridge magnets to state of the art applications such as MRI scanners. The type of magnetic material selected for each application depends on a number of factors. The key properties are usually the magnetic field produced by a given volume of material and its resistance to demagnetisation. Fig.1 shows how the magnetic properties of the most common magnetic materials vary. It can be seen from fig. 1 that for any static gap application a smaller volume of NdFeB could be used in place of the ferrites or Alnico which are commonly used.

The first studies of NdFeB system were made in the Ukrain (1) where the existence of three stable ternary phases was reported. Further investigation revealed the ternary phases to be

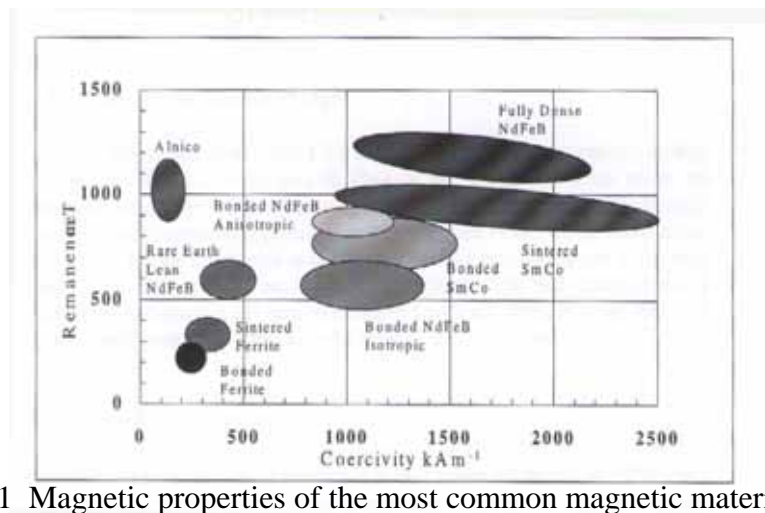


Fig. 1 Magnetic properties of the most common magnetic materials

$\text{Nd}_2\text{Fe}_{14}\text{B}$ ,  $\text{NdFe}_4\text{B}_4$  and  $\text{Nd}_5\text{Fe}_2\text{B}_6$  (2,3,4) The phase responsible for the exceptional hard magnetic

\* Associate Professor, Department of Met Engineering and Materials Science University of Engineering and Technology, Lahore

properties was identified as being the 2:14:1 phase a tetragonal compound with its easy direction of magnetization along the c-axis. The magnets are produced by sintering a green compact of NdFeB powder. The powder is produced by ball milling an alloy of standard composition e.g.  $\text{Nd}_{16}\text{Fe}_{76}\text{B}_8$ . This process was first employed for the production of  $\text{SmCo}_5$  and was adapted for NdFeB by Sagawa and coworkers at Sumitomo. The material is subjected to hydrogen decrepitation before milling (5).

The main method of varying the composition of a sintered magnet is to change the composition of the starting alloy at the pre-casting stage. Although alloy modification is the method used in much of the literature, powder blending provides a practical alternative with distinct advantages (6,7). The blending process used in this work is developed by Ahmad, Mottram and Harris and is described elsewhere in detail (8,9).

### EXPERIMENTAL WORK

Fig. 2 shows a schematic representation of the processing route employed. The blending process employed in this work involves simple powder mixing. In this process pre-milled powder with suitable particle size distribution were mixed together before being aligned and pressed. This was done by tumbling the NdFeB and additive powder in a sealed pot containing stainless steel milling balls. It was found that a tumbling time of 1 hour was suitable to give a homogeneous mixture of powder.

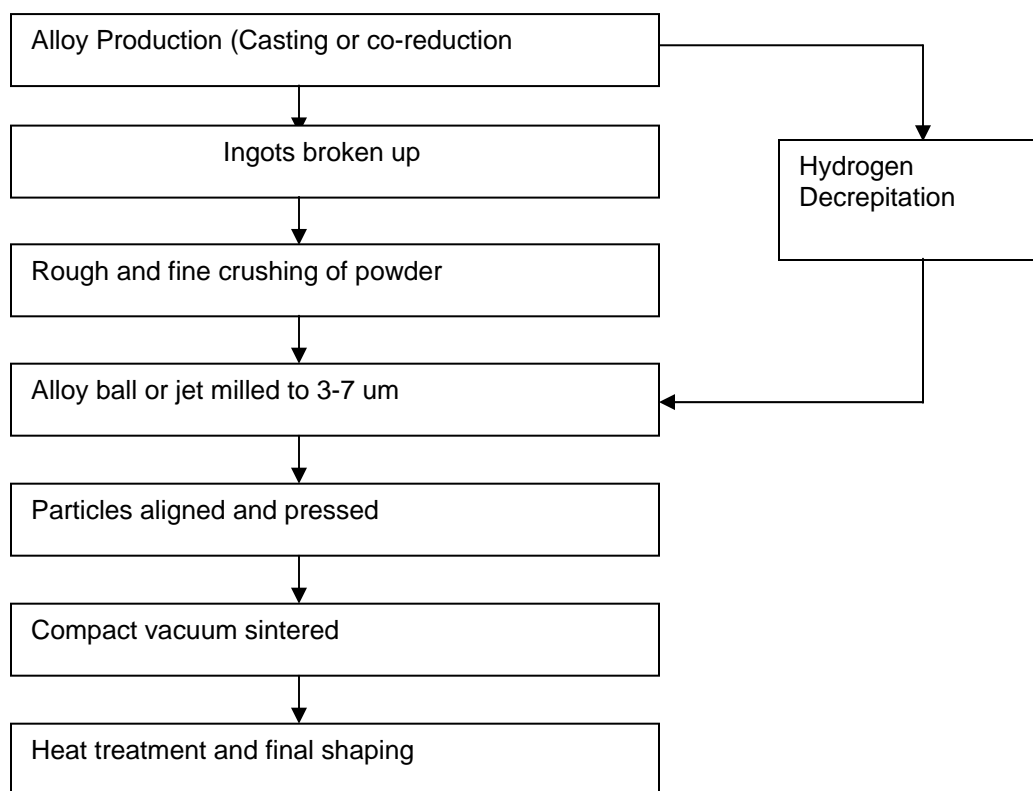


Fig. 2 Processing Route For Sintered Magnets

Sintering of the green compacts was carried out in a vacuum furnace system as shown in Fig. 3, using the temperature profile shown in fig. 4. After sintering any oxide from the sample was

removed from the surface of the magnet using wet grinding paper. The samples were magnetised for magnetic measurements by using a pulse magnetiser. The pulser consists of a bank of capacitors that discharge through a solenoid producing an internal field of 4.5 Tesla. A permeameter was used to obtain demagnetisation curves from which remanence, coercivity and  $BH_{max}$  were calculated. A Pendulum Balance was used to carry out thermomagnetic measurements and assess Curie temperature. SEM-Jeol 6300 was used to obtain images of microstructures and to carry out EDX analysis on samples.

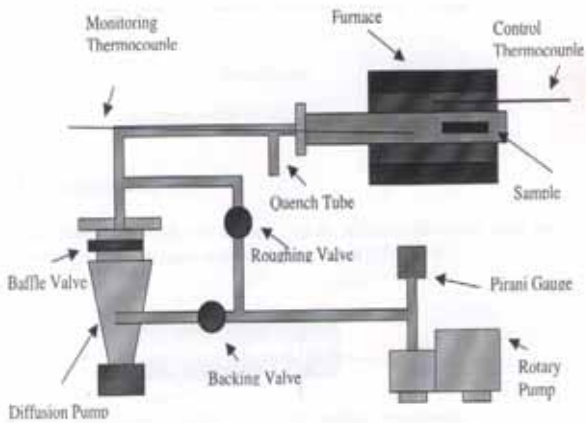


Fig. 3 Vacuum Sintering Apparatus

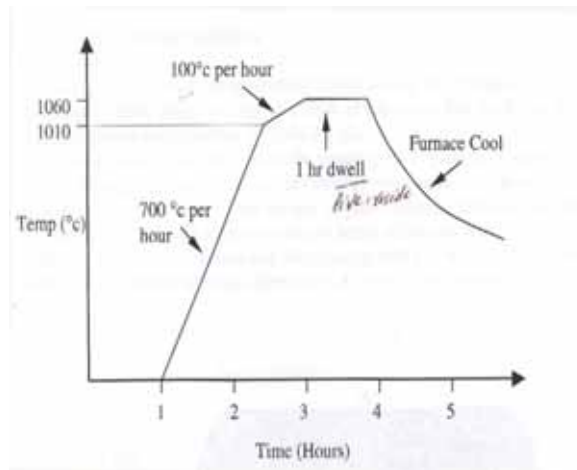


Fig. 4 Temperature Profile For Sintering

## RESULTS AND DISCUSSION

Two types of NdFeB powder were used to produce magnets-freshly (ball) milled powder and a batch of powder jet milled at Crumax, UK. Table 1 shows the average properties of magnets produced with each of these powders. Three magnets were made with each powder, yielding a number of test samples for the permeameter and pendulum balance. It can be seen that the properties of the magnets sintered from freshly milled powder are better than those from the jet milled powder. This is mainly due to the oxidation of the powder.

Table No. 1 Summary of Properties from different powders,

A = As sintered, B = heat treated 1 hr/600 °C

| Powder      |   | Br (mT) | iHc (kA/m) | bHc (kA/m) | BH(max)<br>(kJ/m <sup>3</sup> ) | Squareness<br>% | Curie Temp °C |
|-------------|---|---------|------------|------------|---------------------------------|-----------------|---------------|
| Jet Milled  | A | 1204    | 634        | 598        | 231                             | 0.65            | 312           |
|             | B | 1208    | 751        | 696        | 232                             | 0.78            | -             |
| Ball Milled | A | 1233    | 696        | 663        | 285                             | 0.89            | 312           |
|             | B | 1240    | 854        | 777        | 291                             | 0.85            | -             |

Table 2 shows the magnetic properties of the sintered magnets blended with 0.1 to 0.5 at % Zr. Blending with Zr has led to a decrease in both remanence and coercivity, which is not recovered by post sintering heat treatment. Despite all samples being fully dense ( $7.5 \text{ g/cm}^3$ ) the remanence of these magnets begins to fall with even relatively small Zr addition. An explanation for this is obvious from the backscattered SEM image as shown in Fig. 5. It can be seen that there is much more of the lighter Nd rich phase present than in the standard 16/76/8 microstructure (Fig.6).

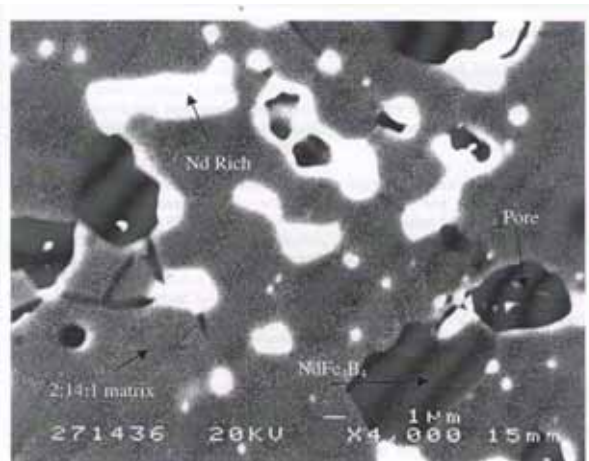
Table No. 2 Properties of  $\text{Nd}_{16}\text{Fe}_{76}\text{B}_8$  magnets blended with zirconium

A = As sintered, B= After heat treatment at  $600 \text{ }^\circ\text{C}$  for 1 hour

| At % Zr | Sample | Br $\pm 10$<br>(mT) | iHc $\pm 5$<br>(kA/m) | iHc $\pm 5$<br>(kA/m) | BH(max)<br>$\pm 15$<br>(kJ/m <sup>3</sup> ) | Squareness<br>% | Curie<br>Temperature<br>$\pm 3 \text{ }^\circ\text{C}$ |
|---------|--------|---------------------|-----------------------|-----------------------|---|-----------------|--|
| 0.1     | A      | 1254                | 628                   | 613                   | 290   | 0.91            | -  |
|         | B      | -                   | -                     | -                     | -   | -               | -  |
| 0.3     | A      | 1232                | 535                   | 423                   | 191   | 0.44            | 312  |
|         | B      | 1250                | 547                   | 500                   | 266   | 0.72            | -  |
| 0.5     | A      | 1226                | 378                   | 320                   | 144   | 0.34            | 312  |
|         | B      | 1227                | 447                   | 396                   | 202   | 0.51            | -  |



Fig. 5 Backscattered SEM image of Fig. 6  
Zr blended magnet



Backscattered SEM image of  $\text{Nd}_{16}\text{Fe}_{76}\text{B}_8$   
magnet

It also appears that this increase has prevented the formation of the 2:14:1 matrix phase. This happens because the Zr has combined preferentially with boron during sintering, causing a depletion in the amount of boron available for formation of the matrix phase. This is supported by the EDX results which indicated that there was no Zr in the matrix phase.

The apparent decrease in coercivity is more difficult to explain. The increased amount of Nd rich and Zr phases between the matrix grains should give better magnetic isolation and an increase in coercivity. A possible explanation for this is that as boron combines preferentially with Zr, excess iron remains in the Nd rich phase, probably forming soft magnetic  $\text{Nd}_2\text{Fe}_{17}$ . This would have a

dramatic effect on coercivity, even at low compositions. The presence of such soft magnetic phases would explain the poor demagnetisation loop shapes as shown in figs. 7 and 8. Fig. 9 shows the pendulum balance trace for 0.5 at % Zr blended magnet.

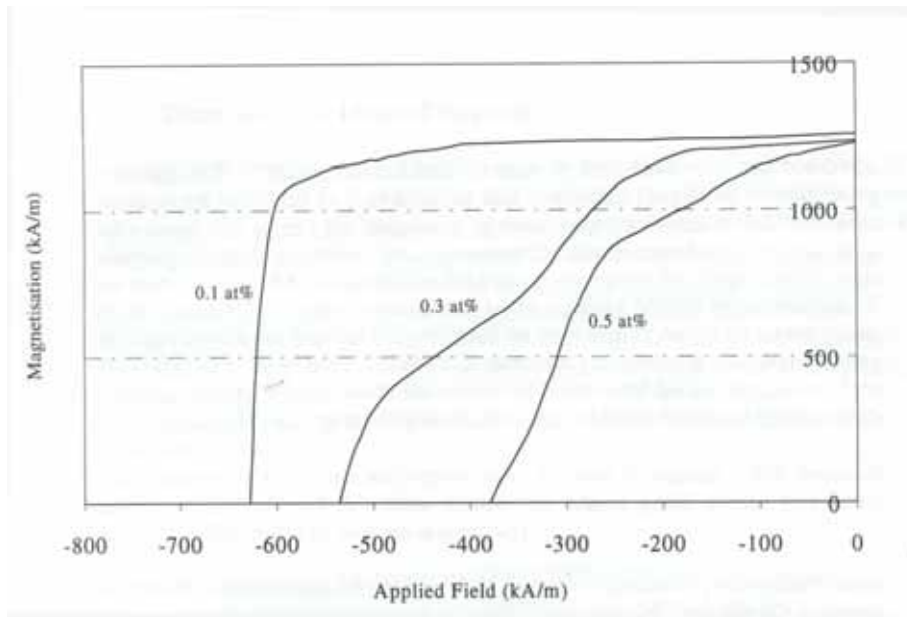


Fig. 7 Demagnetisation curves for Zr blended magnets (as sintered)

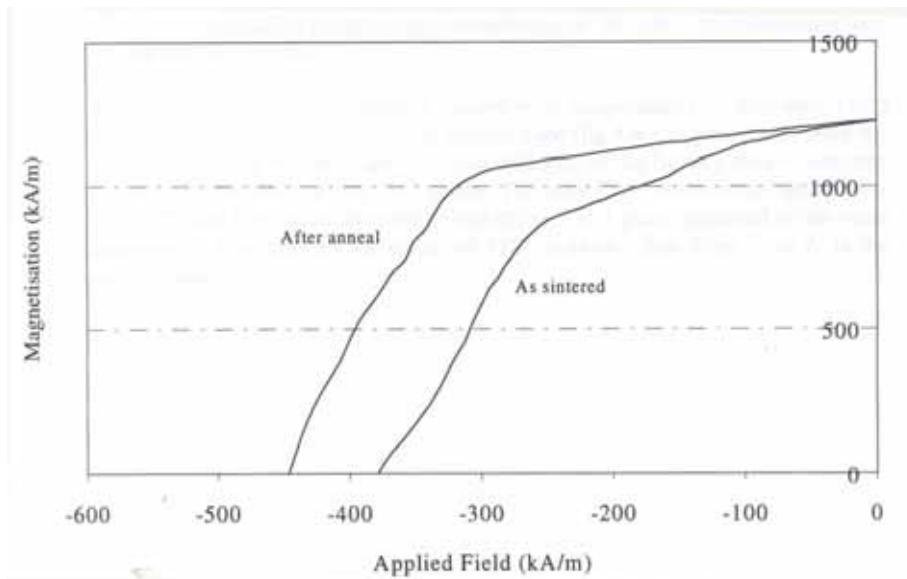


Fig. 8 Effect of Heat treatment for 1 hour at 600 C on 0.5 at % Zr

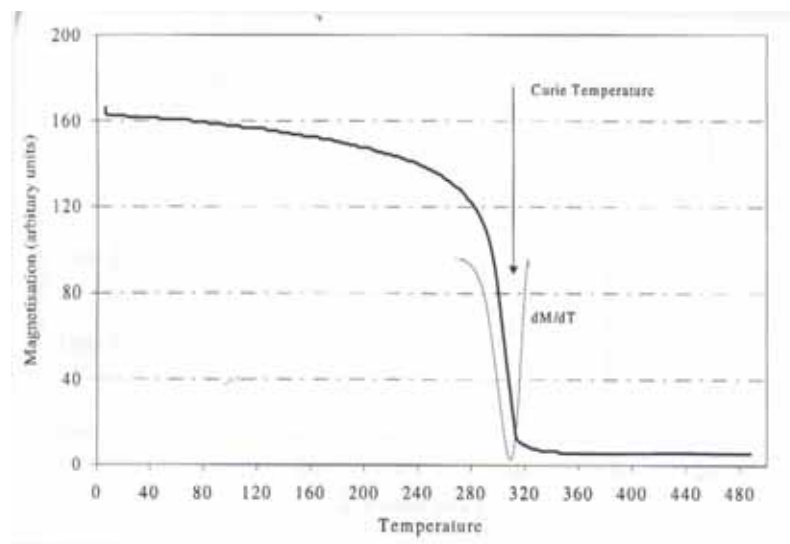


Fig. 9 Pendulum balance trace for 0.5 at % Zr blended magnet

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