EFFECT OF ZIRCONIUM ADDITION ON NEODYMIUM-IRON –BORON (NdFeB) SINTERED MAGNET

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Introduction.

Neodymium-Iron-Boron (NdFeB) produced by powder metallurgy technique is a very promising material for permanent magnet due to its high magnetic product (BH)max of up to 55 MGOe [1]. Thus it is possible to produce smaller magnet without scarifying its magnetic properties. There are several processing techniques available to produce powder for sintered anisotropic NdFeB permanent magnet such as atomisation, crushing [2] and hydrogen-disproportionation-desorption-recombination (HDDR) [3]. In the present work, the NdFeB powders with and without Zr produced by crushing were used to produce magnet by powder metallurgy route. The objectives of the current works are to study the effect of zirconium on the density of the compacts and also the magnetic properties of the magnet.

Experimental

Materials

NdFeB permanent magnet materials with and without zirconium (Zr) were vacuum melted and cast on a chilled copper mould. The alloys were then crushed and milled to form powders. The powders were compacted at 250 MPa using field press with applied field of 2 Tesla in order to align the magnetic domain of the material. The compacts were then sintered under vacuum over a range of temperatures starting from 1000°C to 1080°C for one hour. The density of the sintered compacts was measured by pycnometer. The magnetic properties of the compacts were measured by vibratory sample magnetometer (VSM) with applied field of 2.5 Tesla.

Apparatus and procedures

The chemical compositions of the as-cast alloys were obtained by Inductive Couple Plasma (ICP-OES). The microstructures of the alloy and the sintered magnet were studied by optical microscope and LOE Field Emission Scanning Electron Microscope (FESEM) attached with Energy Dispersive Spectroscopy (EDS). The density of the sintered magnet was measured by pycnometer and the magnetic properties of the sintered magnets were measured by DMS Vibratory Sample Magnetometer (VSM) at saturation value of 2.5 Tesla.

Results and Discussion

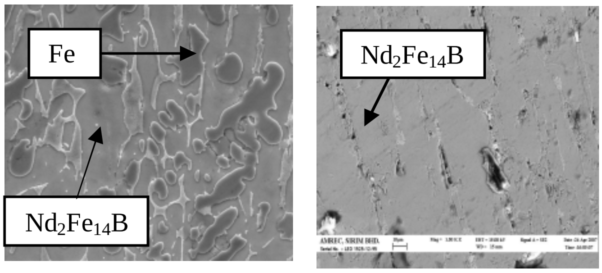
The chemical compositions of the materials are shown in Table 1:

Table 1: Chemical composition of the materials

Sample	Composition (Wt.%)				
	Nd	Fe	B	Zr	0
NdFeB	30.08	68.73	1.07	-	0.12
NdFeB-Zr	30.88	66.90	1.00	1.00	0.22

The scanning electron images of Samples NdFeB and NdFeB-Zr are shown in Figures 1a, and 1b respectively. Four distinct phases were observed in as-cast NdFeB materials. These phases are ϕ phase ($Nd_2Fe_{14}B$), η phase, Nd rich phase, free iron phase and Nd_2Fe_{17} (Ψ) phase. From Figure 1a, it can be clearly seen that free iron phases are present besides ϕ phase ($Nd_2Fe_{14}B$) and Nd_2Fe_{17} (Ψ) phase. This free iron phase is believed to be detrimental to the magnetic properties and cause difficulty in milling processes due to its high ductility. The present of these phases are easily identified by virtue of their morphologies as well as EDX analysis. By EDS analysis it is found that ϕ phase ($Nd_2Fe_{14}B$) contains 26.7 wt.% Nd, free iron phase has 99.5 wt.% iron and Nd_2Fe_{17} (Ψ) phase has 77.4 wt.% iron .

Figure 3 shows the optical micrographs of the compacts after sintering at 1020ºC under vacuum for one hour. The samples were cut, mounted on the bakelite, ground, polished and etched with 3% Nital to reveal the microstructure. The presence of free iron is obvious in NdFeB. As for NdFeB-Zr there is no presence of free iron observed in the microstructure and the grain boundaries are well defined compared to NdFeB which is believed to increase the coercivity of the material.



(a) (b)

Figure 1: SEM image of as cast materials a) NdFeB b) NdFeB-Zr

The effect of sintering temperature on the density of NdFeB with and without Zr is shown in Figure 2. Increasing sintering temperature increased the density of the compacts for both materials due to liquid phase sintering. The maximum density of 7.5 g/cm^3 was achieved by sintering at 1020ºC. Further increase in sintering temperature above 1020ºC did not change the density of both materials studied.

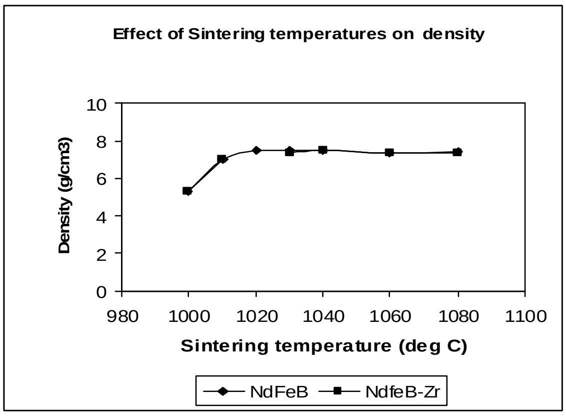
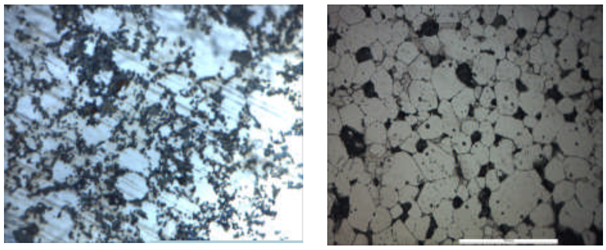


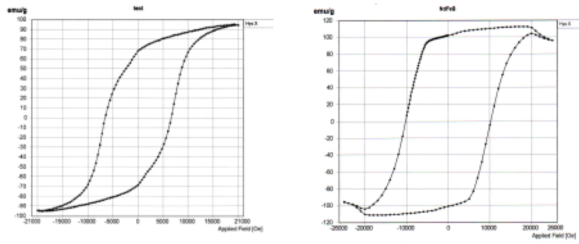
Figure 2: Effect of sintering temperatures on the density of NdFeB and NdFeB-Zr compacts

The magnetic properties of NdFeB with and without Zr addition are shown in Figure 4. Magnetic properties of NdFeB with Zr addition are higher compared to without Zr due to its distinct grain boundary and the absence of free iron. The coercivity (Hc) for NdFeB and NdFeB-Zr are 7.0 KOe and 10.0 KOe respectively. The magnetization saturation (Mr) for NdFeB and NdFeB-Zr are 70 emu/g and 100 emu/g resperctively. The quareness of the hyteresis loop for NdFeB-Zr is better compared to NdFeB. The increase in Hc in NdFeB-Zr alloy compared to NdFeB is believed to be due to the much distinct grain boundaries in the former. The increase in Mr in NdFeB-Zr compared to NdFeB is believed to be due to the absence of free iron as this phase was inhibited by the presence of Zr.



(a) (b)

Figure 3: Optical micrograph of the sintered magnet a) NdFeB b) NdFeB-Zr



(a) **(b)**

Figure 4: Hysteresis loops of a) NdFeB b) NdFeB-Zr

Conclusion

The NdFeB alloys with and without Zr contents were cast and their microstructures and magnetic properties were successfully studied. In Sample NdFeB, free iron phase exists together with ϕ phase and Nd_2Fe_{17} (Ψ). In NdFeB Zr alloy there was no free iron observed indicating that Zr addition has inhibited the formation of free iron during casting. The maximum density of 7.5 g/mm^3 was achieved by sintering at 1020ºC for one hour for both materials. The magnetic properties of NdFeB –Zr is higher than NdFeB due to the absence of free iron and distinct grain boundary in the material.

References

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