FABRICATION AND CHARACTERIZATION OF Nd-Fe-B MAGNETIC ALLOY PREPARED BY ARC MELTING FURNACE METHOD FOR PERMANENT MAGNET

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ABSTRACT

Magnetic alloys of nominal Nd_{12}Fe_{72}B_{6} (at. %) compositions which were prepared by a vacuum arc melting furnace is discussed in this paper. For ingot preparation, cylindrical casting dies of 5 mm diameter respectively, has been used. Based on visual observation, both the cylindrical and the plate shaped ingots obtained from casting appear to be very spotless and shiny, and free from oxidation. The two differently shaped Nd-Fe-B ingots were further studied by means of XRD and SEM. Results of XRD indicated that alloys of Nd_{12}Fe_{72}B_{6} (at. %) composition was confirmed as the single phase alloys with Nd_{2}Fe_{14}B as the only phase. The crystalline orientation is instantly observable on the cross section area of the ingots as observed by SEM method. Phase identification was also studied by a DSC calorimeter in which the thermo scan spectrum shows the appearance of a small and tiny peak at a temperature between 312 and 317 °C. The value is very close to the Curie temperature for Nd_{2}Fe_{14}B phase as reported in various publications. It is concluded that solidification of molten Nd-Fe-B alloys in a plate shape casting die would results in an unblemished and oxidation free ingot alloys. The alloy is suitable as the main component for sintered Nd-Fe-B permanent magnets preparation.

Keywords: Nd-Fe-B, permanent magnets, solidification, arc melting furnace

INTRODUCTION

Permanent magnet plays an important role particularly in the advancement of today's industry. Permanent magnets have found applications in many devices and industries, such as DC motors, actuators, generators, in the automotive industry and as well as in household appliances. Urgent demands for permanent magnetic materials are necessary to encourage the development of permanent magnet materials, both through engineering methods as well as via materials manufacturing or synthesis.

Research in the development of permanent magnets using starting materials based on rare earth metals which are particularly rich in Nd (Nd-rich) and Fe metal (Fe-rich) elements, and endowed with superior magnetic properties, is still being conducted by many researchers. And the same situation could also be said for other magnet materials such as alloys-based magnetic systems rare earth metal RE-TM-B [1-5], Sm-Co [6-8] and also the ceramic system like MO.6 Fe_{2}O_{3} (M = Ba or Sr) [9-10]. Engineering preparation process employing both conventional techniques such as Powder Metallurgy method [11] as well as
modern techniques such as Rapid Solidification method [12] utilized in the manufacture of permanent magnets, has enabled scientists to generate magnetic extrinsic properties which come within the 90-100% range of the intrinsic value. Such methods can not be separated from the preparation phase of an alloy in order to obtain hard magnetic materials as expected. Melting process followed by solidification is expected to govern the formation of crystals. In this research, the fabrication process of magnetic material alloy prepared by arc melting furnace method is described and then discussed.

EXPERIMENTAL METHOD

Sample Preparation

Compiler alloy elements such as Fe and B magnets in the form of powder or solid (granular) were purchased from Merck (99%), whereas Nd (99%) which is very susceptible to oxidation levels has been ordered from Johnson Mathey. Preparation of Nd$_2$Fe$_{14}$B ingot starts with determining the weight fraction of the elements. It was determined that to synthesize Nd$_2$Fe$_{14}$B, the fractional weights of 27% Nd, 72% Fe, and 1% B are needed. The amount required for smelting by arc melting furnace is 4 grams. Smelting tubes were evacuated using a rotary pump and subsequently cleansed by employing argon gas to flush out the remaining air in the tube. Melting elements and all the magnetic compiler base elements are now enclosed in an inert tube having a maximum temperature of 4000°C which ensures that all elements are fused together. The alloy is rapidly cooled down from melting condition via the printing process (casting) with the target drawn on the mold by exploiting the difference in air pressure. In this manner Nd$_2$Fe$_{14}$B ingots having cylindrical shape of five mm diameter each are obtained.

Magnetic Measurement

a. Magnetic Phase Identification

The Nd$_2$Fe$_{14}$B magnetic phase formation was determined by using XRD. XRD intensity measurement in the diffraction angle $2\theta$ interval between 15° to 105° (Phillips with 40 kV and 30 mA) was carried out on magnetic materials prepared in the form of powder or solid flat to ensure detection of crystal fields (miller index) [13] of the magnetic phase. The software package APD [14] has been employed to determine the diffraction peaks (peaks search) from the crystal planes. From the crystal planes that were detected, the crystal plane distance ($d_{hkl}$) could readily be found. Qualitative analysis was completed by matching the results of the APD software calculation with the prerecorded magnetic phases listed in the ICDD card data base.

b. Microstructure Analysis

Evaluation of the microstructure of magnetic materials is accomplished by using scanning electron microscope (SEM) brand JEOL JSM-5310LV type with Au Fine Coater JFC-1200 type and a maximum magnification of 5000 times. Magnetic material to be measured were prepared by metallographic testing process which includes refining the surface and providing the etching solution (3% nital solution) in order to obtain a morphology consisting of grains (grains) and grain boundaries (grain boundary).

c. Thermal Analysis

Thermal analysis was performed to determine the Curie temperature ($T_c$), namely the transition temperature from the ferromagnetic to the paramagnetic phase. Thermal measurements are performed using the differential scanning calorimeter apparatus (DSC, Shimadzu). The magnetic material was prepared at a rate of about 20 milligrams per minute.
and heated from 20 °C to 400 °C maximum temperature, and the value of the Curie temperature (Tc) of the magnetic material is found to be around 310 °C for Nd$_2$Fe$_{14}$B phase.

RESULTS AND DISCUSSION

Preparation of magnetic alloy

One technique employed in the synthesis of metal alloys (metal alloy) with the melting process is the arc melting furnace (AMF) method. AMF has a water cooling system (water cooler) on the control rod (handle) and the sample holder to ensure there is no fusion in that section even though operating at temperatures of up to 4000°C. The air evacuation system in the sample chamber consists of a vacuum pump and argon gas to flush the sample chamber to yield an inert atmosphere condition, in such manner that the melting metal alloys will not undergo any oxidation or decomposition phase.

![Diagram of apparatus for sample preparation using arc melting furnace](image)

Figure 1: Diagram of apparatus for sample preparation using arc melting furnace

The cooling process of the molten metal alloy is very influential on the microstructure and the formation of the desired phase. Results obtained in this study for Nd$_2$Fe$_{14}$B magnetic materials prepared with different cooling processes, followed by slow cooling in the sample chamber, quenching with printing on the mold and finally cooled down from the molten copper alloy magnetic material that flowed. The most important difference is in the formation of the desired magnetic phase and microstructure. Rapid cooling with printing is carried out on cylindrical copper mold.

Phase identification of Nd-Fe-B magnetic phase

The analysis of the phase formation of magnetic materials thus obtained from the AMF method is carried out using the x-ray diffraction analysis. Figure 2 shows x-ray diffraction patterns for an Nd$_{12}$Fe$_{82}$B$_6$ (% atom) ingot cast in cylindrical shape with a diameter of 5 mm, and rapidly quenched to ensure that the cooling phase is maintained in the Nd$_2$Fe$_{14}$B sample. Results of qualitative analysis of x-ray diffraction data on Nd$_{12}$Fe$_{82}$B$_6$ ingot indicate that a single phase has been formed in the phase and that no diffraction intensity for the Nd$_2$Fe$_{14}$B Fe phase (located at the 2θ angle 52°, 80°, and 99°) has been found. This ensures that a magnetic phase formation process has taken place during the rapid cooling process, as expected.
Microstructural investigation of Nd-Fe-B

Microstructure observation by SEM at 1000 times magnification for cylindrical cast Nd$_{12}$Fe$_{82}$B$_6$ ingot samples of 5 mm diameter shows the effects of rapid cooling on the Nd$_2$Fe$_{14}$B phase, with an elongated morphology the size of 10 $\mu$m and limited by porosity (Figure 3). The longer and bulkier grains indicate that the cooling process was running out of control and proceeds at a rapid pace; By casting in the form of cylinders did not only ensure that the Nd$_2$Fe$_{14}$B phase could be maintained, but also that the dendrite structure of the magnetic phase crystals is one of the consequences of the casting and rapid quenching method.
**Thermal Analysis of Nd-Fe-B.**

Curie temperature $T_c$ measurement results, carried out on ingots of Nd-Fe-B using a Differential Scanning Calorimeter (DSC) are presented in figure 4. The Curie temperature $T_c$ was measured for each of the synthesized samples, which were prepared with a different composition and a different casting process. The mass of the measured sample is approximately 20 milligrams and the heating rate is set at 20°C/minute.

![DSC scan of ingot NdFeB casting cylinder shape (diameter 5 mm).](image)

Figure 4: DSC scan of ingot NdFeB casting cylinder shape (diameter 5 mm).

Endothermic peaks show the value of the Curie temperature ($T_c$) and the absence of exothermic peak indicates that there is no oxidation during the measurement. The obtained Curie temperature $T_c$ value is around 314 °C which is shown as the $T_c$ of the hard magnetic phase $\text{Nd}_2\text{Fe}_{14}\text{B}$. This proves that the magnetic material in the form of ingots or after heat treatment has only one single phase namely $\text{Nd}_2\text{Fe}_{14}\text{B}$.

**CONCLUSION**

Results of this study show that the present authors have succeeded in the synthesis of ingot $\text{Nd}_2\text{Fe}_{14}\text{B}$ as hard magnets (hard magnetic) by arc melting furnace method through casting process in a rapid quenching media. Phase identification by x-ray diffraction showed that the hard magnetic phase of $\text{Nd}_2\text{Fe}_{14}\text{B}$ has been successfully grown. SEM microgram of the hard magnetic ingot $\text{Nd}_2\text{Fe}_{14}\text{B}$ microstructure shows that the grains' morphology is not perfect and that the grain size is larger than 10 μm. $T_c$ value of 314 °C shows that the hard magnetic phase of $\text{Nd}_2\text{Fe}_{14}\text{B}$ can be maintained. In this case, $T_c$ was found to be 310 °C.

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**REFERENCES**