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EFFECTS OF CURING TEMPERATURE AND ATMOSPHERE ON THE PROPERTIES OF ISOTROPIC BONDED NdFeB MAGNETS

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ABSTRACT

EFFECTS OF CURING TEMPERATURE AND ATMOSPHERE ON THE PROPERTIES OF ISOTROPIC BONDED NdFeB MAGNETS. The purpose of this paper was to synthesize the isotropic bonded NdFeB magnets at varying curing temperature and atmosphere, and to clarify their properties. For the aforesaid objective, the NdFeB powder was mixed with polyacrylate binder (3wt%) and compacted by hydraulic pressure with a compressive pressure of 30 MPa. The specimens were then separately cured at 100, 150, 180 and 200^{o}C for 1 h in air and vacuum. The effects of curing temperature in air and vacuum on the bulk density and magnetic properties of isotropic bonded NdFeB magnets were studied. The results indicated that the bulk density and flux magnet density of bonded NdFeB magnets decreased with increase in curing temperature. This seems to be due to the binder vaporization and oxidation of element of NdFeB magnet. The bulk density of specimens cured in air was higher than that in vacuum for all temperatures. On the contrary, the magnetic flux density showed the opposite behavior. The optimum magnetic properties were achieved at curing temperature of 100^{o}C for 1 h in vacuum with B_r = 6,61 kG, H_{CJ} = 8,841 kOe, and BH_{max} = 9,01 MGOe.

Keywords: Bonded NdFeB magnet, Curing temperature, Atmosphere, Bulk density, Magnetic properties

ABSTRAK

EFEK TEMPERATUR *CURING* DAN ATMOSFER TERHADAP KARAKTERISTIK *BONDED* MAGNET ISOTROPIK NdFeB. Penelitian ini bertujuan untuk mensintesa *bonded* magnet isotropik NdFeB dengan variasi temperatur *curing*, kondisi atmosfer berbeda dan mengkarakterisasi sifat-sifatnya. Untuk tujuan tersebut, serbuk NdFeB dicampur dengan binder *polyacrylate* (3wt%), dikompaksi dengan tekanan sebesar 30 MPa, di*curing* pada temperatur: 100, 150, 180 dan 200^{o}C selama 1 jam di udara dan vakum, serta diamati pengaruhnya terhadap densitas *bulk* dan *flux magnet density*. Disamping itu juga diukur sifat magnet (B_r, H_{CJ} dan BH_{max}) dan analisa SEM dari *bonded* magnet NdFeB yang di*curing* pada suhu 100 dan 200^{o}C (1 jam) di udara dan kondisi vakum. Hasil penelitian menunjukkan bahwa densitas *bulk* dan *flux magnet density* dari *bonded* magnet NdFeB menurun dengan meningkatnya temperatur *curing*. Hal ini mungkin disebabkan oleh penguapan binder dan oksidasi element penyusun magnet NdFeB. Densitas *bulk* yang di*curing* di udara lebih tinggi daripada vakum, namun sebaliknya nilai *flux magnet density* relatif lebih rendah. Dilihat dari sifat magnetnya, kondisi optimum diperoleh pada suhu curing 100^{o}C (1 jam) dengan kondisi vakum, dan menghasilkan nilai B_r = 6,61 kG, H_{CJ} = 8,841 kOe, dan BH_{max} = 9,01 MGOe.

Kata kunci: *Bonded* magnet NdFeB, Temperatur *curing*, Atmosfer, Densitas *bulk*, Sifat magnet

INTRODUCTION

Currently, bonded Nd-Fe-B magnets are promising material for wide range of applications. It is commonly used in computer storage devices, automobiles, and other family electrical equipments because they offer some advantages such as low cost, high magnetic properties and greater manufacturing flexibility that of being able to be shaped into complex geometry [1]. The performance of bonded magnets relies on the magnetic powders, binders and preparation process [2-5]. The fluidity of the compound and the mechanical properties of the magnets can be enhanced by the appropriate kind of binder. Moreover, polymer binders, if properly selected, can also serve as an insulator to isolate the magnetic particle from exposure to the environment and act as a protection against possible corrosion [6]. The bonded magnets were prepared by mixing a magnetic powder with a polymer binder at an appropriate mixing ratio and then compacted by injection molding or compression molding techniques [3, 7-8]. The compact density was affected by several factors as powder particle size, particle size distribution, loads of compressing pressure, lubricant and compressibility [9, 10]. The typical magnet densities after compaction are 5.8-6.1 g/cm^3 [11]. The samples were then heat treated at a certain temperature. In common, some additives and surface coating are used to improve the corrosion resistance of permanent magnets [12]. The effect of additives on the properties of injection molding magnets is discussed by Wanzhong et al. [7]. It is well known that iron and mainly neodymium in the NdFeB magnets are prone to be oxidized and corroded at elevated temperature [2-3, 13, 14]. This can potentially occur during the powder preparation and process of fabrication which can lead to deterioration of their magnetic properties. Accordingly, the appropriate curing temperature and atmosphere become particularly important to maintain the intrinsic magnetic properties of bonded NdFeB magnets. In this paper we would like to evaluate and discuss the effects of curing temperature and atmosphere (air and vacuum) on the physical and magnetic properties of isotropic bonded NdFeB magnets.

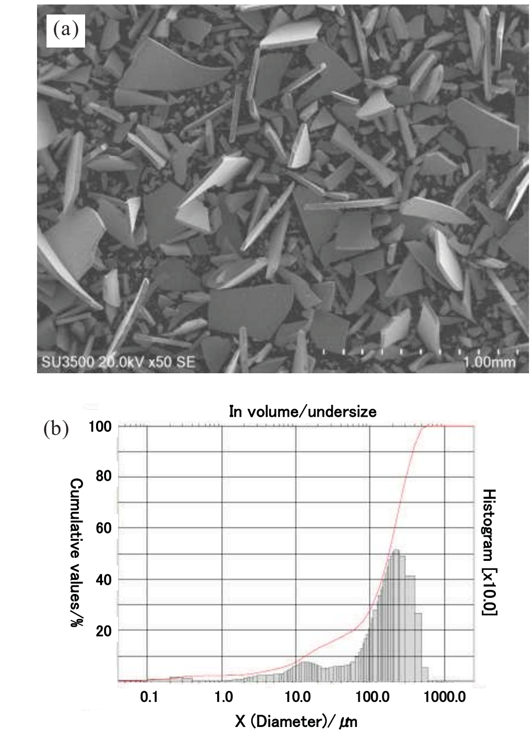

Figure 1. (a). SEM image and (b) particle size distribution of NdFeB powder

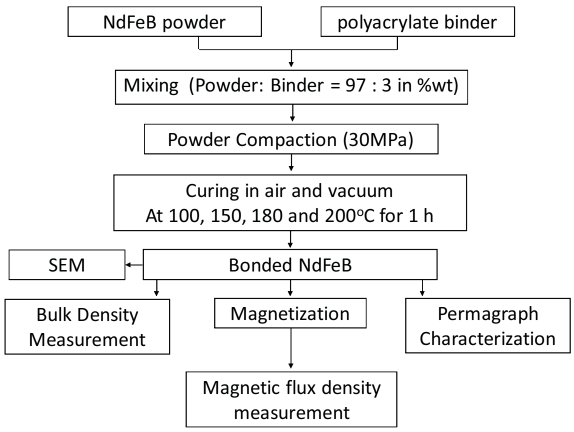

Figure 2. Preparation condition of isotropic bonded NdFeB

EXPERIMENTAL

NdFeB powder (Fig. 1a) from Magnequench Inc. with mean particle size of 179.89 µm (Fig. 1b) was used as a starting material in the present experiment and polyacrilate (WE-518) as the binder.

A detail preparation condition of isotropic bonded NdFeB magnet is shown in Fig. 2.

The plate shape NdFeB powder and binder with the ratio of 97: 3 (in %wt) were mixed and stirred carefully. For each specimen, 3 gram of mixture was placed into disk mold and compressed by hydraulic pressure with a compaction pressure of 30 MPa. This compression molding technique produce the disk pellets with a diameter and thickness of about 11 mm and 5 mm, respectively. The pellets were then heat treated in air and in vacuum separately at elevated temperatures of 100, 150, 180 and 200^{o}C with 1 h in holding time. The samples that have been cured are hereafter referred to as isotropic bonded NdFeB.

The surface morphologies of bonded NdFeB were examined by a scanning electron microscope at accelerating voltage of 20 kV. The bulk density of specimen was determined by using Archimedes principles. Each specimen was magnetized by impulse magnetizer K-series MAGNET-PHYSIK Dr. Steingroever GmbH at voltage of 1300V and current of 4.1A in Research Center for Physics-LIPI. Magnetic characterizations were carried out by Gaussmeter to measure the magnetic flux density and MAGNET-PHYSIK permagraph magnetometer in the Research Center for Electronics and Telecommunications-LIPI to measure the hysteresis curve.

RESULT AND DISCUSSION

Figure 3 shows the bulk density of isotropic bonded NdFeB fabricated at elevated curing temperature of 100, 150, 180 and 200°C in air and vacuum, separately for 1 h.

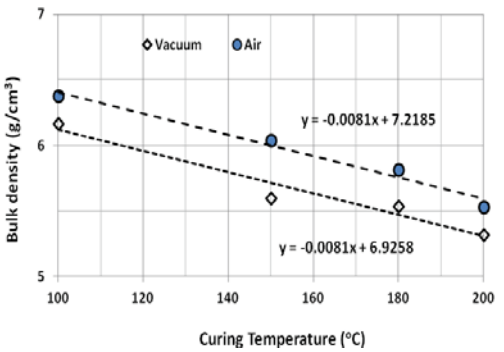

Figure 3. Bulk density of isotropic bonded NdFeB cured in air and vacuum for 1 h at varying temperatures

The graph shows that there is a linear correlation between the bulk density and curing temperature in air and vacuum. The slope of linear line is 0.0081. The bulk density of isotropic bonded NdFeB magnet that is cured in air is a relatively higher by 4.055% compared to the magnet curved in a vacuum.

From both drying conditions, it shows that with increase in curing temperature, the bulk density of samples is likely to decrease. The reason for these is suspected that some amount of binder was vaporized, resulting micropores in the bonded NdFeB. From Figure 3, it can be seen that the optimum condition for fabrication of isotropic bonded NdFeB magnet cured in vacuum or air is 100°C for 1 h.

Figure 4 shows the BSE surface morphologies of isotropic bonded NdFeB cured in vacuum and air at 100 and 200°C for 1 h. From Figure 4, BSE images detect contrast difference of two areas, bright areas and dark areas which show difference in chemical composition. Bright areas indicate the area with heavy elements (high atomic number) of NdFeB. Dark areas show the area with light elements (low atomic number), probably binder and/ or oxides and pores. However, no significant different is found between the samples cured in air and vacuum at 100 and 200°C. The effects of curing temperature and atmosphere on NdFeB microstructure probably clearly visible for longer exposure time (curing time).

Figure 5 shows the magnetic flux density of isotropic bonded NdFeB cured at various temperatures in air and in vacuum for 1 h.

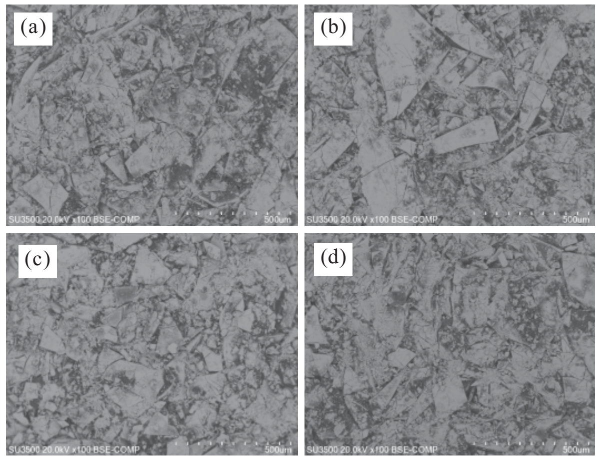

Figure 4. SEM images of isotropic bonded NdFeB cured in vacuum at (a) 100, (b) 200 °C and air at (c) 100, (d) 200 °C

According to the results as shown in Figure 5, the magnetic flux density of isotropic bonded NdFeB cured in both atmospheres decreased with increasing curing temperature. The magnetic flux density of sample cured in vacuum is relatively higher than in air. This could be related to oxidation of NdFeB elements as Fe and Nd which is oxidized easily in air [2, 14]. Air curing appears to accelerate the oxidation of isotropic bonded NdFeB, resulting in decreasing the magnetic flux density of about 0.59 %. Rodrigues et al. [15] reported that high curing temperatures can strongly oxidize Nd, generating more heat and burning may occurs. In this study, the degradation rate of magnetic flux density as function of curing temperature in vacuum and air is $(\delta B/\delta T)_{Vacuum} = 4.47$ and $(\delta B/\delta T)_{Air} = 4.85$, respectively. It is obvious that vacuum curing is relatively better than that of in air.

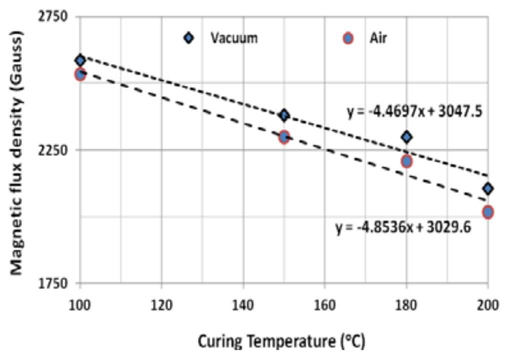

Figure 5. Magnetic flux density of isotropic bonded NdFeB cured in air and vacuum for 1 h at varying temperatures

The hysteresis curves of isotropic bonded NdFeB magnets cured in air and in vacuum at elevated temperatures of 100 and 200°C are shown in Fig. 6.

The summarizing results of remanence B_r, coercivity H_{cj} and maximum energy product BH_{max} of bonded NdFeB magnets are presented in Table 1. It can

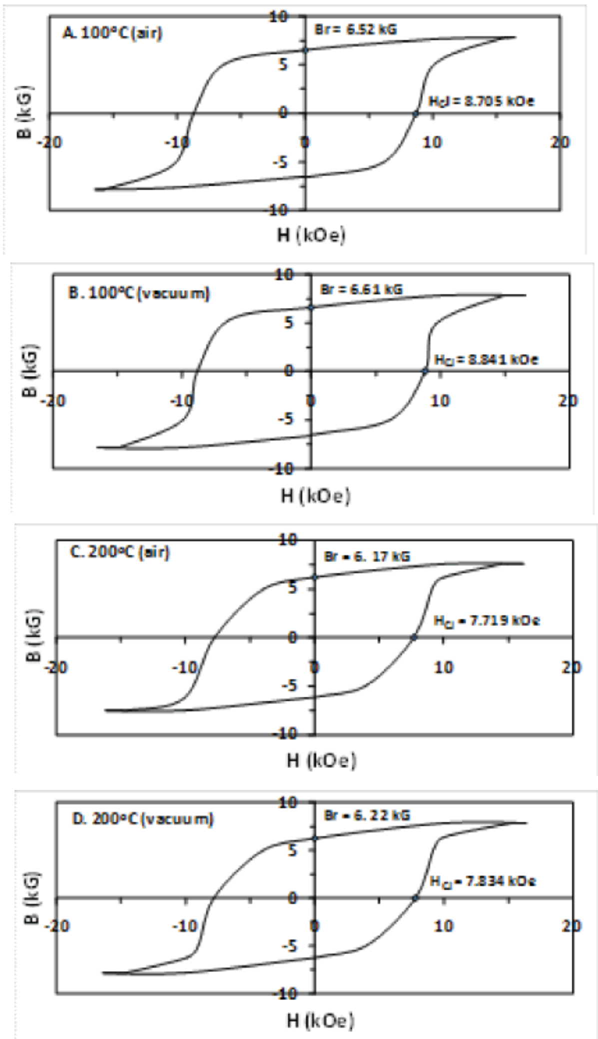

Figure 6. Magnetic hysteresis curve of isotropic bonded NdFeB cured at 100 °C in (a) air, (b) vacuum, and at 200 °C in (c) air, (b) Vacuum

be seen that the results of magnetic flux density measurement of isotropic bonded NdFeB magnets, as presented above are consistent with the results of permagraph characterization as presented in Table 1.

Table 1. The magnetic properties of isotropic bonded NdFeB magnets cured in air and vacuum at 100 and 200°C.

Samples Code	B_r (kG)	H_{CJ} (kOe)	BH_{max} (MGOe)
A: 100°C, 1h, air	6.52	8.705	8.79
B: 100°C, 1h, vacuum	6.61	8.841	9.01
C: 200°C, 1 h, air	6.17	7.719	6.41
D: 200°C, 1h, vacuum	6.22	7.834	6.94

The magnetic properties characterization shows that the remanence B_r, coercivity H_{cj} and maximum energy product BH_{max} of the specimens cured at 100 and 200°C have a tendency to decrease with increase in curing temperature in both atmospheres. The remanence is proportional to sample density [5, 10]. This finding also supports the aforementioned results that the optimum magnetic properties of isotropic bonded NdFeB magnet were obtained at curing temperature of 100°C for 1 h in vacuum.

CONCLUSION

The effects of curing temperature and atmosphere on the properties of isotrophic bonded NdFeB magnets have been studied and discussed. The bulk density and magnetic flux density of isotropic bonded NdFeB magnets decrease with increasing in curing temperature. The bulk density of NdFeB magnets cured in air is relatively higher by 4.055 % compared to the vacuum cured NdFeB magnet. In contrast, the magnetic flux density of bonded NdFeB magnets cured in vacuum is higher compared to in air. As curing temperature increase, the magnetic flux density decrese with degradation rate ($\delta B / \delta T$) of 4.47 for vacuum curing and 4.85 for air curing. This seems to be related to the binder vaporization and oxidation of NdFeB magnet elements. According to the results of magnetic characterization, the optimum magnetic properties of NdFeB magnets were obtained for the specimen that was cured in vacuum at 100°C for 1 h (B_r = 6.61 kG, H_{CJ} = 8.841 kOe dan BH_{max} = 9.01 MGOe). Further work need to be done to establish whether curing temperature (< 100°C) and curing time significantly affect the magnetic properties of bonded NdFeB.

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