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Dynamic mechanical analysis of magnetic rare earth-iron-boron alloys

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Abstract

Dynamic mechanical analysis of magnetic alloys based on the compositions $Nd_{16}Fe_{76}B_8$ and $Nd_{11.8}Fe_{82.4}B_{5.8}$ was carried out. It has been shown that the elastic modulus increased slightly (4–6%) and irreversibly, when the alloys were heated to 400°C and cooled to room temperature. This effect has been found to be detrimental to the intrinsic coercivity of a sintered permanent magnet. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Permanent magnets; Alloys; Mechanical properties

1. Introduction

The effect of temperature variations on the Young's modulus (E) of Nd–Fe–B permanent magnets has been studied using ultrasonic resonance [1]. A sharp decrease in elastic moduli at about 600°C was observed and attributed to the appearance of a liquid phase at the grain boundaries. Recently, it was reported that annealing a Nd–Fe–B magnet at temperatures below the Curie temperature (T_c), caused precipitation of excess neodymium from the lattice of the $Nd_2Fe_{14}B$ matrix phase (ϕ) [2]. It has also been reported that annealing at temperatures higher than T_c is accompanied by changes in stress from compressive to tensile and as a result, dissolution of neodymium in the ϕ-phase lattice. This paper presents data from an investigation in which a dynamic mechanical analyzer was used to study Nd based alloys at around the Curie temperature. A well-known Nd-alloy with composition $Nd_{16}Fe_{76}B_8$ ("Neomax") was selected, along with a stoichiometric alloy, $Nd_{11.8}Fe_{82.4}B_{5.8}$ ($Nd_2Fe_{14}B$) for comparison, in this investigation.

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2. Experimental

Two commercial alloys in the as-cast state were studied. The DMA specimens (single cantilever bending type) were approximately $25 \times 5 \times 2\,mm^3$. Elastic modulus measurements were made in a dynamic mechanical analyzer (Netzsch DMA 242) at an oscillation frequency of 1 Hz. The oscillation amplitude was limited to 15 μm and a heating rate of $2°C\,min^{-1}$ was used (heated in air). The temperature interval used was from room temperature to a maximum of 400°C. The procedure to prepare the sintered magnet using the hydrogen decrepitation process (HD) can be found elsewhere [3,4].

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3. Results and discussion

Fig. 1 shows the variation of elastic modulus with temperature for the $Nd_{16}Fe_{76}B_8$ alloy. The elastic modulus decreases gradually with increase in temperature up to 290°C and then increases significantly as the temperature approaches 400°C. During cooling, the elastic modulus shows a minimum once again at 290°C and is higher by 4% at room temperature.

Fig. 2 shows the variation of elastic modulus with temperature for the stoichiometric Nd–Fe–B alloy. Similar to the other alloy, there is a steady decrease in elastic modulus to a minimum at around 290°C, followed by an increase. A similar pattern was observed upon cooling, with a final increase in the elastic modulus of about 6% at room temperature.

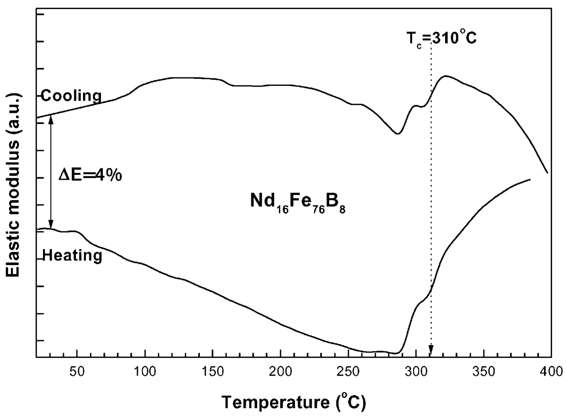

Fig. 1. Variation of elastic modulus with temperature during heating and cooling for a Nd-rich alloy.

Comparison of these two alloys shows that during heating, the alloy with higher Nd content exhibits a less pronounced decrease in E at temperatures below T_c. Beyond T_c, both alloys reveal an increase in the elastic modulus up to 400°C but that for the Nd-rich alloy, is substantially larger. Upon cooling the stoichiometric alloy, a pattern similar to that during heating is observed. The Nd-rich alloy, in contrast, shows a deviation from the heating pattern for temperatures below 200°C, revealing a plateau and a decrease in the elastic modulus.

Fig. 3 shows the variation of elastic modulus with temperature for the $Nd_{16}Fe_{76}B_8$ alloy that had been isothermally heat treated at 350°C for 5 h, both on heating up to this temperature and on cooling back to room temperature. This heat treatment brings about a further irreversible increase in elastic modulus. Upon cooling to room temperature, a 5.5% increase in elastic modulus is observed, which is slightly larger than that exhibited by this alloy in the absence of an isothermal hold at 350°C (4%). The variation of elastic modulus with temperature for the stoichiometric alloy isothermally heat treated at 350°C for 5 h is shown in Fig. 4. Once again, for this alloy, a similar behavior is observed, both upon heating

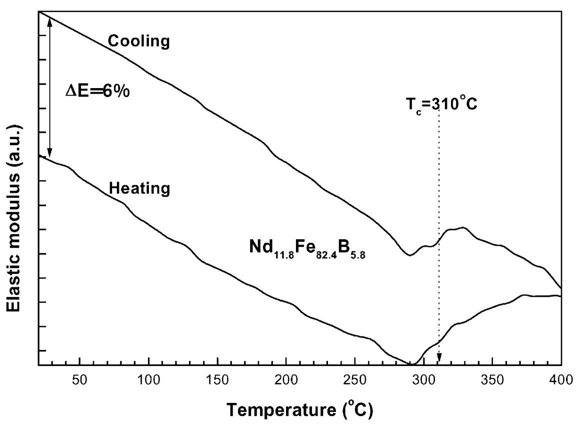

Fig. 2. Variation of elastic modulus with temperature during heating and cooling for a stoichiometric alloy.

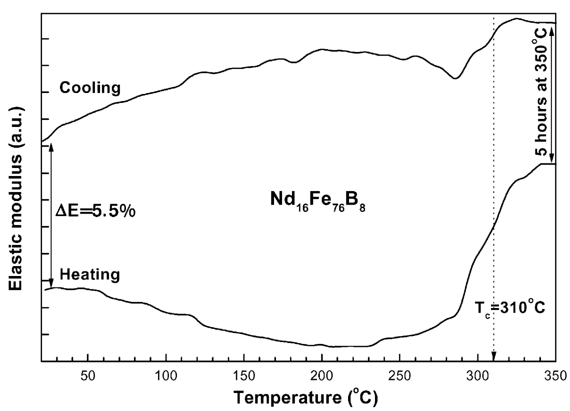

Fig. 3. Elastic modulus versus temperature for a Nd-rich alloy with an isothermal treatment at 350°C for 5 h.

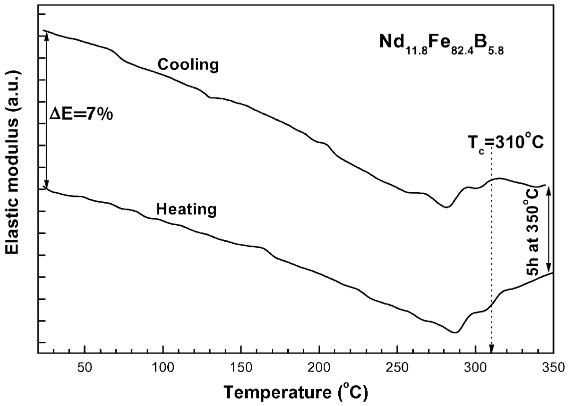

Fig. 4. Elastic modulus versus temperature for a stoichiometric alloy with an isothermal treatment at 350°C for 5 h.

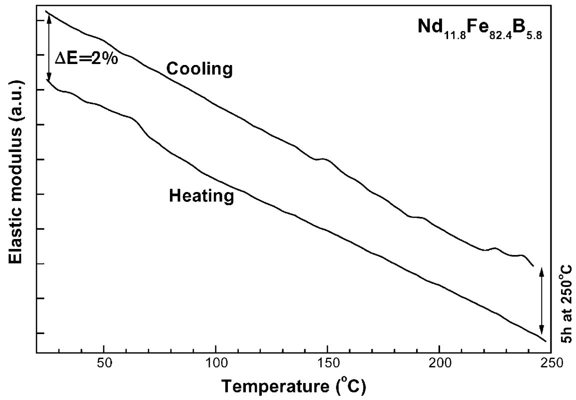

Fig. 6. Elastic modulus versus temperature for a stoichiometric alloy with an isothermal treatment at 250°C for 5 h.

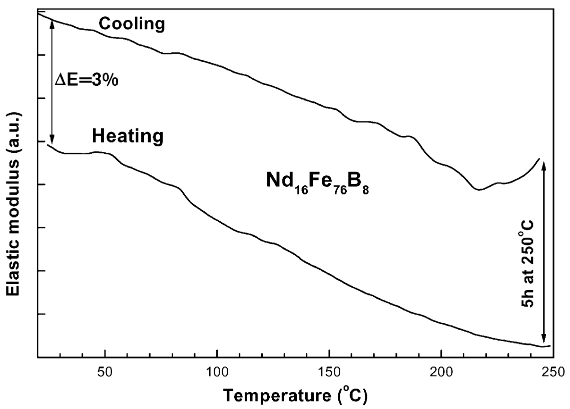

Fig. 5. Elastic modulus versus temperature for a Nd-rich alloy with an isothermal treatment at 250°C for 5 h.

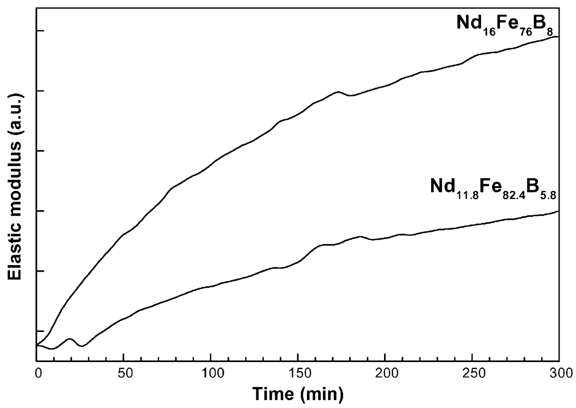

Fig. 7. Variation of elastic modulus with time at a constant temperature of 250°C for 5 h for both alloys.

and cooling, and with an irreversible increase in the elastic modulus of about 7% at room temperature.

Figs. 5 and 6 show the variation of elastic modulus with temperature on heating and cooling for both samples of both alloys that were isothermally treated for 5 h at 250°C. For this lower temperature treatment, the elastic modulus changes in a similar manner, upon cooling and heating, for both alloys. Surprisingly, in this case, the Nd-rich alloy exhibits a slightly larger increase in elastic modulus at room temperature, after heating and cooling than the stoichiometric alloy. The variation of the elastic modulus with time during isothermal heat treatments at 250°C and 350°C, for both alloys, are shown in Figs. 7 and 8, respectively.

The decreasing monotonic behavior of E, commonly found upon heating a metal (see, for example [5]), is due to expansion of the crystal lattice. During an isothermal heat treatment, the elastic modulus should normally remain constant. The elastic modulus of the Nd–Fe–B alloys studied here increased during this treatment and the change was more pronounced in the alloy with higher Nd-content. Other factors that could affect the behavior of E in these alloys include: (1) relief of residual stresses in rare-earth alloys [6]; (2)

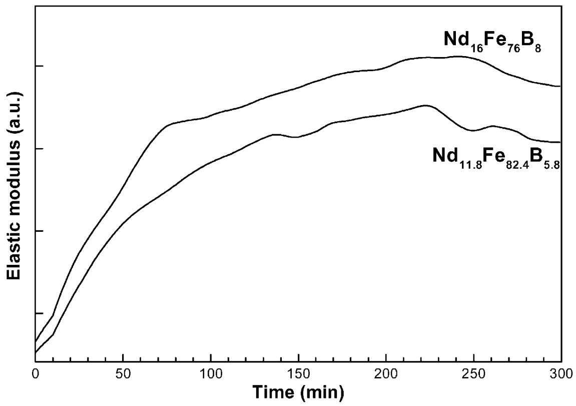

Fig. 8. Variation of elastic modulus with time at a constant temperature of 350°C for 5 h for both alloys.

Table 1
Magnetic properties of HD sintered $Nd_{16}Fe_{76}B_8$ magnet before and after a low temperature heat treatment (under vacuum)

Condition	Br (mT)	iHc ($kA\,m^{-1}$)
Vacuum sintered at 1060°C for 1 h, annealed at 650°C for 1 h and fast cooled to room temperature.	1180±5	912±5
Vacuum sintered at 1060°C for 1 h, annealed at 650°C for 1 h, treated at 350°C for 5 h and fast (or slow) cooled to room temperature.	1180±5	848±5

precipitation of excess neodymium from the lattice of the $Nd_2Fe_{14}B$ matrix phase or dissolution of neodymium in the ϕ-phase lattice [2]; (3) internal friction anomalies in ferromagnetic material near the Curie point [7]; (4) and oxidation of the rare-earth alloy [8]. At this stage the reasons for the change in behaviour of E of both alloys is still unclear. Microstructural studies are in progress in an attempt to help explain the changes in elastic modulus observed in this investigation.

Table 1 shows the magnetic properties of a sintered $Nd_{16}Fe_{76}B_8$ magnet before and after heat treatment at 350°C for 5 h. A decrease in the intrinsic coercivity can be observed after this low temperature heat treatment. This heat treatment at 350°C is therefore detrimental to the coercivity of a sintered Nd-rich alloy magnet. Consequently, the standard post-sintering heat treatment at 630°C for 1 h [9], should be followed by rapid cooling to room temperature.

4. Conclusions

The elastic modulus increases with increasing temperature for $Nd_{16}Fe_{76}B_8$ and $Nd_{11.8}Fe_{82.4}B_{5.8}$ alloys. Up to about 260°C, there is an initial decrease in the elastic modulus brought about by crystal lattice expansion. Above this temperature, the increase becomes predominant for both alloys. This increase in the elastic modulus is more significant for the $Nd_{16}Fe_{76}B_8$ alloy.

Acknowledgements

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