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Magnetic and Magnetostrictive Properties of Slow-Cooled Boron-Added Terfenol-D Alloy

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ABSTRACT: This paper reports the influence of controlled heat treatment on the magnetic and magnetostrictive behaviour of polycrystalline Terfenol-D alloy with 2 at.% boron addition. Samples were annealed at 1000 °C for 72 hours with regulated heating, then cooled at a rate of 0.5 °C/min to room temperature. X-ray diffraction analysis confirmed no major structural changes post-heat treatment; however, Rietveld refinement revealed a substantial reduction in the weight fraction of the secondary (Tb,Dy)Fe₃ phase—from 38.25% to 5.09%—accompanied by an increase in the primary (Tb,Dy)Fe₂ phase. Despite this, a slight decrease in total magnetostriction was observed, from 1041 to 1016 microstrain under an applied magnetic field of 12.2 KOe. Magnetisation measurements showed an increase in saturation magnetisation and a marked reduction in coercivity, from 108.5 Oe to 24.2 Oe, after annealing. The magnetic hysteresis loops of the annealed samples exhibited overlapping and twisted features, possibly arising from altered magnetic phase responses due to the applied thermal treatment.

KEYWORDS: Terfenol-D alloy, Boron addition, Slow cooling, Magnetostriction, Magnetisation

I. INTRODUCTION

Rare-earth-based intermetallic compounds, particularly those crystallizing in the cubic Laves phase structure, have attracted considerable interest due to their promising magnetostrictive properties. Among these, Terfenol-D (Tb_{0.27}Dy_{0.73}Fe₂) stands out as a leading giant magnetostrictive material, extensively studied for its potential applications in actuators, sensors, and transducers. The magnetostriction (MS) in Terfenol-D primarily originates from the 4f–3d exchange interaction between rare-earth and transition metal ions, resulting in substantial magnetostrictive strain under moderate magnetic fields [1,2]. However, despite its high MS capability, the presence of secondary phases and microstructural inhomogeneities adversely affects the magnetostrictive response.

Among various approaches to enhance its properties, partial substitution with boron has been reported to significantly affect the magnetic and magnetostrictive behaviour of Terfenol-D alloys [4–7]. As a small interstitial element, boron can alter phase stability and grain boundary characteristics, thereby influencing domain wall motion and overall magnetic response.

In our previous work, we investigated the effect of boron addition (0–6 at.%) on the structural, magnetic, and magnetostrictive properties of polycrystalline Terfenol-D [6]. The alloy with 2 at.% boron addition (labelled T2) showed a notable reduction in the magnetically inactive RFe₃ phase (R = Tb,Dy). However, this reduction was accompanied by a decrease in magnetostriction compared to the as-cast parent sample. Further increase in boron content beyond 2 at.% resulted in a substantial decrease in MS value. Consequently, the T2 composition was selected for further investigation, with particular emphasis on the effect of thermal treatment.

The present study aims to explore the influence of slow cooling following high-temperature annealing on the structural, magnetic, and magnetostrictive properties of the 2 at.% boron-added Terfenol-D alloy. A comparative analysis of room-temperature magnetic and magnetostrictive behaviour between the as-cast and slow-cooled samples is presented.

II. EXPERIMENTAL DETAILS

Polycrystalline boron-added ($B = 2$ at.%) Terfenol-D ($Tb_{0.27}Dy_{0.73}Fe_2$) alloy was synthesised as ingot by induction melting. The as-cast alloy is labelled as T2. A portion of the T2 ingot was annealed at $1000^\circ C$ for 72 hours with controlled heating and cooling rates. Heating was carried out at $5^\circ C/min$ up to $1000^\circ C$, while cooling was regulated at $0.5^\circ C/min$ down to room temperature. This sample is referred to as T2_1000_SC.

For X-ray diffraction (XRD) measurements, the samples were powdered and analysed using a PANalytical X'Pert PRO diffractometer with $Cu-K\alpha$ radiation. The lattice parameters and phase content were quantitatively evaluated by Rietveld refinement using FullProf Suite.

For magnetostriction measurements, samples were cut into slices of $4\text{ mm} \times 6\text{ mm} \times 1\text{ mm}$. An indigenously designed and developed four-probe-based strain measurement system was used to measure room-temperature magnetostriction [12]. Measurements were conducted in an applied magnetic field ranging from 0 to 12.2 KOe. Both longitudinal ($\lambda_{||}$) and perpendicular (λ_{\perp}) strains were recorded, and the total magnetostriction (λ_s) was computed as $(\lambda_{||} - \lambda_{\perp})$.

Magnetisation measurements were performed using a vibrating sample magnetometer (VSM) at room temperature for both the samples.

III. RESULTS AND DISCUSSION

a) XRD Measurements

The X-ray diffraction (XRD) patterns of the as-cast (T2) and heat-treated (T2_1000_SC) samples are shown in Figures 1(a) and 1(b), respectively. Both samples exhibit the presence of two major phases: the cubic Laves phase of $MgCu_2$ -type structure, corresponding to the $(Tb,Dy)Fe_2$ phase, and a secondary $(Tb,Dy)Fe_3$ phase. The observed peaks have been indexed using standard crystallographic data from ICDD: 04-006-3176 for RFe_2 and ICDD: 04-001-0690 for RFe_3 ($R = Tb, Dy$). A minor fraction of an additional (Tb,Dy) -rich solid solution appearing in the samples is indicated as the 'other phase'. As seen from the XRD patterns, no significant structural change is observed in the alloy as a result of the applied heat treatment.

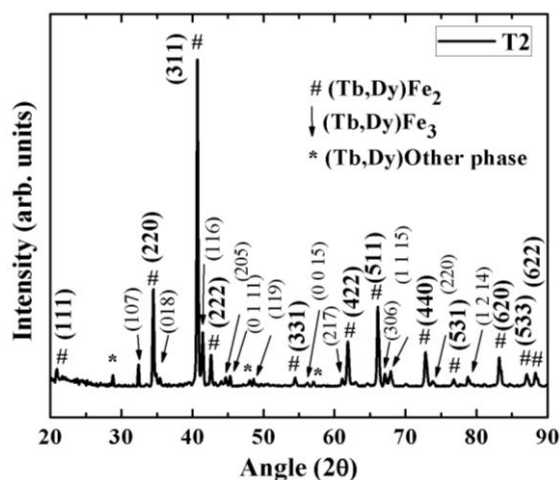


Figure 1(a): XRD of as-cast B-added ($B = 2$ at.%) Terfenol-D alloy.

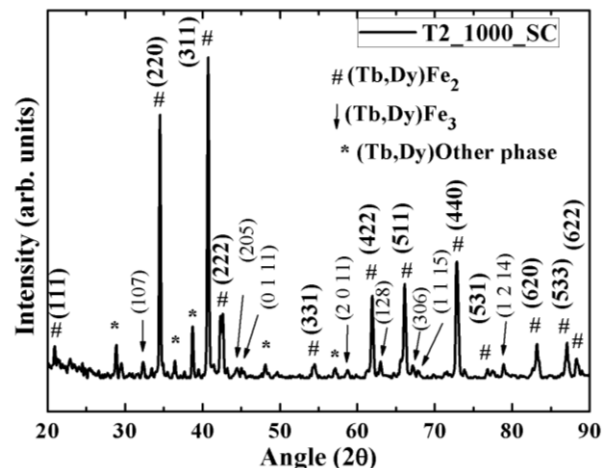


Figure 1(b): XRD of B-added ($B = 2$ at.%) Terfenol-D alloy annealed at $1000^\circ C$ and slow-cooled.

Quantitative phase analysis of the samples was carried out using Rietveld refinement through the FullProf Suite. The crystal structure of the (Tb,Dy)Fe₂ phase was refined using cubic symmetry, and that of the (Tb,Dy)Fe₃ phase with rhombohedral symmetry. The refinement plots for both the samples are shown in Figures 2(a) and 2(b). The observed data (red), calculated data (black), difference plot (blue), and Bragg reflection positions (green ticks) are presented.

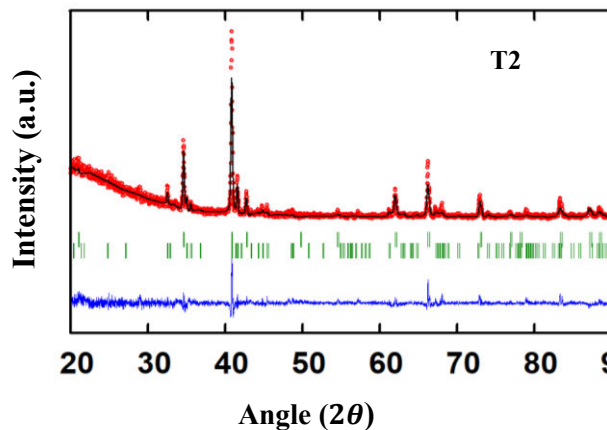


Figure 2(a): Rietveld refinement plot of as-cast B-added (B = 2at.%) Terfenol-D alloy.

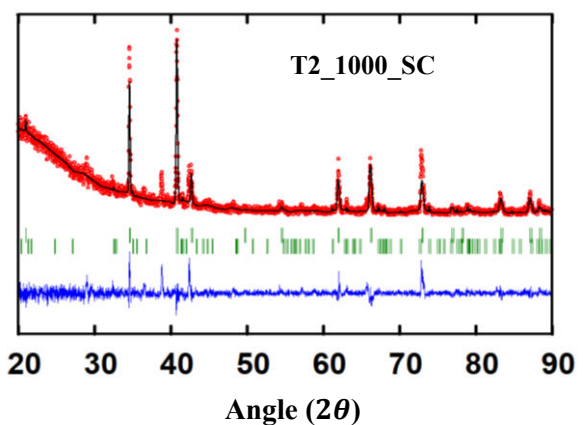


Figure 2(b): Rietveld refinement plot of B-added (B = 2at.%) Terfenol-D alloy annealed at 1000°C and slow-cooled.

The obtained refinement parameters, including lattice constants, phase fractions, and goodness-of-fit values, are summarised in Table 1. The as-cast sample contains 61.75 wt.% of the primary (Tb,Dy)Fe₂ phase and 38.25 wt.% of the RFe₃ phase, while the heat-treated sample shows a significant decrease in the secondary phase content, with 94.91 wt.% of (Tb,Dy)Fe₂ and 5.09 wt.% of (Tb,Dy)Fe₃ phase.

Table 1: Lattice parameters of T2 alloy and weight percent of RFe₂ and RFe₃ phase estimated from Rietveld refinement.

Sample	Lattice parameter of (Tb,Dy)Fe ₂ phase	% Weight Fraction for (Tb,Dy)Fe ₂ phase	% Weight Fraction for (Tb,Dy)Fe ₃ phase	Chi ² value
T2	7.327Å	61.75 (1.63)	38.25 (1.44)	2.28
T2_1000_SC	7.332Å	94.91 (3.67)	5.09 (0.69)	2.42

b) Magnetostriction Measurements

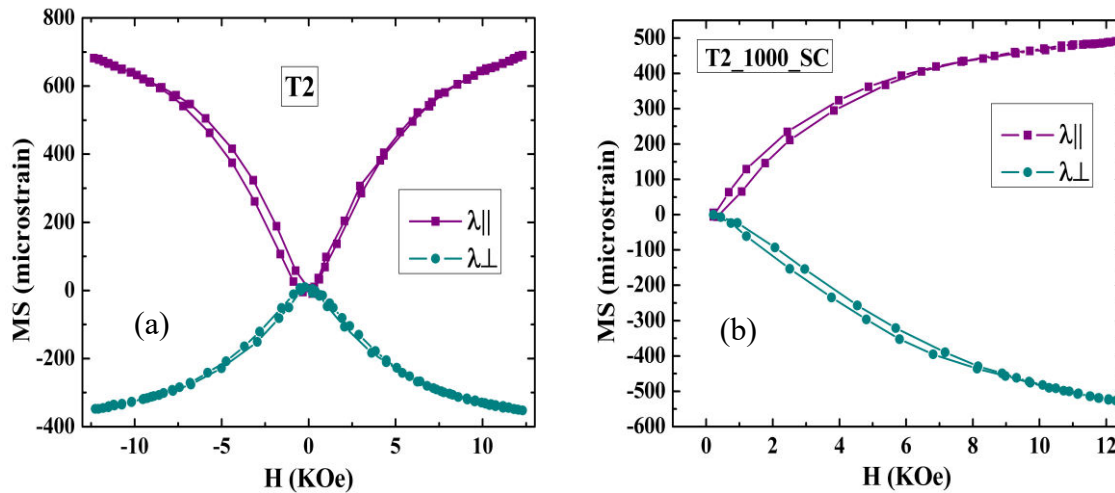


Figure 3: Room temperature MS values of (a) T2 and (b) T2_1000_SC alloy in parallel and perpendicular directions.

Figure 3(a) shows the room-temperature longitudinal and transverse magnetostriction values of the T2 alloy measured in microstrain. The longitudinal strain is observed to be positive, while the transverse strain is negative. The total MS strain (λ_s) reaches a value of 1041 microstrain in the available field of 12.2 KOe. The corresponding MS data for the heat-treated sample (T2_1000_SC) is shown in Figure 3(b). A small reduction in total strain is observed, with λ_s decreasing to 1016 microstrain at the same applied field.

c) Magnetisation Measurements

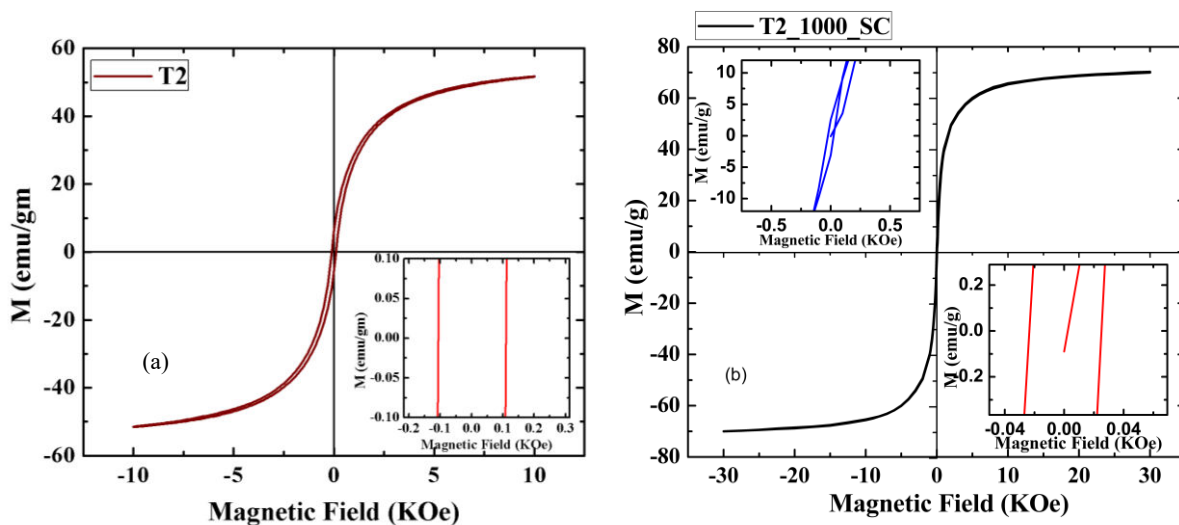


Figure 4: Room temperature M-H curves of (a) T2 and (b) T2_1000_SC samples.

Magnetisation (M-H) measurements at room temperature are shown in Figures 4(a) and 4(b) for the T2 and T2_1000_SC samples, respectively. Both samples exhibit soft ferromagnetic behaviour. The saturation magnetisation (M_s) of the as-cast sample is 56.5 emu/g, while the heat-treated sample shows an increase to 72.3 emu/g. The coercive field (H_c) of the

as-cast sample is 108.5 Oe. For the heat-treated sample, however, the hysteresis curve shows overlapping and twisting, as shown in the inset of Figure 4(b), with a measured coercivity of 24.2 Oe. This behaviour is likely attributed to altered magnetic domain dynamics due to coexisting and competing phases formed during annealing.

IV. CONCLUSIONS

Polycrystalline boron-added ($B = 2$ at.%) Terfenol-D ($Tb_{0.27}Dy_{0.73}Fe_2$) alloy was annealed at 1000 °C for 72 hours, followed by slow cooling to room temperature at 0.5 °C/min, to investigate the effect of heat treatment on their structural, magnetostrictive, and magnetic properties. XRD analysis confirmed the retention of the primary cubic Laves phase (RFe_2), with a significant reduction in the secondary RFe_3 phase from 38.25 wt.% in the as-cast alloy to 5.09 wt.% post-annealing. A slight increase in the lattice parameter of the $(Tb,Dy)Fe_2$ phase was also observed.

Magnetostriction measurements showed a minor decrease in the total strain from 1041 microstrain (as-cast) to 1016 microstrain (annealed), despite the notable reduction in the RFe_3 phase—commonly regarded as detrimental to magnetostrictive behaviour. Magnetic characterisation revealed an enhancement in saturation magnetisation (from 56.5 emu/g to 72.3 emu/g) and a sharp drop in coercivity (from 108.5 Oe to 24.2 Oe), indicating improved magnetic softness. The presence of overlapping hysteresis loops suggests a complex interaction between coexisting phases.

These findings indicate that heat treatment with slow cooling after annealing for polycrystalline boron-added ($B = 2$ at.%) Terfenol-D ($Tb_{0.27}Dy_{0.73}Fe_2$) alloy effectively alters the phase distribution and enhances magnetic performance. However, it does not lead to a corresponding improvement in magnetostriction, possibly due to subtle changes in domain dynamics or residual strain effects.

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