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# Effect of heat treatment on the magnetostriction and microstructure of Terfenol-D

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**Abstract**

Grain aligned rods of the highly magnetostrictive Terfenol compound ($Dy_{0.73}Tb_{0.27}Fe_{1.9}$) prepared by vertical float zoning have been heat treated in order to study the influence on the magnetostriction and microstructure. A significant improvement (10–20%) in the magnetomechanical coupling coefficient was observed on samples treated at 1000 °C for 24 h; further enhancement could be obtained by increasing the treatment time to 7–10 days. A $k_{33}$ value of 0.82 has been recorded on a rod treated in this way. The associated microstructural changes involve a re-distribution of the RE-rich phase, in particular a re-absorption of the intra-grain species into the matrix, thus removing obstacles to domain wall motion.

## 1. Introduction

The giant magnetostrictive rare earth-iron alloys based on the Laves phase $RFe_2$ compound are currently of technological interest in various applications such as transducers, actuators and sensors. Terfenol-D ($Dy_{0.73}Tb_{0.27}Fe_{2-x}$) is the most widely studied material in this context, since it combines a low magnetic anisotropy with a high magnetostriction in the ⟨1 1 1⟩ direction [1]. To realise the maximum strain, crystallographic alignment of the material is required for device quality material. This is usually achieved by directional solidification using the vertical float zoning technique [2]. However, the magnetostrictive properties are dependent on the detailed microstructure of the material, which is determined by the starting composition [3] and the precise processing procedures. Precipitates, inclusions and low angle boundaries and other defects are all thought to hinder domain wall movement and prevent the development of the optimum strains in the material [4,5]. The use of RE-rich initial compositions avoids the formation of unwanted $RFe_3$ precipitates [6–8], and provides a ductile network of grain boundary phase which enhances the mechanical stability of device components. Several studies [9–11] have been performed to correlate the processing variables with the magnetostrictive parameters such as strain and the magnetomechanical coupling coefficient $k_{33}$, and some also on the prevailing microstructures and associated magnetization processes [12–14], with the result that material with magnetostrains of approximately 1500 ppm and $k_{33}$ between 0.6 and 0.7 can be produced. However, problems of property reproducibility and material homogeneity still remain, which require further metallurgical investigation. In a recent study, the effect of heat treatment on the magnetostrictive properties of single crystals, float zoned rods and directionally solidified polycrystalline material have been reported [15]. The magnetostrictive parameters, maximum strain ($\lambda$) and maximum slope ($d\lambda/dH$) were found to be improved by a 1-h heat treatment at 950 °C. In this paper, we describe the effect of heat treatment on the magnetomechanical coupling factor and corresponding microstructure of Terfenol rods produced by float zoning, and report the highest value of $k_{33}$ so far recorded.

## 2. Experimental details

Alloys with composition $Dy_{0.73}Tb_{0.27}Fe_{1.9}$ were prepared by arc melting from high purity start materials (supplied by REP). These ingots were then re-melted into 130 mm long by 8 mm diameter rods by cold boat induction melting. The free standing vertical float zoning was carried out by driving the rod sample downwards through a one turn RF concentrator at the rate of 138 mm/h in an atmosphere of 700 mbar of helium. The as-zoned rods were heat treated under 1 atm purified argon. The magnetomechanical coupling coefficient was measured by the resonance technique on cylindrical rods cut and ground to approximately 50 mm in length and 5 mm diameter. Microstructural obser-

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vation of cross-sections of the rods was performed by optical microscopy.

The experimental procedure involved a sequential heat treatment and measurement followed by the same sequence at a higher temperature on the same rod. Thus, the effects of annealing were essentially cumulative.

## 3. Results and discussion

The coupling coefficients of several directionally solidified rods were measured and the values ranged from 0.55 to 0.65. A typical plot of $k_{33}$ versus bias field is shown in Fig. 1. The field at which the maximum coupling was achieved varied from sample to sample in the range 25–35 kA/m. Heat treatment for 1 day at temperatures up to 850 °C had little measurable effect on the value of $k_{33}$, or its field dependence. At 900 °C and above, significant changes were observed. Both the maximum $k_{33}$ values and the field at which it was realised were affected. The maximum $k_{33}$ increased with temperature, reaching a maximum at 1000 °C, and decreasing slightly at higher temperature; the field at which the $k_{33}$ was maximised decreased significantly with increasing temperature, with a corresponding increase in the rate of increase of $k_{33}$ with field, as seen in Fig. 1. The maximum values of $k_{33}$ for three similar rods are plotted as a function of heat treatment temperature in Fig. 2. All exhibit the same trend which indicates an optimum annealing temperature in the region of 1000 °C. All the rods so far treated show a similar increase, with enhancement of $k_{33}$ varying between 10 and 20%. These results are consistent with those observed previously [15], where $\lambda$ and $d\lambda/dH$ were optimised between 950 °C and 1000 °C, with no change below 850 °C. An exactly similar trend has also been observed in rods prepared in the $Tb_{1-x}Ho_xFe_2$ system [16].

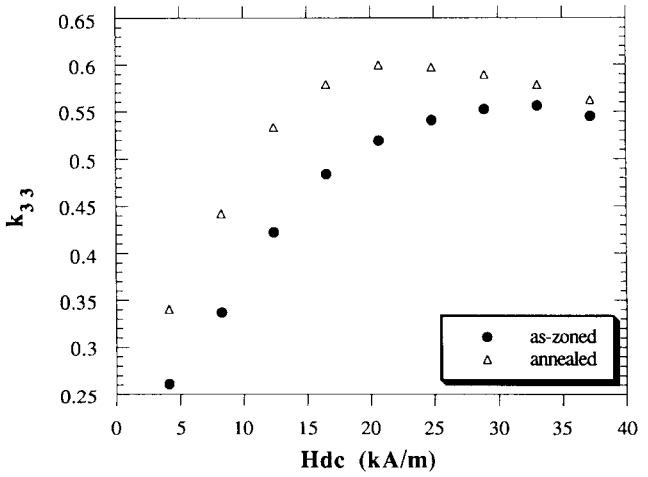


Fig. 1. Magnetomechanical coupling factor $k_{33}$ as a function of bias field for a typical Terfenol rod in the as-zoned and heat treated states.

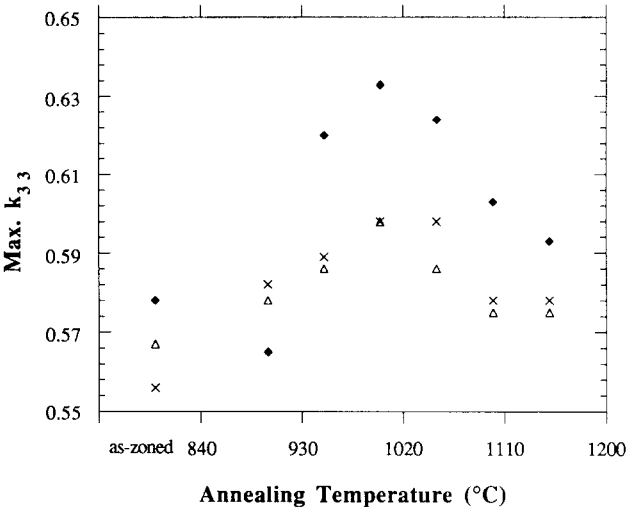


Fig. 2. Maximum $k_{33}$ as a function of heat treatment temperature for three Terfenol rods prepared under the same conditions.

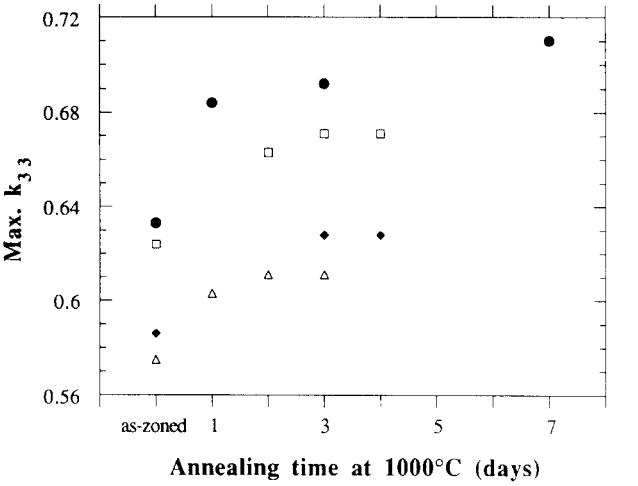


Fig. 3. Maximum $k_{33}$ as a function of heat treatment time at 1000 °C for four similar rods.

Having optimised the heat treatment regime with respect to temperature, several rods of the same composition were subjected to treatment at 1000 °C for different lengths of time, again on a sequential basis for 1–4 days. The results are shown in Fig. 3. Rods with varying as-zoned values all showed the expected increases after 1 day at 1000 °C, and subsequent anneals for longer periods produced further enhancement up to 4 days (10 days cumulative). No further increase was observed for longer periods, and in some cases some deterioration occurred, presumably related to oxidation during extended treatment. This behaviour contrasts with the previous study [15], where no significant change in $\lambda$ and $d\lambda/dH$ was found by varying the time from 1 h to 4 days.

Thus, the optimum heat treatment, based on these observations would appear to be at 1000 °C for 7 days. Whether the treatment is performed as a one step operation, or as a cumulative exercise as described here, is still being investigated, but the preliminary results indicate that thermal cycling of the type reported here may in fact be beneficial to the generation of improved magnetostrictive behaviour. However, the highest $k_{33}$ value we have recorded was obtained on a specimen which was heat treated in one step at 1000 °C for 7 days; this yielded a value of 0.82, which we believe to be the highest reported value for this material.

The microstructural changes associated with this heat treatment were observed by optical microscopy on longitudinal sections cut from a rod which was subjected to the same sequence of events as the specimens in Fig. 2. The micrographs taken from rods heated at different temperatures are shown in Fig. 4. The as-zoned microstructure reveals the typical long plate-like grains running parallel to the zoning direction with the fine network of RE-rich phase delineating the grain boundaries, but also occurring as an intragranular feature. No significant change in microstructure was observed below 900 °C, in agreement with the measured magnetostrictive properties. Above this temperature the RE-rich phase begins to be re-absorbed into the matrix [17], and what does remain agglomerates at grain boundary triple points. At 1000 °C, most of the re-distribution of this phase is complete, and at higher temperatures the most significant development is the occurrence of microcracking.

This re-distribution of the RE-rich phase at elevated temperature is consistently observed in Terfenol material, whether it be in as-cast material [7,8,17], sintered powders [18], zoned rods [11] or even in crystal boules prepared by the Czochralski or Bridgman techniques [2]. Its presence, like that of $RFe_3$ Widmanstatten precipitates, will hinder domain wall movement, particularly where it occurs within grains, and its reabsorption into the matrix clearly is beneficial to the magnetostrictive properties[15]. The associated removal of strain and other defects such as stacking faults and dislocation networks [19] also leads to an improvement, particularly in the required rapid response to ac fields. The occurrence of microcracking at higher temperatures is probably responsible for the observed decrease in magnetostrictive properties above 1000 °C.

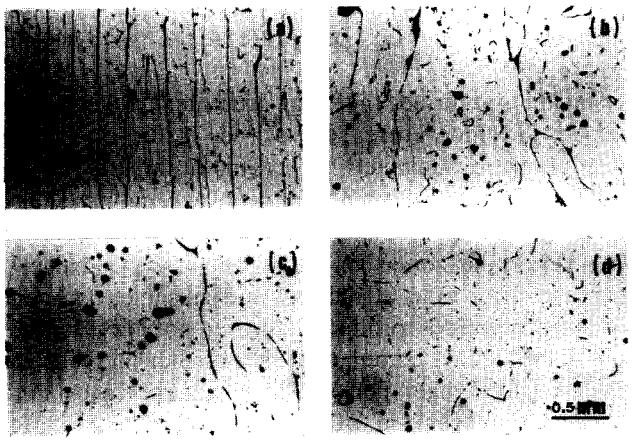


Fig. 4. Optical micrographs of longitudinal sections of rods heat treated at different temperatures according to the sequence depicted in Fig. 2, (a) as-zoned, (b) 950 °C, (c) 1000 °C and (d) 1050 °C.

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