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The effect of magnetically aligned powder on the magnetostriction of sintered rare earth-iron Laves phase compounds

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Abstract

A powder metallurgical approach is utilized to prepare grain oriented Laves phase compounds of $\text{Tb}_x\text{Dy}_{1-x}\text{Fe}_2$. The magnetostrains observed in the oriented compounds, though containing ~20% porosity as presently prepared, are far superior to those of arc cast and highly dense liquid phase sintered materials. Also, it is shown that the alignment achieved is strongly dependent on the Tb/Dy ratio.
Recently, a number of studies have been carried out on the magnetic properties of rare earth-iron Laves phase compounds. Of particular interest are the high room-temperature magnetostrains observed in several of these compounds which make them promising for a variety of technological applications. However, since these materials are intermetallic compounds, they do not lend themselves to conventional fabrication processes such as machining or casting. Recently, a power metallurgical approach has been used successfully in this laboratory to obtain these Laves compounds in suitable sizes and shapes. Their magnetostrictive properties have been reported elsewhere.

In this study, it is shown that the magnetostrictive properties can be greatly enhanced by magnetically aligning the power particles prior to sintering. For this study, the pseudobinary system \((\text{Tb}_x\text{Dy}_{1-x}\text{Fe}_2)\) with \(x\) close to 0.3 was selected because this composition is particularly well suited for a wide range of device applications. At this composition, a relatively large magnetostriction is associated with minimal magnetic anisotropy. Spin orientation diagrams show that this compound, at room temperature, has its axis of easy magnetization along the major cubic symmetry \([111]\) direction. A power metallurgical approach can utilize the direction of easy magnetization by alignment of the power compound in a magnetic field.
The compound preparation consisted of arc melting elemental rare earth metals and iron, all of 99.9% purity, on a water cooled copper hearth under a Zr gettered argon atmosphere. After a homogenizing anneal at 1000°C in evacuated quartz capsules, the buttons were crushed and pulverized by ball milling under toluene in a steel planetary mill for 20 minutes. The resulting powder was rinsed with acetone and vacuum dried. Rubber tubing, 1/4" I.D. and 1-1/2" long, was manually filled with powder. Magnetic alignment of the powder particles was one of the most critical processing steps. The objective during the compacting stage was to maintain a good degree of alignment in the compound by mechanically interlocking the powder particles. For this purpose, the powder was aligned in DC fields up to 20 KOe, then the volume was reduced by evacuating the rubber tube to lock the particles in position. The tube was subsequently hydrostatically compressed at 70 Kg/mm². Although in the first set of experiments X-ray examination showed only a small degree of alignment, nevertheless, a definite improvement in magnetostriction was observed. A substantially higher degree of alignment was achieved when an alternating field of approximately 1000 Oe peak-to-peak at frequencies up to 500 Hz was superimposed on the DC field. A field of this type produced sufficient particle vibration to facilitate orientation of the loose powder. While in the magnetic field, the powder was compacted by hand applied end compression with a plunger before it was
isostatically compressed. The cold pressed samples were subsequently wrapped in Ta foils and sintered in a dynamic vacuum of $3 \times 10^{-6}$ mm Hg in the 950-1050°C temperature range for durations up to 12 h. The samples were evaluated by optical microscopy, X-ray diffraction patterns, X-ray pole figures and magnetostriction measurements. Samples measuring approximately 2 cm$^2$ by 0.3 cm thick were polished through a 1 μ diamond wheel and, after etching, were used to obtain X-ray diffraction patterns and pole figures. Plane orientations were determined by the Schulz method$^9$ using a Picker diffractometer with an X-ray monochromator. Intensities of (440) reflections were recorded as $\omega$ (the angle of reflecting plane with sample surface), and $\phi$ (the azimuth angle from the longitudinal directions of the sample) were changed. A temperature compensated circuitry with commercially available strain gauges from Micro-Measurments was used to determine the magnetostrains.

Figure 1 shows the X-ray diffraction patterns of random and magnetically oriented Tb$_{.3}$Dy$_{.7}$Fe$_2$ compound. In the surface perpendicular to the magnetic field, the diffraction intensity from (220) planes has decreased while the intensity of (222) reflection has increased, to a substantial degree, with respect to the intensities of reflections from these planes in an almost randomly oriented powdered compound. Low field magnetostriction measurements at room temperature show that with regard to both magnetostrains and rate of approach
to saturation, the aligned material, though containing considerable porosity, is much superior to a highly dense (95% theoretical density) liquid phase sintered compound which is not aligned (Fig. 2a). This superiority is possibly due to the effect of the preferred grain orientation on reducing the large internal strains at grain boundaries associated with highly magnetostrictive materials. Also, it is noteworthy that in rare earth-iron Laves phase compounds \( \lambda_{\text{111}} \gg \lambda_{\text{100}} \) due to a structural distortion associated with the [111] easy direction of magnetization.\(^{10}\) (\( \lambda_{\text{100}} \) and \( \lambda_{\text{111}} \) are single crystal magnetostriction constants of cubic crystals in directions <100> and <111> respectively.) Since for a polycrystalline material with random grain orientation, the saturation magnetostriction can be expressed as

\[
\lambda_s = \frac{2 \lambda_{\text{100}} + 3 \lambda_{\text{111}}}{5},
\]

magnetic alignment of these compounds orienting the crystallites along the [111] easy axis, will enhance their saturation magnetostriction drastically. This is illustrated in Fig. 2b, where the saturation magnetostriction for the aligned \( \text{Tb}_{0.3} \text{Dy}_{0.7} \text{Fe}_2 \) shows a 20% increase over the highly dense liquid phase sintered material.

It was also found that the alignment improves as the value of \( x \) in \( \text{Tb}_x \text{Dy}_{1-x} \text{Fe}_2 \) is increased, as is shown in Fig. 3, where the relative intensity of reflections from (222) and (220) planes are plotted against values of \( (x) \). This is attributed to the fact that \( \text{Tb}_x \text{Dy}_{1-x} \text{Fe}_2 \) has a minimal anisotropy near \( x = 0.3 \). An increase in \( (x) \) will increase the
anisotropy making an improvement in the alignment degree possible. This is evident in X-ray diffraction patterns taken from aligned sintered TbFe$_2$ materials (Fig. 4a). In the plane perpendicular to the direction of the field, the (220) reflection is almost non-existent whereas the reflection from (222) planes has the strongest intensity. Figure 4b shows a (440) pole figure for an aligned TbFe$_2$ sintered rod with contours of constant intensity around the (440) pole, at the center. Although a decrease in (x) toward a Dy richer compound also will increase the magnetic anisotropy, it was not considered as beneficial, since DyFe$_2$ has its easy direction of magnetization along the [100] instead of along the [111] direction, thus not contributing much to the magnetostriction.

Thus, it has been shown that an aligned sample, though porous, has superior magnetostriction as compared with an unaligned dense sample. Work is underway to eliminate this porosity which is considered to be detrimental, and therefore, even greater magnetostrictions are anticipated.

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REFERENCES

FIGURE CAPTIONS

Fig. 1: X-ray diffraction patterns for an almost randomly oriented powder and a magnetically aligned specimen of Tb$_{0.3}$Dy$_{0.7}$Fe$_2$ compound.

Fig. 2:
(a) Low field room temperature magnetostriction of an aligned and a liquid phase sintered specimen of Tb$_{0.3}$Dy$_{0.7}$Fe$_2$.
(b) High field room temperature magnetostriction of the same specimens as in (a), indicating a saturation magnetostriction for the aligned sample approximately 20% greater than that of the highly dense liquid phase sintered specimen.

Fig. 3: Change of X-ray reflection intensity ratios, (220)/(311) and (222)/(311), vs. the concentrations of Tb and Dy in magnetically aligned specimens.

Fig. 4:
(a) X-ray diffraction patterns for a magnetically oriented TbFe$_2$ specimen.
(b) The (440) pole figure for the specimen.
Fig. 2
Fig. 3
Fig. 4
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