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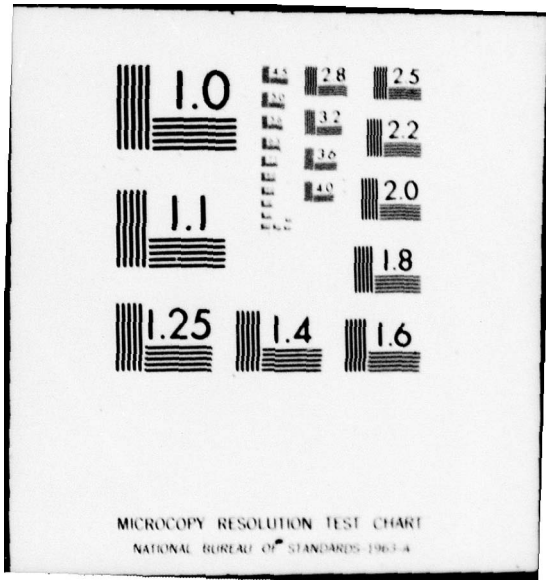
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DOMAIN ORIENTATION IN SINGLE CRYSTAL $TbFe_2$ AND $Tb_{.27}Dy_{.73}Fe_2$

BY R. S. SERY
H. T. SAVAGE

RESEARCH AND TECHNOLOGY DEPARTMENT

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NAVAL SURFACE WEAPONS CENTER
Dahlgren, Virginia 22448 • Silver Spring, Maryland 20910

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We have observed 71° and 109° walls in single crystal $TbFe_2$ and $Tb_{.27}Dy_{.73}Fe_2$ (Terfenol) via the Kerr effect and synchrotron radiation topographs. A Terfenol (110) surface exposed to synchrotron radiation showed stripe-like domains with [110], 109° walls & [001], 71° walls. Cooling to ≈273K caused an easy axis switch from <111> to <100>; no domains were visible. Warming back to ≈293K brought out mostly 109° walls. Stripelike

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domains were also seen in (110) and (100) $TbFe_2$ surfaces via the Kerr effect. In the (110) sample H fields along [001] and [110] directions created 109° and 71° walls, respectively. With H applied at ±45° to both [001] and [$\bar{1}$10] directions 71° and 109° walls appeared simultaneously. The domain widths varied from 5 to 250 μm and fields of ±3000 Oe away from [001] and [$\bar{1}$10] directions saturated the sample. Walls in the (100) sample were also 71° or 109° but were only 2 to 15 μm wide.

SUMMARY

The domain wall study reported here is part of a research and development program to develop magnetostrictive materials for high power projectors for sonar applications. Here is detailed the domain wall configurations in single crystal $TbFe_2$ and $Tb_{.27}Dy_{.73}Fe_2$ metallic compounds which exhibit huge magnetostriction and magneto-mechanical coupling.

It has been demonstrated that 109° and 71° walls exist in these highly magnetostrictive materials. The existence of these highly energetic walls was unexpected. In polycrystalline samples the above walls were also present but a greater variety of walls, many of them of the strain-induced type were seen.

Synchrotron radiation topography proved successful in revealing not only the presence of 109° and 107° walls but their disappearance on going from room temperature to ≈273K where an expected easy axis transition from <111> to <100> directions occurred.

The study reported herein was carried out in the Solid State Branch of the Materials Division as part of the Transduction Block Program of the Naval Oceans Systems Center (F11121). The development of rare earth materials for this study was approved jointly by the Naval Research Laboratory Material Block Program (Code 5220) and the Office of Naval Research (PO4-0081,NR 039-110).

Domain wall observations by synchrotron radiation were made using the electron synchrotrons NINA at Daresbury Laboratory and DESY at Hamburg under the direction of B. K. Tanner and G. F. Clark, University of Durham, Durham, England.

J. R. DIXON
By direction

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INTRODUCTION

We have observed domain configurations in single crystal and polycrystalline $TbFe_2$ and $Tb_{.27}Dy_{.73}Fe_2$. The two compounds are Laves (cubic) phase materials. They are ferrimagnets with a dominant rare-earth sublattice. The magnetostriction[1] is quite anisotropic with $\lambda_{111} = 1.6 \times 10^{-3}$ and $\lambda_{100} \simeq 10^{-4}$ in $Tb_{.27}Dy_{.73}Fe_2$ at room temperature. In $TbFe_2$ $\lambda_{111} = 2.4 \times 10^{-3}$ and λ_{100} is much smaller at room temperature. In $TbFe_2$ the magnetocrystalline anisotropy, K_1, is about -7×10^7 ergs/cm^3 and ΔK_1 (the magnetostrictive contribution constant) is a factor of 10 lower. The <111> axis is easy. In Terfenol the magnetocrystalline anisotropy is greatly reduced by the choice of the Tb/Dy ratio[1]. The easy axis is <111> above ≃273K and switches to <100> below it. The anisotropy energy is about -2×10^5 ergs/cm^3 at room temperature*. ΔK_1 is $\simeq -5 \times 10^6$ erg/cm^3.

Table 1 summarizes the values of these properties for Terfenol ($Tb_{.27}Dy_{.73}Fe_2$) and $TbFe_2$ and, for comparison, shows the corresponding values for Ni.

EXPERIMENTAL PROCEDURES

Crystallographic (110) and (100) surfaces and directions of interest were determined by Laue back-scattering x-ray techniques and samples were cut along these planes. A typical surface was prepared by mounting the sample in koldmount, a fast setting resin. Next the surface with the exposed face of the sample was given a two to six minute fine grinding on lubricated 600A paper. This was followed by successive polishings on diamond paste impregnated felt wheels using 6 μm diamond particles and 1 μm particles respectively. This was followed by electropolishing or light (2 to 15 secs) etching with a 5% Nitol solution to relieve surface strains and thus eliminate the appearance of strain induced domains.

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1. Clark, A. E., et al., "Rhombohedral Distortion in Highly Magnetostrictive Laves Phase Compounds," Amer. Inst. Phys. Conf. Proc. No. 29, 9-12 Dec 1975; publ 1976, pp. 192-193.

* Private communication, Conrad Williams, Naval Research Laboratory, Washington, D.C.

TABLE I

	$Tb_{.27}Dy_{.73}Fe_2$	$TbFe_2$	Ni
λ_{111}	1.6×10^{-3}	2.4×10^{-3}	-24×10^{-6}
λ_{100}	-10^{-4} to -10^{-5}	$\simeq 1 \times 10^{-4}$	-46×10^{-6}
K_1, $\frac{ERGS}{cm^3}$, (300K)	2×10^5	-7×10^7	-5×10^4
ΔK_1, $\frac{ERGS}{cm^3}$	-5×10^6	$\simeq -10^7$	-0.3×10^4
Easy Axis $T \gtrsim 273K$	<111>	<111>	<111>
Easy Axis $T \lesssim 273K$	<100>	<111>	<111>

An adjustable pole-piece electromagnet was used to produce magnetic fields. A Harrison 6386A dc power supply furnished 0 to 11.5A dc at 24 volts. Fields of up to 3.2 kOe were available with the minimum gap width used.

A Vickers Projection Microscope with polaroid type analyzer and polarizer lenses was used for visual observations. Data recording was effected with photography and videotape. Normal incidence Kerr Magneto-Optics are utilized in the instrument. A second method of observation employed was that of the Bitter solution technique using 200G water based Ferrofluid in Dodecylamine Hydrochloride. The third technique was that of using synchrotron radiation for making surface reflection topographs taken in the Bragg geometry, wherein only the first few micrometers of the strain free surface are explored. This work was done on a single crystal of $Tb_{.27}Dy_{.73}Fe_2$.

RESULTS

The domain wall configurations observed in two single crystal $TbFe_2$ samples having nominal (110) surfaces is shown in Figs. 2 and 3. For these samples the experimental observations fit the model shown in Figure 1. Applying a field of $\gtrsim$ 350 Oe in a [001] direction caused straight parallel domains to form with 109° walls along a $[\bar{1}10]$ direction and orthogonal to the field direction. (See Figure 2). Such walls were stable against magnetic fields of up to 3 kOe showing only slight changes in some domain widths, e.g., $[\bar{1}1\bar{1}]$ domains increasing, $[1\bar{1}\bar{1}]$ decreasing. If the 350 Oe field was next applied momentarily in the $[\bar{1}10]$ direction these walls disappeared and 71° walls running in the [001] direction appeared at right angles to the direction of the previous walls. These were also barely affected by repeated applications of 3 kOe fields normal to the wall direction. Although the domains shown in Figures 2 and 3 are mainly straight parallel domains others, not shown, have appeared as very elongated spike domains.

Figure 1 depicts all magnetic moments as being in plane e.g. for $\underset{\sim}{M}_1$ along $[\bar{1}11]$ for the 109° walls. However normal components of magnetization did exist and thus were visible as in Figures 2 and 3 via the normal incidence Kerr effect. Repeated cycles of polishing and etching caused the sample surfaces to depart from true (100) surfaces by several degrees. Both samples also had 1250Å thick ZnS coatings on for Kerr effect enhancement.

Fields of two to three kOe but at angles of $\gtrsim$30° from either the [001] or $[\bar{1}10]$ direction had effects similar to the ones mentioned above. The differences were that it was easier to move domain walls, i.e., to cause near saturation with the field and a return to the original or similar wall configurations with field removal. However, if similar fields were applied at $\approx$45 ± 5° to these directions orthogonal sets of domains appeared to be stable. The domain widths

in Figures 2-3 vary from about 5 to 250 μm. The application of 2 to 3 kOe fields in a direction normal to the walls caused changes (narrowing of some domains) of ≈10% in some domains and negligible changes (<5%) in others.

A $TbFe_2$ single crystal with a polished (100) face also contained sets of long straight domains. The widths of these domains were very much smaller than in the two (110) samples being, for most of them, within a range of 2 to 15 μm wide. In addition numerous more complicated types of small dagger and zigzag domains appeared in between many of the long straight ones. These appeared, at least photographically, in more detail when Ferrofluid was used (Bitter solution technique) than were brought out by the Kerr Effect (Figure 4).

The orientation of domain walls and easy axes of magnetization for the (100) samples are shown in the model depicted in Figure 5. Easy <111> directions are shown at angles of ± 35° to the surface. The walls tend to lie along [110] or $[\bar{1}10]$ directions, i.e. along either of two orthogonal directions of the <110> set. Applied fields along the domain wall directions [110], (or $[\bar{1}10]$) wiped out the domains and on removal caused new ones to form, orthogonal to the field alignment in $[\bar{1}10]$ (or [110]) directions. Fields applied orthogonally to the walls in either case caused slight changes in wall appearance, one set widening the other narrowing but, at values up to ≈2700 Oe, did not cause saturation. Here the visibility of the domains via the Kerr effect is insured by the sizable components of magnetization which exist normal to the (100) face.

We observed the appearance of lines caused by Widmanstätten precipitates in the (100) surface. These needle-like precipitates in a (100) face appear as two sets of lines orthogonal to each other. One set appears along a [110] direction and the other along the $[\bar{1}10]$ direction. The long straight domains were aligned in one or the other of these directions. In some areas of the surface right angle jogs in the domains appeared. The domains on either side of the jog lay in one direction, say, [110] and the short parts correspondingly were in the $[\bar{1}10]$ direction. Successive jogs were offset in a fixed direction. A steadily increasing field caused the jogs to shorten and also to move giving the appearance (noticed more particularly, on occasion, when the domain patterns were observed using Ferrofluid) of a diagonal wall moving in the [110] direction. Figure 4 is an example of the presence of jogs. Similar jogs-though not right angle ones-have been seen in some areas of polycrystalline samples.

Several polycrystalline $Tb_{.27}Dy_{.73}Fe_2$ samples observed showed evidence of parallel straight domains but in limited areas of a given sample. Stripe-like structures of ≈2 μm widths have been seen. In some areas of a sample no domains could be detected. Presumably the magnetization in these areas was all in plane. Other areas revealed quite complicated patterns some even resembling those seen on the c plane of Cobalt samples, i.e., starlike domains where the moments are aligned principally into or out of the face observed. In some

instances bubble domains were also seen. In general a greater variety of domain configurations was seen in the polycrystalline samples.

In the synchrotron studies done at the University of Durham[2-4] the effect of cooling from room temperature to ≈273K on domain formation in $Tb_{.27}Dy_{.73}Fe_2$ was observed, Figure 6 shows the (110) face of the sample at room temperature in which 109° and 71° walls appear. Figure 7 shows that no domains were visible at ≈273K (past an expected transition of easy axes from <111> to <110> the relatively small magnitude of λ_{100} prevents the observation of the domain configuration using Synchrotron radiation). On warming, the resulting domain pattern was dominated by many parallel 109° walls running in the $[1\bar{1}0]$ direction (Figure 8). A 400 Oe field did not cause any changes but application of a 1.5 kOe field and return to zero (H_o = 36 Oe) resulted in a pattern consisting mainly of walls parallel to $[1\bar{1}1]$. Further increases to 400 Oe caused slight changes in domain widths $[1\bar{1}1]$ increasing slightly while $[\bar{1}1\bar{1}]$ decreased.

In summary a number of different patterns have been found which depend to some extent on the previous history of the sample. In both Terfenol and $TbFe_2$ single crystals 109° and 71° walls have been found by means of conventional microscopy using the Kerr effect and the Bitter solution technique and by a much newer method, for the Terfenol samples, that of synchrotron radiation topography. The presence of 71° and 109° walls is surprising because of the high magnetoelastic energy associated with such a configuration. The inability to observe any walls with synchrotron radiation when the easy axis is <100> is further proof of the small size of λ_{100}.

Polycrystalline patterns were more varied and domain widths were an order of magnitude smaller than single crystal widths. This is due to the large internal strains that take place when the temperature of the polycrystal is lowered through the Curie point. Domain widths were roughly 50 μm in the single crystals. In $TbFe_2$ the (100) surface domain widths were smaller than those seen on the (110) surface.

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2. Tanner, B. K., "Crystal Assessment by X-ray Topography Using Synchrotron Radiation", Progress in Crystal Growth and Characterization, Pergamon (Oxford), 1977, 1(1), 23-56.

3. Tanner, B. K., X-ray Diffraction Topography, (London: Pergamon Press, 1976), pp. 93, 119.

4. Tanner, B. K., et al., "Cryogenic X-ray Topography Using Synchrotron Radiation," Journal of Applied Crystallography, 10, Pt. 2, 1977, pp. 91-99.

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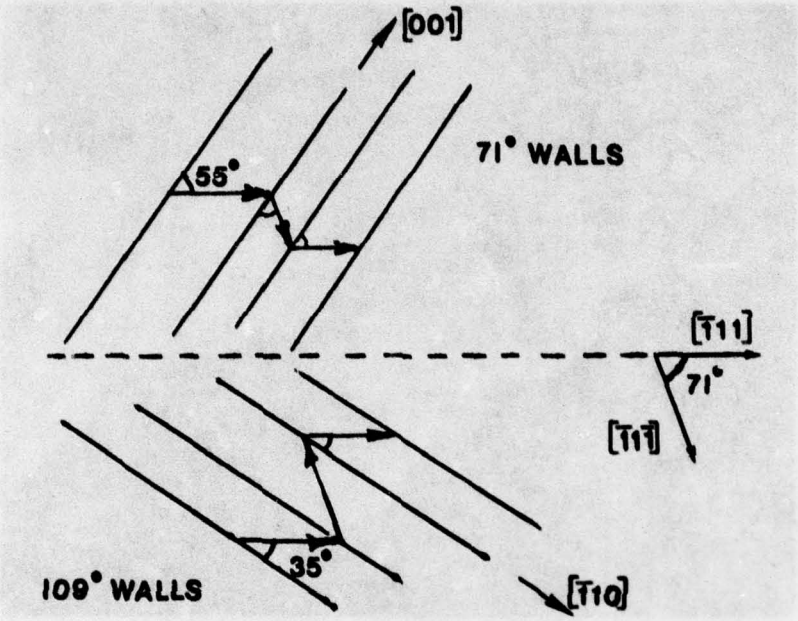

FIGURE 1 MODEL OF 71° AND 109° WALLS OBSERVED IN (110) SURFACES OF $TbFe_2$ SINGLE CRYSTALS.

FIGURE 2 109^{o} WALLS OBSERVED IN THE (110) SURFACE OF A $TbFe_2$ SINGLE CRYSTAL, KERR EFFECT.

FIGURE 3 SIMULTANEOUS APPEARANCE OF 109° AND 71° WALLS IN THE $TbFe_2$ SINGLE CRYSTAL, KERR EFFECT. LINES ADDED TO DELINEATE DOMAINS.

FIGURE 4 DOMAIN WALLS IN A $TbFe_2$ SINGLE CRYSTAL (100) FACE AS REVEALED BY FERROFLUID. APPLIED H $\simeq$ 1.2 kOe AND IS UP IN THE PHOTO.

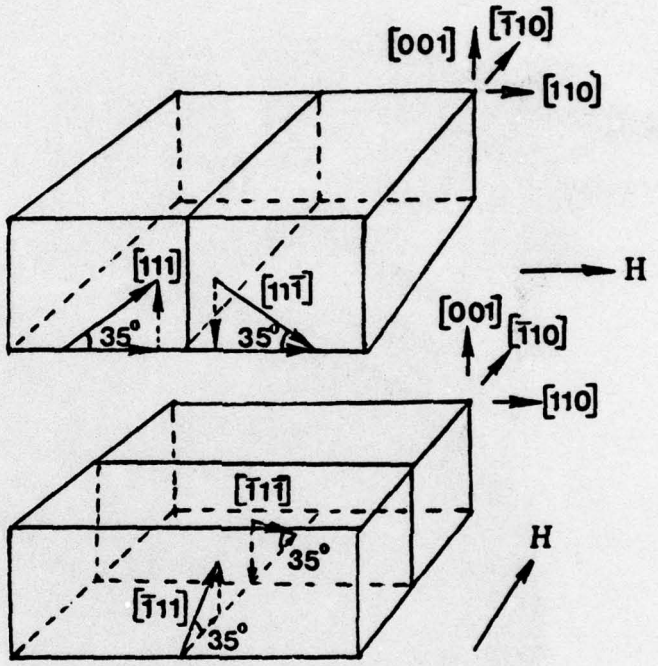

FIGURE 5 MODEL OF 71° WALLS OBSERVED IN A $TbFe_2$ SINGLE CRYSTAL (100) SURFACE.

FIGURE 6 SURFACE REFLECTION TOPOGRAPHS OF 109° AND 71° WALLS IN A (110) SURFACE OF A $Tb_{.27}Dy_{.73}Fe_2$ SINGLE CRYSTAL AT≃300 K.

FIGURE 7 SAME SURFACE AS FIGURE 6 SHOWING NO WALLS VISIBLE AT $\simeq$ 273 K.

FIGURE 8 SAME SURFACE AS FIGURE 6 AFTER REWARMING TO ≃300 K. MOSTLY 109° WALLS ARE PRESENT RUNNING IN THE [1$\bar{1}$0] DIRECTION (AVERAGE SPACING ~ 50μm).