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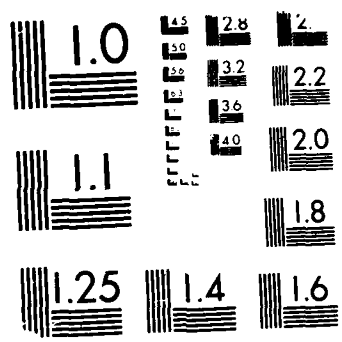

MICROCOPY RESOLUTION TEST CHART

Final Report

Dr. Joseph P. Teter

Report period ending November 1, 1987

Acknowledgments:

I wish to start my final report by expressing my gratitude for all the help that I received during the last two years. Specifically I wish to recognize the efforts of three groups; Dr. Louis Scmidt and this staff at ONR, Charles Carter and David Thornell at ASEE and, Dr. Arthur E. Clark, Dr. James Cullen and all the fine people of the Radiation Division at the Naval Surface Warfare Center (NSWC).

Abstract:

The two man-years worth of my Post-doctoral appointment to the Solid State group of the Radiation Division of NSWC has produced major advances in the understanding of the phenomenon of magnetostriction as evidenced by the 4 papers published in scientific journals. An appendix lists these papers plus 1 other paper that has been published in colaboration with numerous scientists at various institutions such as the Applied Physics Lab of the John Hopkins University and the University of Salford in England. There are three papers yet to be written for which I have contributed small portions of experimental datas. The two most recent papers listed in the appendix have not been included in previous quarterly reports and are, therefore, attached to this final report for completeness.

Other major achievements include :

1) A contributed section to a large multi-institution proposal in the field of superconductivity. This proposal has been approved and will be funded starting January 1988.

2) Participation in eight conferences ranging from the very large attendance American Physical Society March Meetings to the small, intense Terfenol Workshop at Iowa State U. I have presented three papers at topical conferences on magnetostriction and have given two general lectures in the field of superconductivity.

3) It was my pleasure to participate in the CEOP program at NSWC. This involved interviewing and subsequently teaching a gifted high school student how work in experimental science is accomplished.

The major topics are covered in detail in the following sections. The minor topics were adequately covered by the previous quarterly reports.

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MEMORANDUM

TO: Defense Technical Information Center

FROM: David Thornell
Associate Program Manager, Projects and Federal Relations

SUBJECT: Final Technical Report, ONT Postdoctoral Fellow
Contract N00014-83-D-0689

DATE: February 9, 1988

Under the subject contract for administering the ONT Postdoctoral Fellowship Program final technical reports are to be submitted by participating Fellows at the conclusion of their tenure.

Enclosed are twelve (12) copies of the final technical report for Dr. Joseph Paul Teter relating to the research work completed at the Naval Surface Warfare Center.

Please do not hesitate to contact me if you have any questions.

A. Magnetostriction

The work done in the field of magnetostrictive materials centers on the base intermetallic compound known commercially as Terfenol-D . This compound has the nominal stoichiometry of 3 tenths Dysprosium plus 7 tenths Terbium plus 2 Iron atoms. The rare earth ellements, Dysprosium and Terbium, are magnetically balanced to produce a compound with Iron that can exhibit strains in excess of 2000 parts per million at room temperature when a sufficiently large magnetic field is induced parallel to the 111 crystallographic axis. This material was developed by Dr. A.E. Clark here at NSWC over 10 years ago. Work continues on the problems of understanding the basic physics of the material and developing new material with higher field induced strains.

My contribution to these efforts has been to characterize and help interpretate the experimental datas that I have collected using apparatus that I have designed and built. These datas can be grouped into four categories; alloy studies, polycrystals, twinned pseudo-single crystals, and true untwinned single crystals.

The first two, alloy studies and polycrystaline properties resulted in the publication of reference #3. Here the substitution of cobalt and nickel in place of iron was examined with the goal of producing Terfenol type material that had both high magnetostriction and enhanced resistance to corrosion. The results were encouraging but not spectacular enough to warrant a continuing research project.

The third set of datas, twinned pseudo single crystals, represents one of the better successes as it yielded a new piece of information relating to the basic physics of magnetostriction. Specifically, a section of twinned rod that has been grown in the preferred 112 direction will, when energized by a magnetic field, display switch-like behavior of the magnetostriction as a function of magnetic field. The model which explains this behavior in terms of magnetization vector rotation in the twins and the data was presented by me at the conference of Magnetism and Magnetic Materials held November 9 - 12 in Chicago. This paper is attached to this report and will be published (reference #1).

The last category, true single crystal work, produced data that showed that the direction in which the magnetic field is applied may not be the direction in which the maximum crystalographic strain is observed. This apparent discrepancy was explained through use of a rigorous calculation of magnetoelastic energies by Dr. Clark (reference #4). For this study a large single crystal was sectioned in such a way as to produce thin discs each of which had a different crystalographic orientation. Then each disc was examined by placing sensors along different principle and non-principle crystal axes. One of the discs (112 face) along with several pieces were subjected to topological surface observation using a phase interference microscope. This study revealed the size and structure of the magnetic domains (reference #2). Work is still progressing on the single crystal samples.

B. Superconductivity

One year ago, a new class of superconducting materials was announced to the world. That announcement caused nearly everyone working in solid state physics to drop what they were doing and examine this new phenomenon. NSWC was no exception.

My involvement in this new field started early on when I brought back, from Temple University, a piece of one of the first batches of the material. This early involvement evolved to active participation in all phases of Navy interest. Specifically, Dr. James Cullen and myself have received one third of all internal research money that the Center is spending on superconductivity. This money is used by me to examine the basic magnetic properties of the material and to examine innovative approaches to the problem of producing the material in different forms, compositions, and purities.

I have also helped initiate a proposal which deals with sample preparation problems which has been submitted to the Office of Naval Research. This proposal is for a million dollars plus and has been written on the subject of processing thick and thin films of the new high temperature superconductor. This proposal includes a consortium of industry, universities and government laboratories. My part was to write the section on characterizing the infared response of these films. I will also be responsible for carrying out the proposed research in infared response.

C. Metal Matrix Epoxies

This program involved the study of a NASA epoxy previously used to hold the heat shield tiles onto the space shuttle. A problem with the material was that it is hydroscopic. It was determined that the addition of small amounts of metal ions into the epoxy drastically reduced the materials affinity for water. A program was initiated to determine how these metal ions were able to effect the rate of water absorption. My part of this program was to determine the valence state of the ions using magnetic moment determinations on samples provided. As a part of this program I employed a CEOP student during the summer to do measurements of magnetic moment using a vibrating sample magnetometer. The results along with other experiments done by other researchers showed an effective decrease in the valence of the metallic ions as the concentration of ions was increased. (reference 5)

Appendix:

Refereed Publications

1. Magnetostrictive "Jumps" in Twinned Tb.27 Dy.73 Fe1.9 submitted to MMM-87, with A.E. Clark and O.D. McMasters

2. Optical Observation of Closure Domains in Terfenol-D Single Crystals submitted to the European conference on magnetic materials with D.G. Lord, V. Elliot, A.E. Clark, H.T. Savage and O.D. McMasters

3. Magnetostrictive properties of Tb.3 Dy.7 [Fe(1-x) Co(x)]1.9 and Tb.3 Dy.7 [Fe(1-x) Ni(x)]1.9 presented at INTERMAG 1987 and published by same, with A.E. Clark and O.D. McMasters

4. Anisotropic Magnetostriction in Tb.27 Dy.73 Fe2, submitted to MMM-86 and published in J. Appl. Phy. 61, 15 April 1987 pg. 3787 with A.E. Clark and O.D. McMasters

5. Effects of Transition Metal Ions on Positron Annihilation Characteristics in Epoxies, Nuclear Instruments and Methods in Physics Research Journal, B26 Jan 1987 pg. 598 with J.J. Singh, D.M. Stoakley, W.H. Holt and W. Mock, Jr.
   Also presented at the APS January Meeting, 1987

MAGNETOSTRICTION 'JUMPS' IN TWINNED $TB_{.3}DY_{.7}FE_{1.9}$

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ABSTRACT

Large 'jumps' in the magnetostriction have been observed in twinned single crystals of $Tb_{.3}Dy_{.7}Fe_{1.9}$ (Terfenol-D) for magnetic fields parallel to the crystalline $[11\bar{2}]$ direction. The interpretation of these large magnetostriction discontinuities is based upon a model of twinned dendritic Terfenol-D in which the magnetization of one twin jumps between two [111] directions while the magnetization of the remaining twin undergoes a continuous rotation of the magnetization. The field dependence of the magnetization and magnetostriction of cubic single crystals with $\lambda_{111} >> \lambda_{100}$ was calculated using an expression which included the anisotropy constants K_1 and K_2 and compressive loads along [112]. With $K_1 = -0.6\ J/m^3$ and $K_2 = -2.0\ J/m^3$ (values appropriate for Terfenol-D near room temperature), magnetization 'jumping' is predicted. For the twinned crystal, the jump in the magnetostriction was calculated to be greater than 1000 ppm. Because of this large magnetostriction, it is possible to configure a device to perform a substantial amount of work by the application of only a triggering magnetic field centered about an optimum bias field.

Temperature dependences of the magnetostriction and magnetization were made on twinned [11$\bar{2}$] Terfenol-D with applied stresses up to 50 MPa and temperatures from -50 C to +90 C. The character of the magnetization and magnetostriction curves change drastically as the anisotropy constant K_1 changes from negative to positive.

INTRODUCTION

Recent studies have shown that the highly magnetostrictive alloy $Tb_xDy_{1-x}Fe_{2-y}$, x = .3, 0 < y < .2 (Terfenol-D) grows perferentially along a crystallographic $[11\bar{2}]$ direction in twinned dendritic sheets.[1] Because the growth direction is a non-principal direction and because $\lambda_{111} >> \lambda_{100}$, magnetostrains along $[11\bar{2}]$ depend upon the magnetization in an unexpected way. In untwinned single crystal Terfenol-D, for example, the maximum magnetostriction does not occur with the magnetization direction parallel to the $[11\bar{2}]$ measurement direction, but with the magnetization directed at some angle between the measurement direction and the nearby $[11\bar{1}]$ direction.[2] In a twinned crystal, the magnetostriction is even more complex and difficult to analyze. The purpose of this paper is to examine the magnetostriction of twinned single crystalline Terfenol-D over a broad temperature range spanning room temperature. At low temperatures, the non-magnetostrictive <100> axes are magnetically easy. At high temperatures, the highly magnetostrictive <111> axes are easy and magnetization 'jumping' can occur between the perpendicular [111] direction and the $[11\bar{1}]$ direction close to the $[11\bar{2}]$ measurement axis. Huge changes in sample length accompany these jumps. Internally stored magnetic energy is released abruptly during the jump and can appear as work done against an externally applied stress.

EXPERIMENTAL

Twinned $[11\bar{2}]$ rods of $Tb_{.3}Dy_{.7}Fe_{1.9}$ (1/4" dia. x 6" long) were prepared by a free standing r. f. zone method (FSZ) using a zoning rate of 20 in./hr. The $[11\bar{2}]$ crystal axes are parallel to the rod axes to within 2 degrees. Magnetostriction measurements were made on 2.2" long samples using standard strain gage techniques employing a Tenney temperature controlled chamber. The magnetic field was applied quasi-statically in 30 ms steps by a solenoid placed around the cylindrical Terfenol sample. A soft iron path provided flux return. In a conventional fashion, the magnetization was calculated from the flux change in a pick-up coil wound around the sample. Measurements were made under static compressive loads (σ) from 0 to 25 MPa at temperatures (T) from -50 C to 90 C.

Fig. 1 illustrates the progression in the temperature dependence of the magnetization and magnetostrictive behavior at 13.3 MPa as the sample undergoes the transition from a strong positive anisotropy (<100> easy) at low temperature to a strong negative anisotropy (<111> easy) at high temperatures. At -50 C a conventional magnetization curve with a small magnetostriction is observed. The primary magnetization process occurs by 180 degree wall motion, followed by some magnetization rotation away from the non-magnetostrictive <100> axes. From -50 C to -10 C the magnetostriction gradually increases until at -10 C, $K_1 \approx 0$. At -10 C with the compressive stress of 13.3 MPa providing a preferential transverse easy direction, the magnetization lies along the magnetostrictive [111] direction for zero applied field. As the field in the solenoid is increased, a relatively minor change in length is observed until at a critical field, H_{cr}, an abrupt change in length occurs with $\Delta\ell/\ell > 1000$ ppm. A still further increase in length occurs at greater fields. At 0 C and above, the <111> directions are sufficiently easy to cause the magnetization to 'jump' between two [111] directions as the magnetic field is applied. For 20 C < T < 80 C, the break in the curve at H_{cr} is even more distinct. At these higher temperatures, K_1 is more negative and the barrier between the [111] directions is higher. The decreasing value of magnetostriction with increasing temperature for 20 C < T < 80 C is due to the decrease in the saturation magnetostriction. At the higher temperatures (T > 0 C) both the magnetization and magnetostriction curves consist of three distinct regions: (1) a very low field region where there is little magnetization and magnetostriction, (2) a magnetization jumping region in which the magnetic moment jumps between two directions which have widely different magnetostrictions, and, (3) a magnetization rotation region where the magnetic moment rotates toward the $[11\bar{2}]$ rod axis. Substantial magnetostriction also occurs in the high field region.

Similar magnetostriction and magnetization curves are observed at compressive stresses $\sigma > 7.6$ MPa. As the compressive load is increased, however, a larger external field must be applied to produce the greater work done by the expansion against the load. This is illustrated in Fig. 3 for σ = 7.6MPa and 18.9 MPa. At very low applied stresses (σ < 5 MPa), internal stresses developed during the growth process become significant and prevent the H = 0 state from being the minimum magnetostriction state (one in which all domains point in the transverse direction). Fig. 2 shows that for zero applied stress there is no magnetization 'jumping'. In this case the magnetization process proceeds by 180 degree wall motion at low fields, followed by magnetization rotation at high fields. For σ = 0, only this latter process gives rise to magnetostriction. At 20 C approximately 5 MPa is required to achieve 70 degree wall motion and magnetostriction 'jumping'.

The experimental results are summarized in Fig. 3 for σ = 7.6 MPa and 18.9 MPa. Here H = 250 Oe, 500 Oe, 1000 Oe and 2000 Oe. The onset of the magnetostrictive state (<111> easy) occurs between -10 C and 0 C in $Tb_{1-x}Dy_xFe_{1.9}$ with x = 0.3. For samples where x < 0.3, this onset moves to higher temperatures.[3] The decrease in the saturation magnetostriction (λ_{111}) of Terfenol-D with temperature is also clearly seen in the curves for H = 1000 Oe and 2000 Oe. We find $\Delta\lambda_{111}/\Delta T$ = 5.6 x 10^{-6}/C. The important experimental feature of twinned single crystal Terfenol-D is the large change in length at low applied fields. Because of magnetization jumping, magnetostrictions ≈ 800 ppm exist at 250 Oe for σ = 7.6 MPa, and ≈ 600 ppm at 500 Oe with σ = 13.8 MPa.

DISCUSSION

Our proposed model of the magnetization process is depicted in Fig. 4. Here at H = 0 (and with pressure sufficient to populate only the [111] direction perpendicular to the rod axis), a single domain exists which traverses the dendrites. As the field is first increased the magnetic moments remain close to the perpendicular [111] direction until the Zeeman energy is sufficient to surmount the anisotropy energy barrier and perform the work required against the compressive load. In this region only a small magnetostriction results. At the critical magnetic field, H_{cr}, the magnetization of one twin 'jumps' to a new direction close to the $[11\bar{1}]$ direction 19.5 from the $[11\bar{2}]$ rod axis, while the magnetization of the other twin remains close to the perpendicular [111] direction (Fig. 4b). With a further increase in magnetic field, rotation of both twins toward $[11\bar{2}]$ occurs (Fig. 4c).

The application of this model to the data of Fig. 2 shows that the strain, $\Delta\lambda$, that occurs with magnetization jumping is given approximately by 1/2 of the saturation magnetostriction. For measurements along $[11\bar{2}]$ and rotation from [111] to $[11\bar{1}]$, the saturation magnetostriction = $(4/3)\lambda_{111}$. Thus we predict the strain in the twinned sample to be $\Delta\lambda = \Delta\lambda_p/2 + \Delta\lambda_t/2 = 0 + (2/3)\lambda_{111} = 1067$ ppm. Here we assume an equal volume of parent (p) and twin (t) and $\lambda_{111} = 1600$ ppm. The calculated value is very close to the observed value of 1000 ppm. Similarly the magnetization jump (ΔM) is simply given by $(M_s/2)\sin 70.5° \simeq .5$ T, where we take the saturation maganetization M_s to be 1.05 T. This is close to the observed value of $\Delta M \simeq .58$ T. At higher compressive loads, the magnetostriction and magnetization becomes somewhat smaller since the angle change is less than 70.5°.

Other magnetization processes were also considered. Two of these were: (a) simultaneous 'jumping' of both twins to their individual [111] directions, and (b) 'jumping' of one or both twins to <111> directions 61 degrees from [112] (out of the (110) plane). Model (a) gives a magnetostriction far in excess of that observed, ($\gtrsim$ 2000 ppm) and is unlikely to be correct because of the large dipole energy required. Model (b) yields magnetostrictions which are too small.

Finally, it is important to compare the work done against the compressive load to the magnetic energy supplied through the magnetic field. For pressures of 7.6 MPa, 13.3 MPa, 18.9 MPa and 24.5 MPa, we measured critical fields, H_{cr}, of 295 Oe, 500 Oe, 705 Oe, and 1000 Oe respectively. In Fig 5 we show the fraction of the magnetic energy ($M \cdot H$) converted to mechanical energy ($\sigma \cdot \Delta\lambda$) during the magnetization 'jumping' process. (The additional work done during the final rotation process is not addressed in the figure.) As the compressive load is increased, a larger fraction of the magnetic energy is converted to work.

Because of magnetization jumping, only a moderate triggering magnetic field (superimposed on a static bias field) is required to transfer the energy between the magnetic state and the mechanical state. The size of this field depends upon the magnetic hysteresis. The quality of the single crystal used in this experiment required ≈ 100 Oe to trigger the 'jump'. If a bias magnetic field (H_b) is introduced and the magnetic energy given by the triggering magnetic energy, $M \cdot (H - H_b)$ rather than by $M \cdot H$, the ratio of work to the magnetic energy, W/E_{mag}, can beome far greater than unity. Using this phenomenon and samples with low hysteresis, large amounts of energy can be transferred from the internal magnetic state to the external mechanical state by a small applied magnetic field. The strain discontinuities for twinned [112] samples are limited to ≈ 1000 ppm at room temperature. However in $[11\bar{2}]$ untwinned single crystals these discontinuities would be increased to ≈ 2000 ppm, and in single crystal $TbFe_2$ strains up to ≈3500 ppm might be achieved. A proposed application of these magnetostriction jumping alloys is a rapid solid state mechanical switch in which a large amount of energy is transferred between the magnetic and mechanical states. An "inchworm" or "magnetostriction" motor developed from these materials would require only small external magnetic fields.

REFERENCES

1. A. E. Clark, J. D. Verhoven, O. D. McMasters and E. D.

Gibson, IEEE Trans. on Magnetics, MAG-22, 973 (1986)

2. J. P. Teter, A. E. Clark, and O. D. McMasters, J. Appl. Phys. 61, 3787 (1987).

3. A. E. Clark and D. N. Crowder, IEEE Trans. on Magnetics, MAG-21, 1945 (1985).

FIGURE CAPTIONS

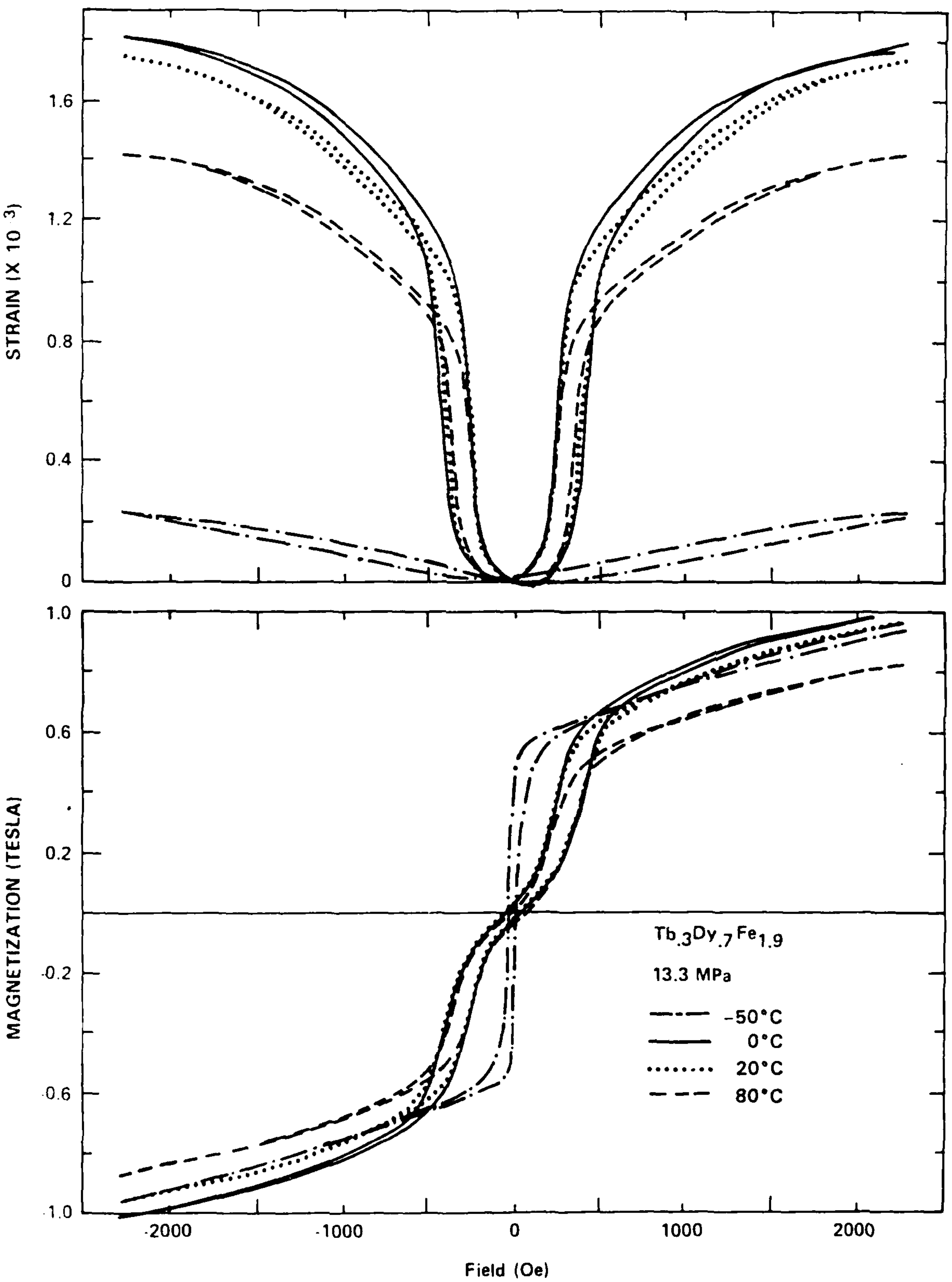
Fig. 1. Magnetostriction and Magnetization Curves of $Tb_{.3}Dy_{.7}Fe_{1.9}$ at 13.3 MPa Compressive Stress.

Fig. 2. Magnetostriction of the $Tb_{.3}Dy_{.7}Fe_{1.9}$ at 0 MPa, 7.6 MPa and 18.9 MPa.

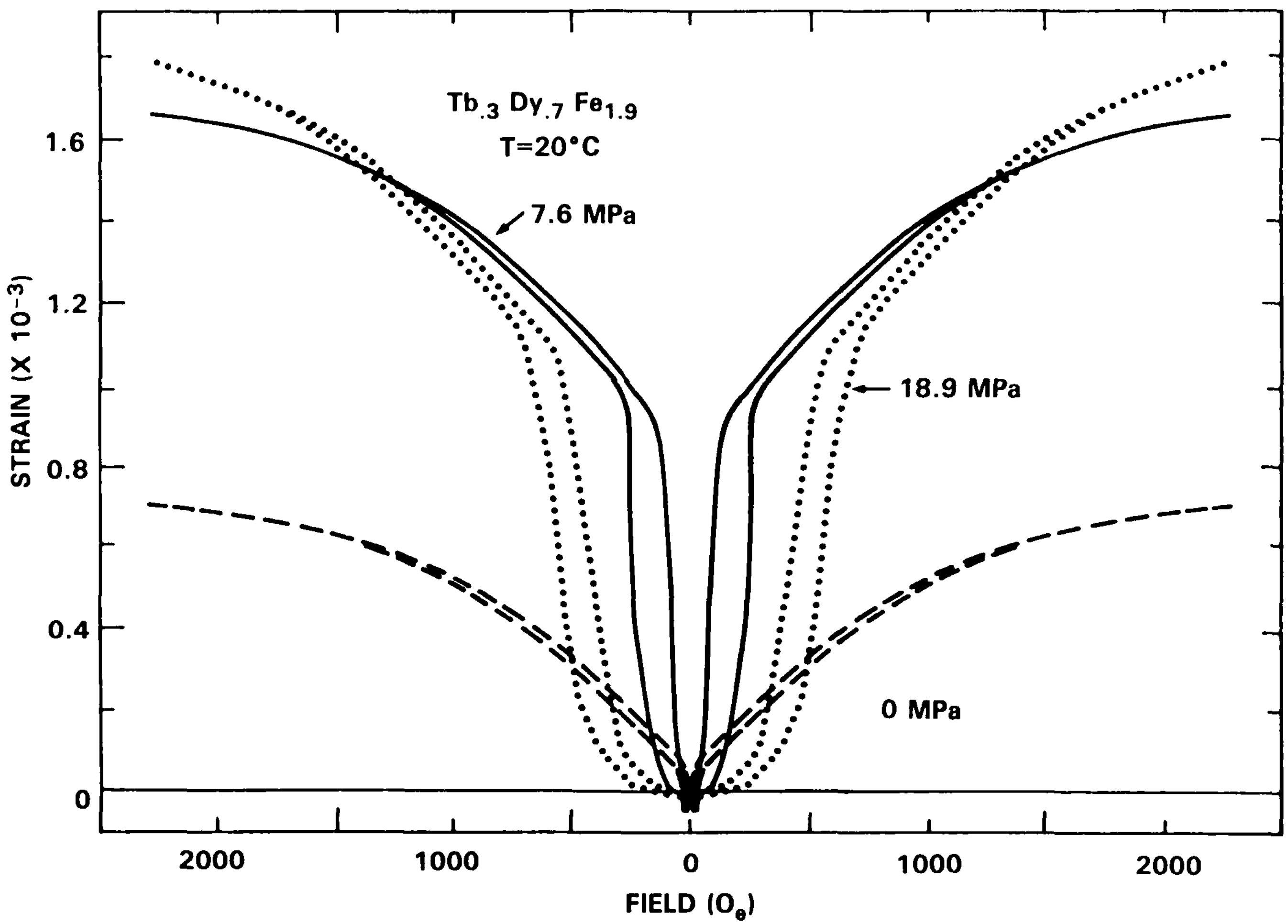
Fig. 3. Magnetostriction of $Tb_{.3}Dy_{.7}Fe_{1.9}$ vs Temperature for 7.6 MPa and 18.9 MPa.

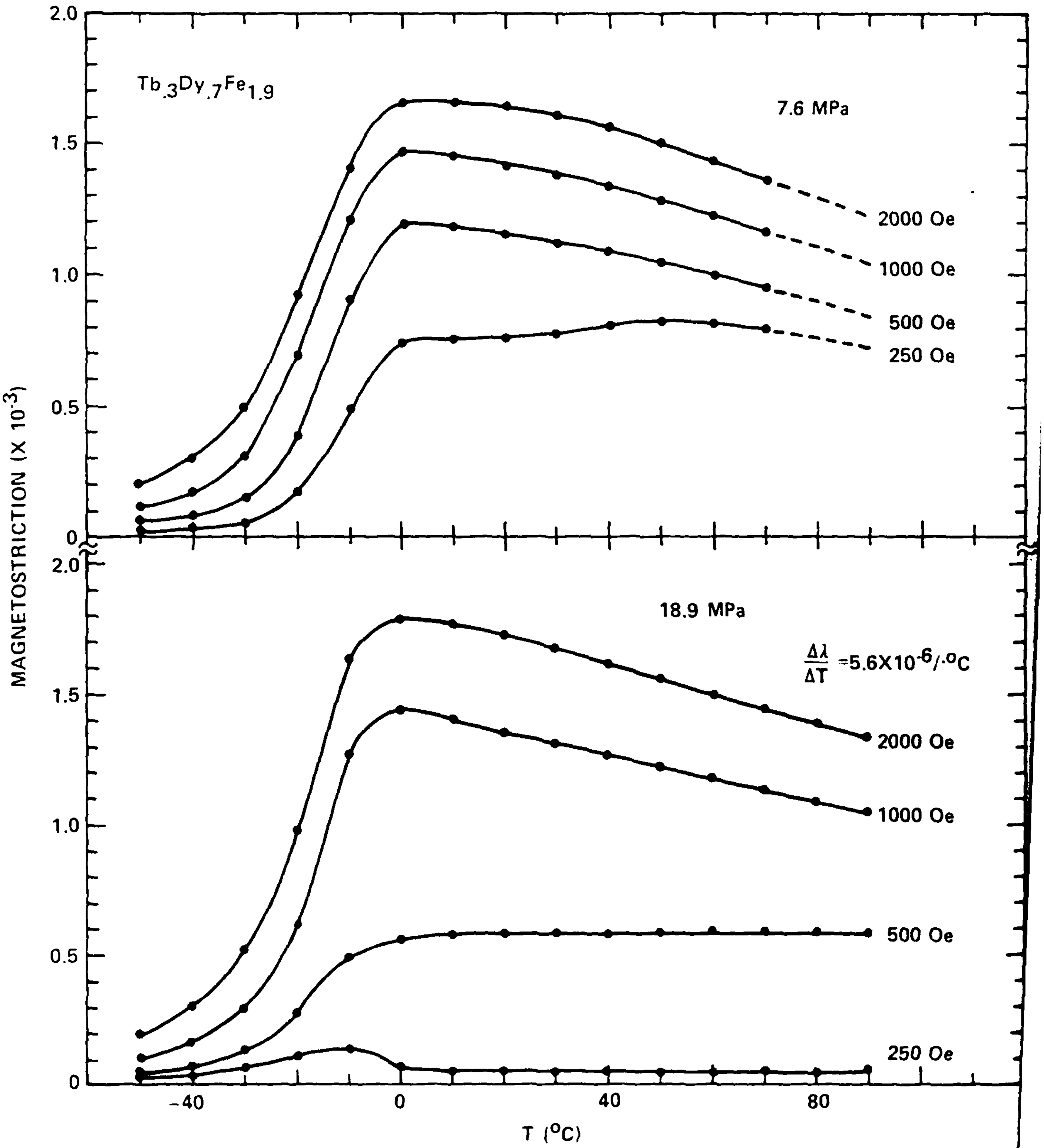
Fig. 4. Model of the Magnetization Process for [112] Twinned Terfenol-D Single Crystal.

Fig. 5. Magnetic Energy, $(M \cdot H)$, converted to Work $(\Delta\lambda \cdot \sigma)$ during the magnetization process. The dotted line represents lossless conversion. The vertical difference between the solid line and dotted line is a measure of the energy stored within the sample.

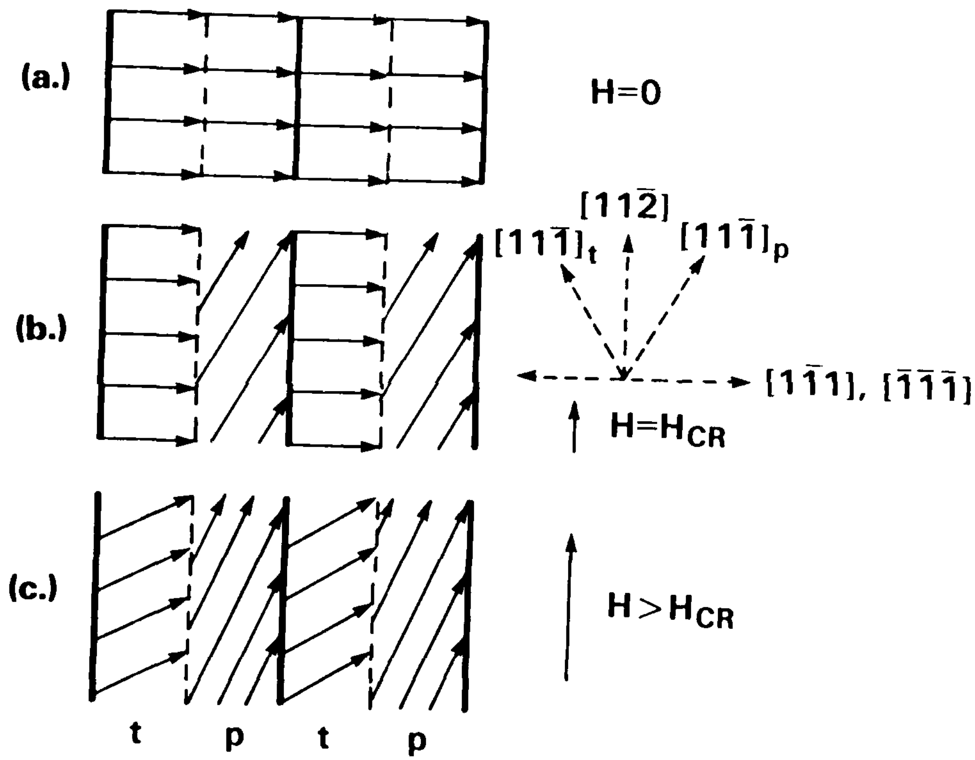


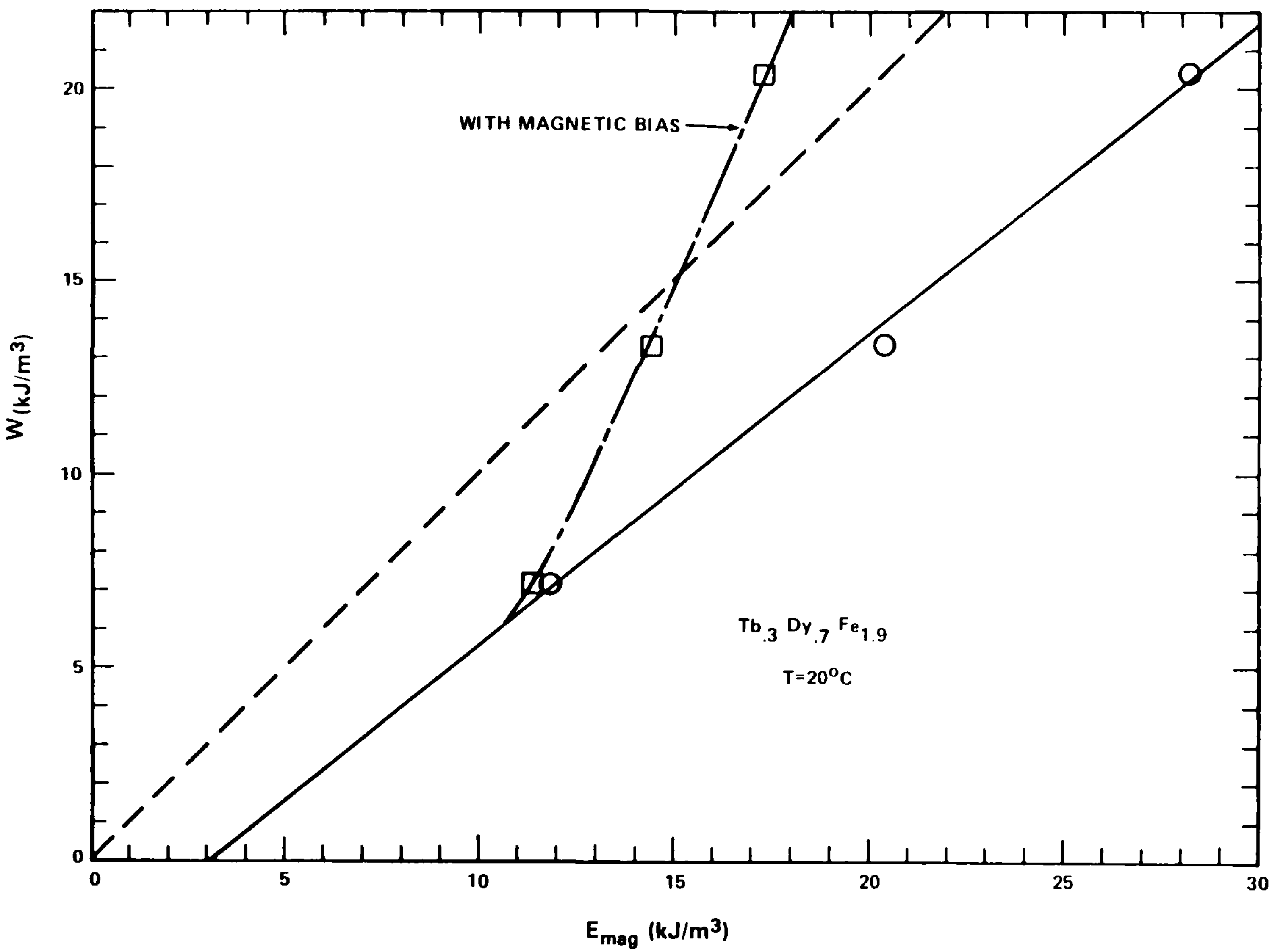
STRAIN (X 10 -3)
1.6
1.2
0.8
0.4
0
1.0
0.6
0.2
-0.2
-0.6
-1.0
MAGNETIZATION (TESLA)
Tb.3Dy.7Fe1.9
13.3 MPa
-50°C
0°C
20°C
80°C
-2000
-1000
0
1000
2000
Field (Oe)


Tb.3 Dy.7 Fe1.9
T=20°C
7.6 MPa
18.9 MPa
0 MPa
STRAIN (X 10−3)
1.6
1.2
0.8
0.4
0
2000
1000
0
1000
2000
FIELD (Oe)



$Tb_{.3}Dy_{.7}Fe_{1.9}$
7.6 MPa
2000 Oe
1000 Oe
500 Oe
250 Oe
18.9 MPa
$\frac{\Delta\lambda}{\Delta T} = 5.6 \times 10^{-6}/^{\circ}C$
2000 Oe
1000 Oe
500 Oe
250 Oe
MAGNETOSTRICTION (X 10^{-3})
T (^{o}C)


(a.)
H=0
(b.)
[11$\bar{2}$]
[11$\bar{1}$]$_t$
[11$\bar{1}$]$_p$
[1$\bar{1}$1], [$\bar{1}\bar{1}\bar{1}$]
H=H$_{CR}$
(c.)
H>H$_{CR}$
t
p
t
p


WITH MAGNETIC BIAS
$Tb_{.3}Dy_{.7}Fe_{1.9}$
T=20°C
$W(kJ/m^3)$
E_{mag} (kJ/m^3)
0
5
10
15
20
25
30

OPTICAL OBSERVATION OF CLOSURE DOMAINS IN TERFENOL-D SINGLE CRYSTALS

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ABSTRACT

Optical differential interference contrast microscopy has been employed to observe the topological features in surfaces of single crystal Terfenol-D ($Tb_{.27}Dy_{.73}Fe_{1.95}$) which arise from macroscopic lattice tilts due to the magnetostrictive strain between neighbouring magnetic domains at the surface. The domain configurations observed can all be interpreted as being composed of low energy 71° and 109° walls which have components of magnetization normal to the surface. Domain widths of the order of 2 µm can readily be resolved. Observations from surfaces polished parallel to (110) and (112) are presented as a function of temperature, between 250 K and 350 K, and as a function of magnetic field applied parallel to the specimen surface.

INTRODUCTION

The cubic Laves phase compound Terfenol-D ($Tb_{.27}Dy_{.73}Fe_{1.95}$), developed for transducer and actuator applications, exhibits a huge linear magnetostriction (1600 ppm) with compensated magnetocrystalline anisotropy near room temperature[1]. The highly anisotropic magnetostriction, with $\lambda_{111} >> \lambda_{100}$, results in significant internal rhombohedral strain within domains when the magnetization is along the easy ⟨111⟩ directions at room temperature and above. Measurements of the field dependence of magnetostrictve strain, and magnetization, from single crystal and grain oriented samples have been interpreted in terms of initial 180° domain wall motion followed by magnetization rotation[2,3]. A number of techniques have been previously employed to observe domain structures in both polycrystalline and single crystal Terfenol in order to investigate the magnetization process. These include the use of X-ray topography[4,5], SEM surface replication[6] and optical microscopy[7].

This paper presents observations of domain structures revealed by optical differential interference contrast microscopy (DIC) from single crystal Terfenol surfaces. The effects of specimen temperature and applied magnetic field are discussed and the configurations interpreted in terms of surface closure structures formed by domains possessing magnetization directions inclined to the surface.

EXPERIMENTAL

Observations were made on specimens cut from a single crystal of Terfenol-D which had been prepared by the Bridgman technique using a flat bottom BN crucible. The preferred growth axis of the crystal was [112] and the growth rate used was below 30 mm/hr. The slightly eutectic-rich alloy forms via a dendritic solidification front resulting in an interconnecting skeleton network of rare earth-rich micro-constituent between the rare earth Fe_2 dendrites[3,8]. Specimens of dimensions 5 mm by 5 mm by 1 mm thickness were cut from the crystal boule with their large area faces within 0.5 degree to the (112) and (110). The surfaces were polished to 0.25 μm diamond finish and then lapped with Syton. Low temperature observations were carried out with the specimen under vacuum using heating and cooling rates of approximately 1K/min. All specimens were demagnetized prior to observation.

RESULTS AND DISCUSSION

Typical examples of the complex contrast obtained from Terfenol surfaces are shown in fig.1 illustrating the domain changes occuring as a function of magnetic field applied parallel to the <100> direction in the (011) surface. With reference to the stereographic projection of the (011) plane given in fig.2, domain traces can be identified along the <100>, <011>, <111> and <211> directions in the plane. The observed contrasts can be interpreted as arising from macroscopic surface tilts created by the magnetostrictive strain within neighbouring domains intersecting the surface of the crystal.

An example of such tilting is shown in fig.3 for the case of (100)109° walls where the lattice distortions in neighbouring domains with magnetization in the $[\bar{1}\bar{1}\bar{1}]$ and [111] directions create tilting in the (011) surface[9] plane. The tilt angle, α, may be expressed as $\sqrt{2}\lambda_{111}$ such that for domains of width 10 μm, height undulations of the order of 10 nm may be expected which are readily detectable by DIC microscopy. Contrast from such (100)109° walls will produce the wall traces parallel to the <011> in fig.1. All other observed traces can be similarly interpreted in terms of walls having at least one component normal to the specimen surface.[4]

Little change in the domain configuration occured upon field application until the field exceeded 400 Oe. On increasing the field to 800 Oe, the narrow walls with <011> traces moved rapidly in the <100> direction leaving isolated <100>-traces which are associated with (010)109° walls having one component of magnetization in the <111> directions in the surface plane. The contrast boundary along the <211> direction, indicated in fig.1 was observed to move rapidly in a direction normal to the <211>, severely reducing the <011> - trace wall contrast in its path.

It should be noted that no contrast would be expected using DIC microscopy from 180° walls or from walls formed between domains with magnetizations coplanar with the specimen surface. The small areas exhibiting no contrast in fig.1 may well contain walls of either of these configurations.

Observations of the influence of applied field on the domain contrasts exhibited from the (112) surface are shown in fig.4. With reference to the stereographic projection of the (112) plane given in fig.5, wall traces in the surface <110>, <111>, <021> and <201> directions can be identified in fig.4a in the demagnetized state. Examples of wall types associated with the wall traces seen on the (112) plane are given in Table 1 and are all consistent in being associated with domains having at least one component out of the surface plane.

Table 1

Examples of wall-types seen on the (112) plane

Wall trace	Wall-type	Magnetization directions
<111>	(110)109°	$1\bar{1}1$ and $\bar{1}11$
<110>	(001)109° and (110)71°	111 and $11\bar{1}$
<021>	(100)109°	111 and $\bar{1}11$
<201>	(010)109°	111 and $1\bar{1}1$

Upon application of a field parallel to the <111> easy axis in the specimen surface, little change in configuration is observed until the field exceeds about 200 Oe. The majority of the wall contrast disappears in fields approaching 800 Oe as can be seen in fig. 4b when the crystal can be assumed to be nearly saturated. A curious observation, shown in figs. 4b and 4c, is the result of applying a 400 Oe field parallel to the <110> direction in the (112) plane. The <111> traces, created by the high, out of plane symmetry (110)109° walls, are found to rotate about 5 degrees from the <111> upon rotation of the field about 10 degrees from the <110>

direction.

The effect of temperature on domain structures observed on the (112) plane is shown in fig.6. As the temperature is increased from 260K to 315K, a pronounced change in configuration is observed, the predominant feature being the reduction in the density of (110)109° walls having traces parallel to the <111>. This change can be explained by consideration of domain wall energies. As indicated in fig.7, such (110)109° walls will become energetically unfavourable at the higher temperature investigated. However, it is curious that such complex domain structures as shown in fig.6a, explicable in terms of assuming <111> easy axes, are observed at temperatures where previous data[1] would indicate a <100> easy axis anisotropy. The spin re-orientation temperature of this crystal is considered to be below 260K and recent evidence from X-ray diffraction experiments[3] would tend to support this conclusion.

CONCLUSIONS

Observations of suitably prepared surfaces of Terfenol-D by optical DIC microscopy reveal by a simple technique, and in real time, the complex nature of the surface domain structure. The geometry of all configurations investigated can be interpreted in terms of macroscopic lattice tilts at the crystal surface due to the magnetostrictive strain between neighbouring domains having a mean inclination of magnetization normal to the surface. The explanation of the persistence of domain structure in fields where bulk saturation is nearly complete, and of the detailed magnetization process is the subject of continuing study.

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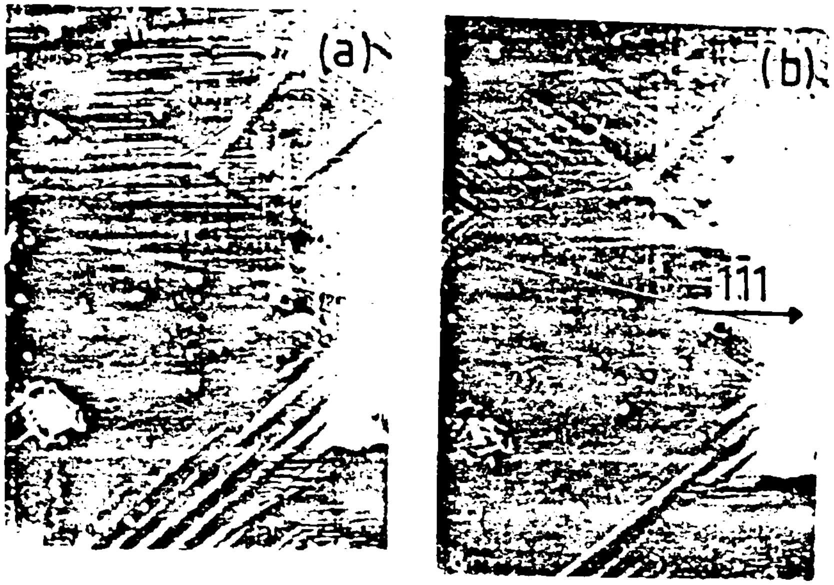


Fig.6: Optical micrographs of domain structure on (112) surface in zero field at a) 260K and b) 315K.

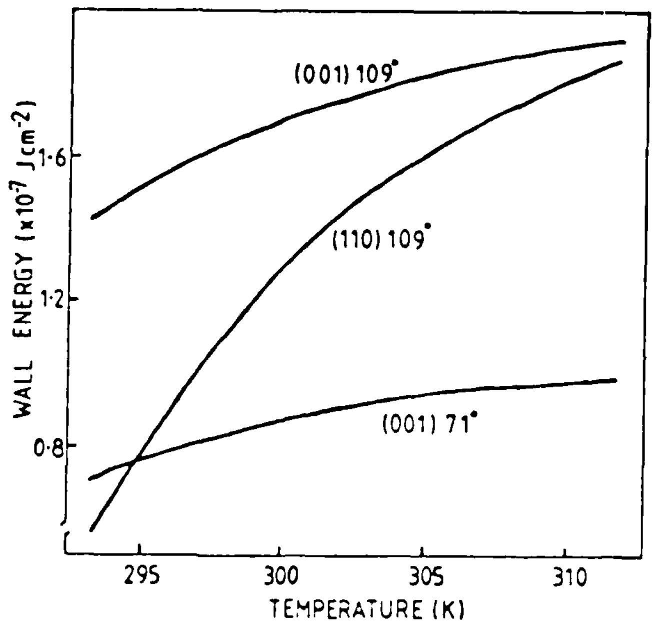


Fig.7: Energy of the indicated domain walls as a function of temperature (after ref.4).

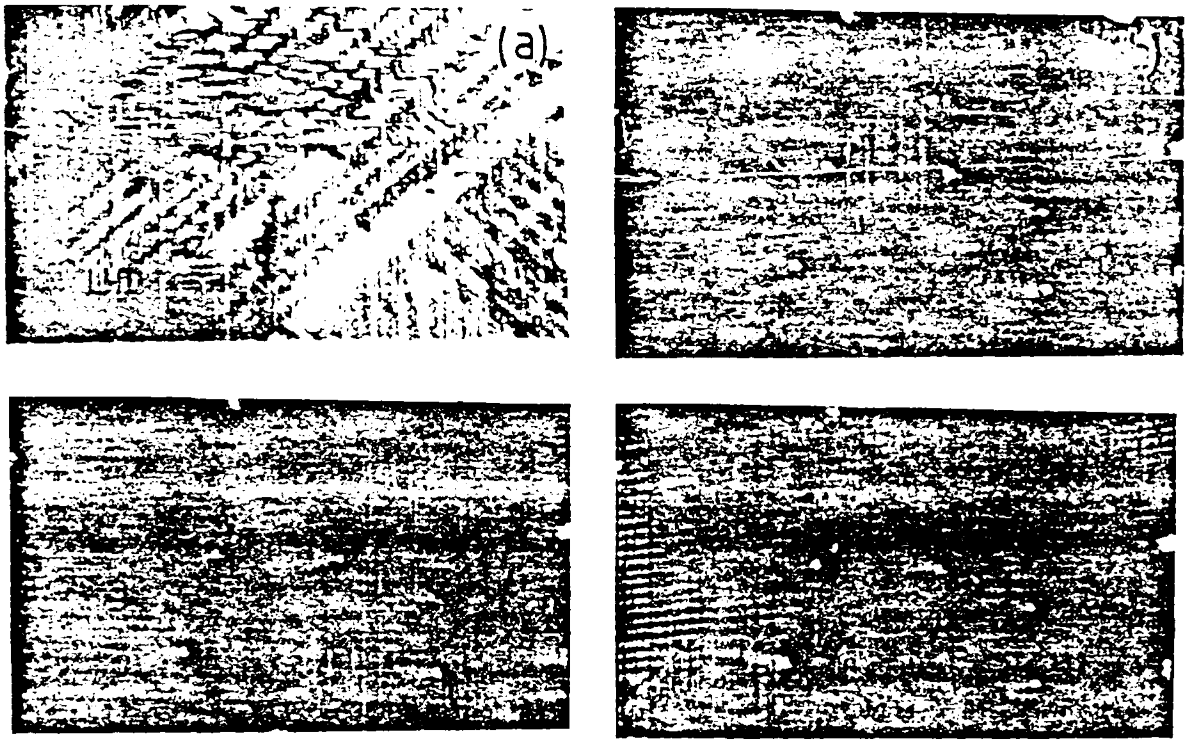
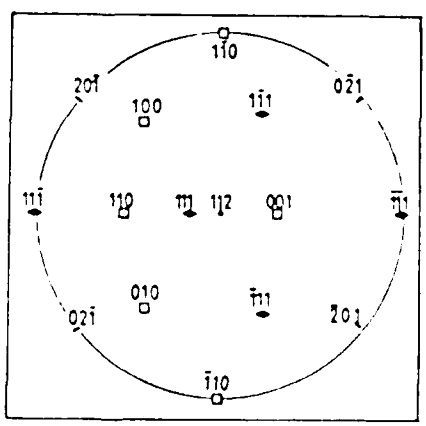


Fig.4: Optical micrographs of domain structures on (112) surface at 290K in a) zero field, b) 800 Oe parallel to <111>, c) and d) 400 Oe within 5° of <110> as indicated.



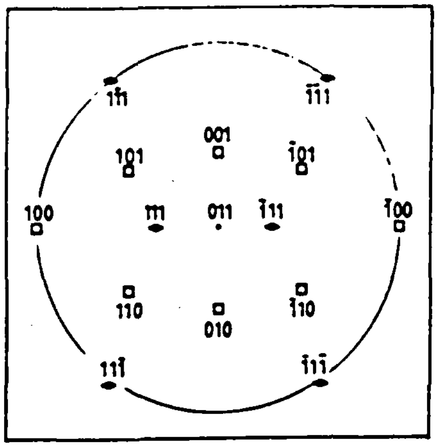


Fig.2: Stereographic projection of (011) plane showing easy axes ◆ and wall plane normals ◘ .

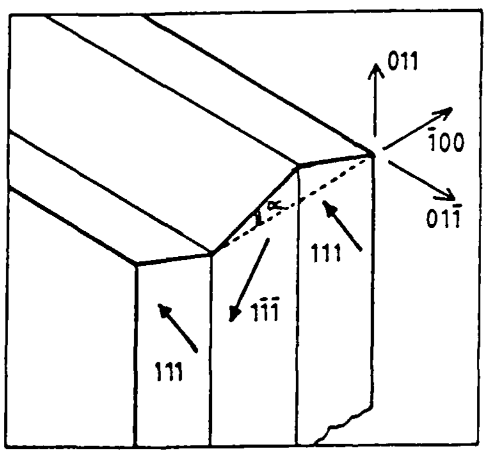


Fig.3: Lattice tilts (∝) on (011) surface caused by magnetostrictive strain within neighbouring (100)109° domains.

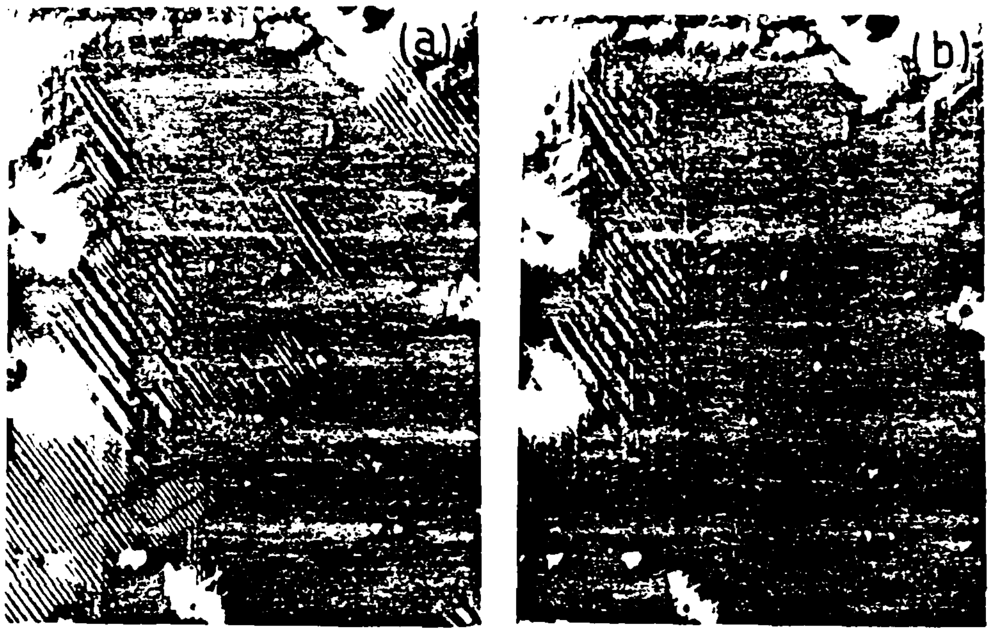


Fig.1: Optical micrographs of domain structure on (011) surface at 290K with a field applied parallel to <100> of a) 400 Oe and b) 800 Oe.

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