Ferroelastic behavior and the monoclinic-to-orthorhombic phase transition in MP_5O_{14} (M = La–Tb)

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(Received 14 October 1974)

The crystallographic transitions of the pseudo-orthorhombic monoclinic I rare-earth pentaphosphates are examined. The atom positions allow a smooth transition into strict orthorhombic symmetry and a second-order monoclinic-to-orthorhombic phase change is observed for LaP_5O_{14}, PrP_5O_{14}, NdP_5O_{14}, TbP_5O_{14}, and $Nd_xLa_{1-x}P_5O_{14}$. The monoclinic distortion angle δ ($\delta = \beta - 90° \approx 0.5°$) is determined using a He-Ne laser to measure the divergence between the two reflections obtained from the top surface of a twinned crystal. The transitions are monitored by observing the change in δ with temperature. The transition temperatures increase linearly with the decrease in the ionic radius of the rare-earth ion from 118 °C for LaP_5O_{14} to 174 °C for TbP_5O_{14}. The two types of twin boundary possible in the monoclinic I crystals are observed by several microscopic techniques and both are found to be very mobile via ferroelastic reorganization. A shear stress of only 14 ± 3 kN/m^2 is required to induce motion of the twin boundary perpendicular to [001]. This stress is two orders of magnitude lower than values that have been reported to induce ferroelastic reorganization in other crystals. The twin boundaries can be readily introduced mechanically into, and removed from, the crystals using very small forces.

I. INTRODUCTION

The pentaphosphates (MP_5O_{14}) of the rare-earth metals,[1] yttrium,[1] and bismuth[2] exhibit three structure types (designated I, II, and III by Beucher[1]). Some compounds appear in more than one type. The crystals formed by the larger ions (La–Tb) have a monoclinic cell with space group $P2_1/c$ (type I), and the smaller ions (Tb–Lu, Y, Bi) give a different monoclinic cell with space group $C2/c$ (type II). The pentaphosphates of Dy, Ho, Er, and Y also form with an orthorhombic cell (space group $Pnma$, type III). The pentaphosphates have recently attracted attention because of their unusual fluorescence properties and NdP_5O_{14} has been studied in some detail in this regard.[3,4] The deviation of type-I crystals from orthorhombic symmetry is small and the investigation of some properties which are a consequence of the pseudo-orthorhombic character of these crystals is the subject of the present investigation.

Pseudosymmetry suggests the possible presence of a phase transition at higher temperature, and it is of interest to determine the nature of the transition (first or second order) and to study the effect of the radius of the rare-earth ion on the transition temperature. Also, such pseudosymmetry may permit the existence of ferroelastic properties.[5] In NdP_5O_{14} two types of twin planes have already been reported,[6] suggesting that type-I monoclinic crystals may exhibit ferroelastic behavior via twin-boundary motion. Whether these properties will be observed in any particular case depends, of course, on the details of the crystal structure.

In the present paper we report the optical observation of ferroelastic behavior (mechanically induced twin-boundary motion) along the two possible crystal directions and measure the shear stress necessary to induce ferroelastic reorganization for one direction of switching. This stress is two orders of magnitude lower than values that have been reported to induce ferroelastic reorganization in other crystals. The twin boundaries can be readily introduced mechanically into, and removed from, the crystals using very small forces. The atomic arrangement of type-I crystals is such that a second-order monoclinic-to-orthorhombic phase transition is possible at higher temperature with a particular dependence on the rare-earth ionic radius. We measure optically the variation of the monoclinic distortion with temperature for La, Pr, Nd, Tb and $Nd_xLa_{1-x}P_5O_{14}$, and demonstrate the presence of the expected phase transition.

II. CRYSTAL-STRUCTURE DISCUSSION

The crystal structures of all three types of the rare-earth pentaphosphates have been determined recently. The monoclinic I[7,8] and orthorhombic III[9] materials have very similar structures, consisting essentially of infinite ribbons of repeating $P_{10}O_{28}^{6-}$ units, the ribbons being linked to each other only through electrostatic rare-earth–oxygen bonds. The ribbons consist of two parallel $(PO_3^-)_\infty$ chains linked at every second tetrahedron by a PO_2^+ group. (Fig. 1). The principal difference between the two structure types lies in the relative orientations of the cross-linking units.[7] The ribbons can be alternatively described as $P_8O_{24}^{8-}$ rings joined by PO_2^+
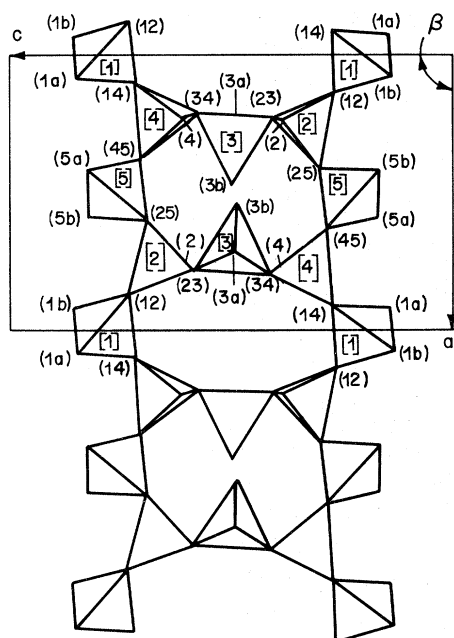
FIG. 1. Arrangement of PO_4 tetrahedra in the $(P_{10}O_{28}^{6-})_\infty$ ribbons occurring in the monoclinic I rare-earth pentaphosphates MP_5O_{14}. (M = La–Tb). The labeling is after Ref. 7, where the O's are indicated by ( ) and the P's by [ ].

groups at four corners. In the monoclinic II structure [10,11] these $P_8O_{24}^{8-}$ rings are linked in a different manner so that ribbons are not present.

We find x-ray precession photographs of monoclinic I NdP$_5$O$_{14}$ crystals (Mo $K\alpha$ radiation) to show pseudo-orthorhombic symmetry characteristic of space group $Pncm$, in agreement with earlier observations. [3,7] This pseudosymmetry is apparent in the structure determinations. Using the nomenclature of Ref. 7, the O(1a) and O(1b) atoms (Fig. 1), for example, are related by the pseudodiagonal glide plane along the [100] axis and the pseudo-mirror-plane perpendicular to the [001] axis, and in all cases only very small atomic shifts (< 0.05 nm) are necessary to produce true orthorhombic symmetry. Moreover, if we assume that the small deviation δ ($\sim \frac{1}{2}°$) of the monoclinic angle β from 90° is an indicator of the distortion at the atomic level, then the lattice parameters of the monoclinic I pentaphosphates given by Beucher[1] show that this distortion increases in the direction La → Tb, i.e., with decreasing ionic radius. A larger trivalent ion than La^{3+}, if available, should therefore stabilize the orthorhombic structure; alternatively, we expect a smooth transition on heating to strict orthorhombic symmetry (δ = 0°) with the transition temperature increasing from La to Tb. The space
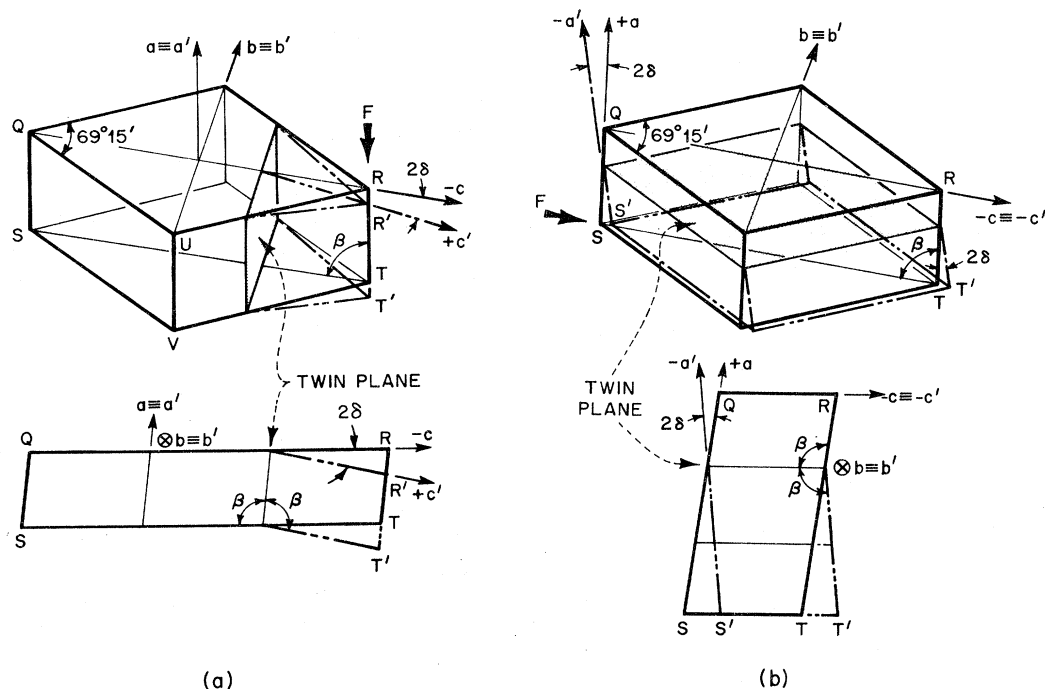

(a)   (b)

FIG. 2. Schematic representation of twinned diamond-shaped crystals of the monoclinic I rare-earth pentaphosphates MP_5O_{14} (M = La–Tb) showing the two possible types of twin. Force (F) is applied to induce ferroelastic reorganization. (a) Crystal containing an a-type twin with reversal of the crystallographic c axis at the twin boundary (+c → −c). (b) Crystal containing a b-type twin with reversal of the crystallographic a axis at the twin boundary (+a → −a).

group of this high-temperature orthorhombic I phase (*Pncm* if the axes corresponding to the monoclinic cell are maintained) is, of course, different from that of the orthorhombic III pentaphosphates (*Pnma*) because of the different orientations of the cross-linking tetrahedra already mentioned. Such a transition was first suggested[2] in the case of BiP_5O_{14} in the light of the many twin planes observed in these crystals.

A crystal shows ferroelastic behavior if, by applying a force, a mutual exchange of two lattice parameters may be induced.[5,12] Several examples, where two axes of very similar length are interchanged upon application of uniaxial compressive stress along the longer, are known or predicted.[12,13] In the monoclinic I pentaphosphates the angle β ($\sim 90.5°$) should be able to be ferroelastically switched to $(180° - \beta)$ upon application of a suitable shear stress. There are two directions in which this may be accomplished and the twin planes corresponding to the two directions of switching are shown in Fig. 2. The *a* twin [Fig. 2(a)] is associated with a change in direction of the crystallographic *c* axis and the *b* twin [Fig. 2(b)] with a change in direction of the crystallographic *a* axis. For NdP_5O_{14}, for which the atom positions are known,[7,8] the atomic movements within the unit cell needed to switch from one cell orientation to another (Fig. 2) are < 0.1 nm. In the transition, pseudoequivalent atoms such as $O(1a)$ and $O(1b)$[7] are interchanged. The basic positional relation between two such atoms in the unit cell is

$$(x_1, y_1, z_1) = (x_2, y_2, 1 - z_2) + \Delta , \qquad (1)$$

where Δ is a small distance. In the transformation the Nd atoms transform into themselves through about 0.01 and 0.02 nm; P(1), P(3), and P(5) (Fig. 1) transform into themselves, and P(2) into P(4), through distances up to about 0.04 nm; $O(3a)$ and $O(3b)$ transform into themselves and the other oxygens into pseudoequivalent independent atoms through up to about 0.055 nm. In such a ferroelastic reorganization there is no coupling to other properties except the refractive indices of the crystal.

To our knowledge only one other monoclinic crystal, Eu_2SiO_4, has been reported[14] to show a transformation of this type, but for that crystal, although twin boundaries were introduced mechanically, no measurement of the required shear stress was reported. Eu_2SiO_4 (space group $P2_1/c$, $\beta = 92.6°$ at room temperature) transforms[14] to an orthorhombic cell (space group $Pmcn$) at 165 °C so that the point-group transformation ($\frac{2}{m}$ to mmm) is the same as for MP_5O_{14}.

III. MICROSCOPIC OBSERVATION OF TWINNING

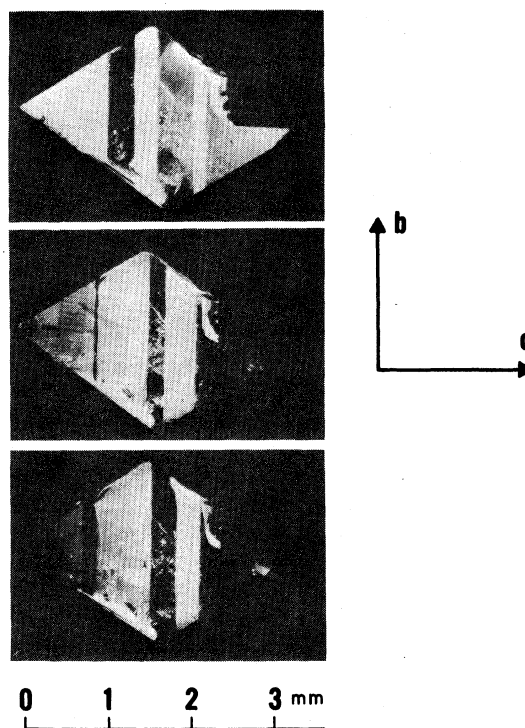We have reported[6] previously the presence of the expected (Fig. 2) twins in NdP_5O_{14}. The *a*-twin

FIG. 3. Specular reflection from the (100) surface of an NdP_5O_{14} crystal showing the presence of several *a*-type twin boundaries. The twin planes were mechanically moved between exposures.

boundary is more frequently found in as-grown crystals. For diamond-shaped plates which are frequently observed,[6] and where the crystallographic *a* axis is perpendicular to the plane of the plates, crystal surfaces separated by an *a*-twin plane are tilted to one another at an angle of $\sim 1°$ and are easily revealed by observing specular reflection from the diamond surface (Fig. 3). Alternate areas of similar orientation are seen as light or dark regions. We have also observed these twins to be induced mechanically in the course of polishing the (100) face of an initially twin-free crystal. This indicated the occurrence of ferroelastic behavior, and subsequent qualitative experiments with NdP_5O_{14} and other rare-earth pentaphosphates (La, Pr, Tb), as well as with $Nd_{1-x}La_xP_5O_{14}$ mixed crystals, revealed that the twin boundaries could be induced, and moved across and off the crystals with very little applied stress—with a toothpick, for example.[15] The ability to ferroelastically remove such twins is important in the use of rare-earth pentaphosphates as laser crystals.

In Fig. 3 we show three consecutive photographs of the same NdP_5O_{14} crystal, where the twin planes have been mechanically shifted between exposures by pressing gently with a toothpick on the top sur-
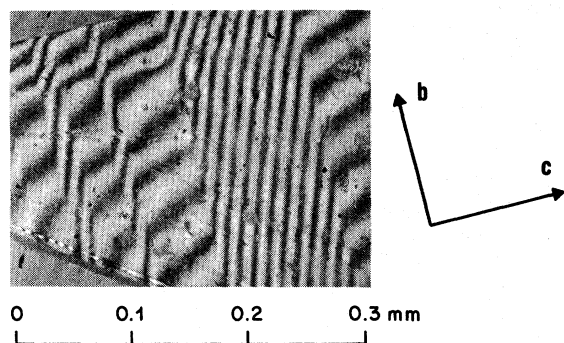
FIG. 4. Reflection interference pattern from the (100) surface of an a-type-twinned NdP_5O_{14} crystal showing the equal inclination angles of alternate regions separated by two twin boundaries.

face. In all three photographs we show several twin planes; by stroking the toothpick along the surface we could easily remove them all. The photograph shown in Fig. 3 was taken using a long-working-distance microscope. Using an interference reflection microscope and focusing on the top surface of a diamond-shaped NdP_5O_{14} crystal, the number and direction of the fringes (Fig. 4) also reveals the different surface inclination on either side of the twin planes, and the similar inclination of alternate regions separated by two twin boundaries. We did not attempt to ascertain the minimum thickness of the twinned regions that could be obtained, but we note that the narrowest domain in

Fig. 4 is only ~ 10 μm wide. The observed width of the boundary regions (2 μm) is resolution limited by the microscope, but it is likely that the boundary extends over at least several unit cells. Using a transmission interference microscope, we verified that the optical thickness is the same in different twin areas.

The presence of both types of twin boundary was also revealed under conoscopic observation, by observing the rotation of the optic figures and, in some directions of observation, by observing a different orientation for extinction between crossed polarizers for the regions of crystal separated by a twin boundary. The behavior described is common to all the compositions investigated, and the crystals shown in this section were chosen for investigation because of their availability in the desired form rather than for their composition.

To examine the characteristics of the a-twin plane, one such twin was mechanically introduced into a diamond-shaped NdP_5O_{14} crystal which had been cleaved on two sides (the plane of easy cleavage is perpendicular to the crystallographic b axis[4,6]). Placing the crystal between crossed polarizers and viewing along [100], there is no significant contrast between the two regions [Fig. 5(a)], but viewing along [010] the two regions show different extinction orientations and are clearly distinguished [Fig. 5(b)]. We also examined between crossed polarizers the a-twin plane in a diamond-shaped $Nd_{0.5}La_{0.5}P_5O_{14}$ crystal viewed along [011]. The twin boundary is at an angle to the direction

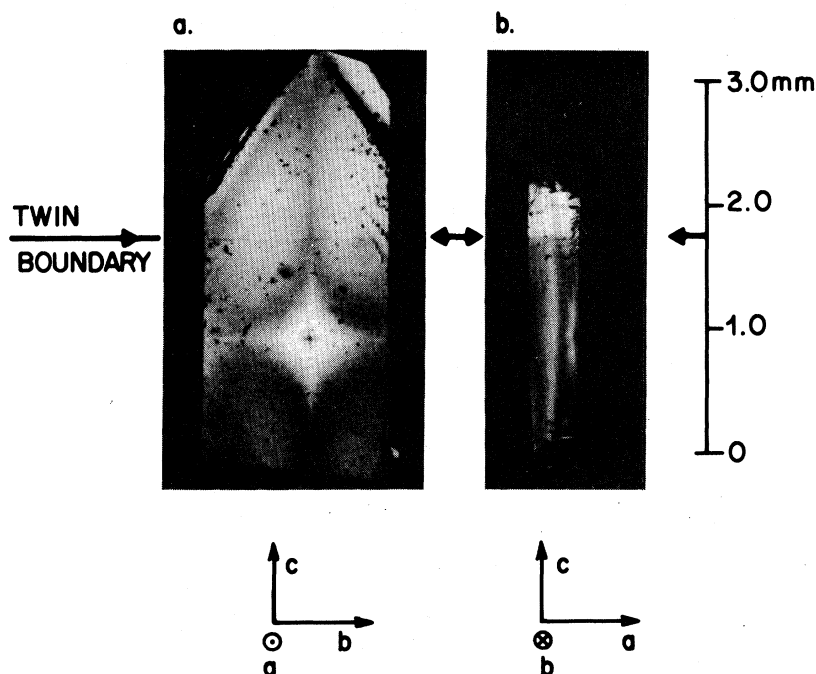

FIG. 5. Transmission of light for a cleaved NdP_5O_{14} crystal containing one a-type twin boundary observed between crossed polarizers. (a) Viewing along [100] showing no significant contrast between the two regions. (b) Viewing along [010] showing the different extinction orientations of the two regions.
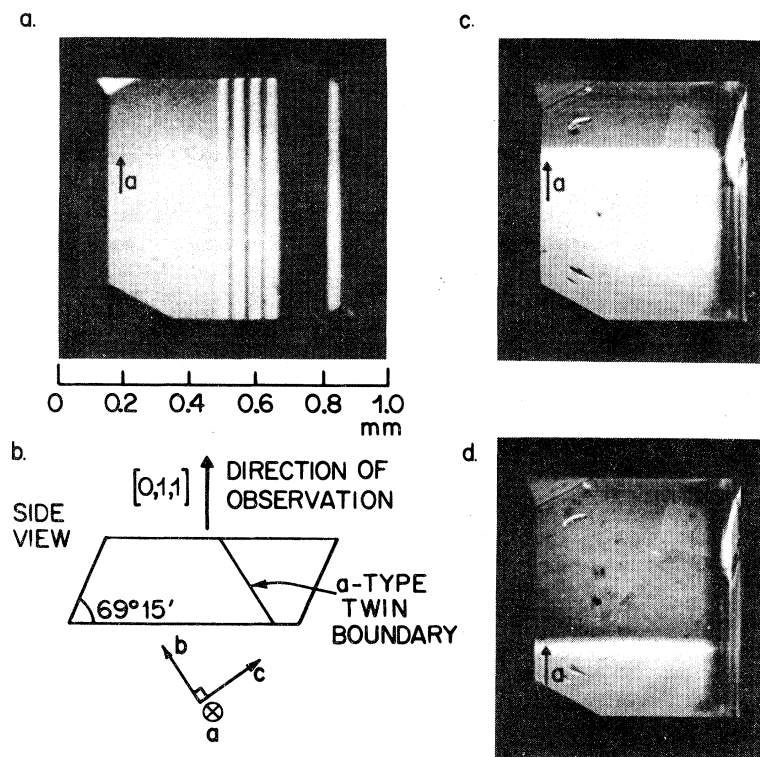
a.

O   0.2   0.4   0.6   0.8   1.0
                                mm

b.

SIDE
VIEW

$[0,1,1]$ ↑ DIRECTION OF OBSERVATION

69°15′

b

c

⊗
a

a-TYPE TWIN BOUNDARY

c.

d.

FIG. 6. Transmission of light for two $Nd_{0.5}La_{0.5}P_5O_{14}$ crystals containing one a-type and one b-type twin, respectively, observed normal to the (011) plane between crossed polarizers. (a) Crystal with a-type twin showing a striped pattern revealing the smooth change in optical thickness across the twinned region. (b) Schematic orientation of the crystals. The a-type twin observed in Fig. 6(a) is also shown. The b-type twins in Figs. 6(c) and 6(d) would be in the plane of the paper. (c), (d) Crystal with b-type twin showing the different extinction orientations of the two regions. The twin plane was moved mechanically between exposures.

of the light [Fig. 6(b)], and in this case the crystal regions distant from the twin plane are distinguished by their extinction orientations but the region of the crystal containing the twin plane shows a striped pattern [Fig. 6(a)] revealing the smooth change in optical thickness across the crystal. On moving the twin by applied stress the striped pattern also moves, which clearly demonstrates that the phenomenon under observation is a bulk and not a surface effect.

In crystals with b twins, the twin boundary is also clearly seen between cross polarizers for observation along [011]. Figures 6(c) and 6(d) show a b-twin plane in a $Nd_{0.5}La_{0.5}P_5O_{14}$ crystal which was present at the center of the crystal after growth. This twin is also very mobile via ferroelastic reorganization, and the figures show the twin after movement away from the center by gentle application of pressure to the crystal surface. This twin may also be removed entirely from the crystal.

It was found that it was generally much easier to introduce the a twin rather than the b twin into an initially twin-free crystal. The presence of and the ease of ferroelastic reorganization of the crystal via b-twin boundaries show, however, that a pseudo-mirror-plane is not essential for twinning as has been suggested.[16] Unlike the a-twin plane, the b-twin boundary is not a pseudo-mirror-plane

in the monoclinic crystal.

The angle of inclination of the two surfaces of a twinned crystal may be readily measured by reflection of a parallel light beam at nearly normal incidence. The angular divergence of the reflected beams from the two surfaces is 4δ and the values of δ determined in this way agreed with those obtained from x-ray lattice-parameter measurements.[1]

IV. MONOCLINIC-TO-ORTHORHOMBIC PHASE TRANSITION

As mentioned above, δ ($= \beta - 90°$) can be readily determined in a crystal with twin boundaries by measuring the angular divergence of light reflected from the surfaces of different orientation. Using this technique we measured the variation of δ with temperature from -170 to $+200$ °C for LaP_5O_{14}, PrP_5O_{14}, NdP_5O_{14}, and TbP_5O_{14}, and for mixed crystals in the $Nd_xLa_{1-x}P_5O_{14}$ system, for reflection from the (100) surface of diamond-shaped crystals using 632.8-nm light from a helium-neon laser. For the measurements below room temperature the samples were placed in a Dewar on a cold finger. From room temperature to 200° C a temperature-controlled hotplate was used. The temperature was measured by inserting a copper constantan thermocouple into the support for the crystals. Care was taken to ensure that the mea-

suring thermocouple was at the same temperature as the crystal and that measurements were taken at equilibrium.

The variation of δ with temperature is shown in Fig. 7 for the four pure pentaphosphates. As expected, we find that δ decreases with increasing temperature, and the data indicate a transition to the orthorhombic I unit cell (δ = 0) for all the crystals, with T_c increasing, as predicted above, from LaP_5O_{14} to TbP_5O_{14}. The transition temperatures are (118 ± 2)°C for LaP_5O_{14}, (140 ± 2)°C for PrP_5O_{14}, (146 ± 2)°C for NdP_5O_{14}, and (174 ± 2)°C for TbP_5O_{14}. The Nd–La mixed crystals had transition temperatures between those observed for the end members [(126 ± 2)°C for $Nd_{0.1}La_{0.9}P_5O_{14}$ and (135 ± 2)°C for $Nd_{0.5}La_{0.5}P_5O_{14}$]. In Fig. 7 the angle δ is plotted on a square-law scale. The linear variation of $δ^2$ with temperature for T close to T_c is characteristic of a second-order transition.[17] This conclusion is supported by the fact that differential thermal analysis (DTA measurements) on powdered samples revealed no anomalies over the whole temperature range studied (–170 to 200°C) that would indicate any first-order character for the transition. Also, no abrupt changes in fluorescent properties were observed for $Nd_{1-x}La_xP_5O_{14}$ crystals in this temperature region.[18] The gradients of the $δ^2$-T curves are all similar in the straight-line region. No variation of T_c was observed for a repeated experiment on the same crystal or with different crystals of the same composition. Also, there was no indication of any hysteresis upon cooling from below T_c. By cooling samples from above
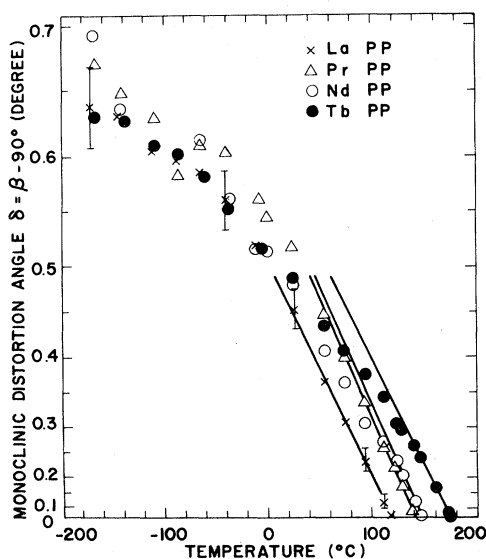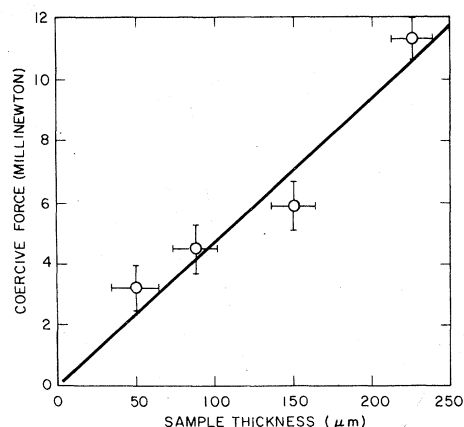

FIG. 8. Critical force necessary to induce ferroelastic reorganization in NdP_5O_{14} crystals vs crystal thickness.

the transition temperature the twinning was not always reintroduced spontaneously.

V. FERROELASTIC REORIENTATION

We have not analyzed in detail the motions of the phosphate tetrahedra during the ferroelastic reorganizations described above. As already mentioned, we observed that the a twin is apparently more easily introduced, although both types of twin are very mobile. We did not make a precise quantitative comparison between the mobilities of the two twins, but the shear stress required to initiate movement of the a twin was determined for a series of NdP_5O_{14} crystals of varying thickness. One end [QS in Fig. 2(a)] of a diamond-shaped crystal was fixed and a force F applied with a leaf spring at the other end close to R in the direction T.

The shear force equals F for all possible positions along the Q-R axis, but the shear stress is inversely proportional to the twin-plane area, and hence varies along Q-R. It is the shear stress which is responsible for twin-boundary movement. We observed that with increasing F a twin boundary moved from R toward the center of the crystal, and having passed the center moved rapidly to the clamped end Q. On reversing F the boundary moved monotonically with increasing force back from Q toward the center and then rapidly to R. Under these conditions we always moved the same boundary back and forth and did not create new ones by our applied force. This shows that the stress required to create a boundary at any position within the crystal is considerably higher than that required for boundary movement. We conclude that there is a higher potential barrier for the creation of a twin (or, as is often observed, a pair of twins) than for movement of an already-existing

FIG. 7. Dependence of the monoclinic distortion angle δ vs temperature for LaP_5O_{14}, PrP_5O_{14}, NdP_5O_{14}, and TbP_5O_{14}. δ is plotted on a square-law scale.

twin boundary, and this is consistent with the fact that any twinned crystal is in a state of higher free energy than an untwinned crystal.

We measured the force needed to move the boundary when it was close to the center of a crystal. This force was very reproducible as the crystal was flipped back and forth from one orientation to the other. All the crystals measured had a width b at the center of 3.5 ± 0.2 mm, but were of different thicknesses d, ranging from 50 to 250 μm. The force needed to create the critical shear stress should be proportional to the crystal thickness, and this was verified by the measurements plotted in Fig. 8. The critical shear stress is given by

$$\sigma_c = F/bd. \qquad (2)$$

From the slope of Fig. 8 we find $\delta_c = 14 \pm 3$ kN/m^2.

Few quantitative measurements of this kind have been reported for ferroelastic crystals and the shear stress required to switch Eu_2SiO_4 was not given.[14] A few data are available for orthorhombic crystals where application of compressive stress along the longer of two very similar principal axes interchanges the two axes. For β-$Gd_2(MoO_4)_3$, $LaFeO_3$, and $Mg_3B_7O_{13}Cl$ coercive stresses of 1 MNm^{-2} (Ref. 19), 300 MNm^{-2} (Ref. 20), and 120 MNm^{-2} (Ref. 21), respectively, have been reported. Direct comparison with our measurements is not possible. However, we may consider our crystals to be subdivided into cubes with one face in the plane $QSRT$ [Fig. 2(a)] but with the edges oriented at an angle of 45° to the a and c crystal axes. For such cubes the applied shear stress is represented as two uniaxial stresses on the surfaces normal to $QSRT$. This geometry permits a comparison with the published values for the orthorhombic crystals. We note that for the twin-boundary movement in NdP_5O_{14} stress two orders of magnitude smaller is required than for the ferroelastic reorganization in β-$Gd_2(MoO_4)_3$. These results confirm the qualitative observation already mentioned of the great facility with which the ferroelastic reorientation may be induced in the pentaphosphates.

VI. DISCUSSION

The consequences of the small deviation ($\delta \approx 0.5°$) of MP_5O_{14} (M = La–Tb) from orthorhombic symmetry at room temperature have been examined. It was shown that the atom positions allow a smooth transition into strict orthorhombic symmetry and this explains why twin planes of one or the other of the two possible types are often observed in as-grown crystals. The observation of both twin planes by several microscopic techniques was discussed. The transition to orthorhombic symmetry was shown to take place on heating, and the decrease of δ was monitored by observing the angle 4δ between light beams reflected from the surface

of twinned crystals. From the linear dependence of δ^2 versus temperature the monoclinic-I-to-orthorhombic-I transition was presumed to be of second order and this conclusion was supported by DTA measurements. The transition temperatures observed (from 118 °C for LaP_5O_{14} to 174 °C for TbP_5O_{14}) show an approximately linear variation with the ionic radius of the rare-earth ion,[22] and for mixed crystals T_c can be estimated by linear interpolation. Extrapolation indicates a T_c of ~ 200 °C for LuP_5O_{14}, which contains the smallest rare-earth ion, if this compound were to be found with the monoclinic I structure (Tb is the heaviest rare-earth reported to form in the monoclinic I structure[1]).

Under the usual conditions of preparation,[6] which involve a sudden cooling from 500 °C or higher to 100 °C, TbP_5O_{14} is found mainly in the monoclinic I structure, but also occasionally in the monoclinic II structure; the pentaphosphates of Dy, Ho, Er, and Y are found in both the monoclinic II and orthorhombic III structures. The preparation of these latter compounds with two different structures suggests that first-order phase transitions may be expected to occur on heating the pentaphosphates of the heavier rare earths in the probable order monoclinic II→ orthorhombic III→ orthorhombic I. In accordance with this expectation we have found that ErP_5O_{14} with the monoclinic II structure at room temperature shows an endotherm in DTA at 280 °C, which is no doubt associated with a transformation to one of the orthorhombic structures.

It seems likely, that by choosing an appropriate average ionic radius, mixed crystals containing any of the rare-earth ions may be tailored to yield any of the three structure types. The study, for example, of the effect of structure on rare-earth fluorescence lifetimes and linewidths may be interesting. This suggestion is supported by the results of Krühler et al.,[23] who find the $Y_xNd_{1-x}P_5O_{14}$ system to have the monoclinic I structure for x up to ~ 0.85 and the orthorhombic III structure for higher yttrium contents. The average ionic radius for x = 0.85 is 0.103 nm, and for the unmixed crystals only rare earths having ionic radii $\lesssim$ 0.103 nm form the monoclinic I structure at room temperature.[1]

At room temperature both twin boundaries occurring in the monoclinic I pentaphosphates have been shown to be very mobile. Twinned crystals can be ferroelastically detwinned with ease and the a twin especially [Fig. 2(a)] can be readily induced mechanically. The shear stress needed to induce a-twin motion was determined to be 14 ± 3 kN/m^2. This stress is two orders of magnitude smaller than the values reported elsewhere to introduce ferroelastic transitions in other materials by application of uniaxial stress, and is perhaps a con-

sequence of the unusual one-dimensional structure of the $(P_{10}O_{28}{}^{6-})_\infty$ pentaphosphate ribbons which are linked only by electrostatic rare-earth–oxygen bonds, although conceivably many other pseudo-orthorhombic crystals with $\beta \sim 90°$ might show similar behavior. The ablilty to be able to ferroelastically detwin crystals is of importance in achieving optically undistorted samples for laser action. Conversely, the possibility of introducing twins into a laser material in an ordered and regular fashion also raises interesting optical possibilities, e.g., to form waveguides.

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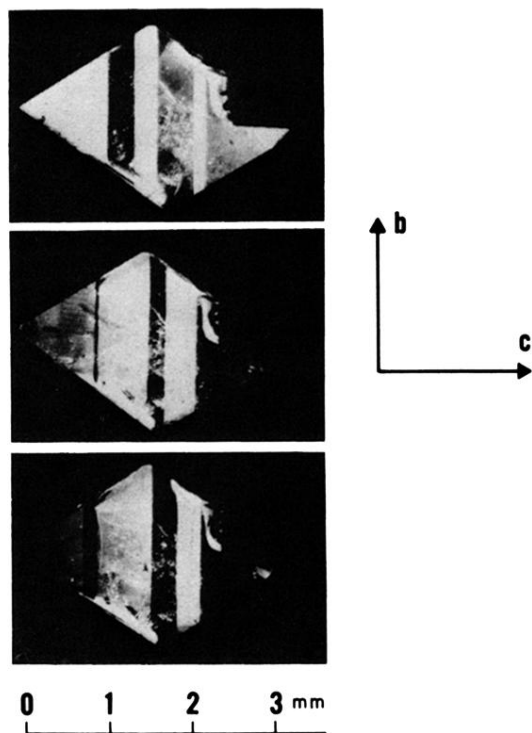[23]W. W. Krühler, J. P. Jeser, and H. G. Danielmeyer, Appl. Phys. 2, 329 (1973).

FIG. 3. Specular reflection from the (100) surface of an NdP$_5$O$_{14}$ crystal showing the presence of several a-type twin boundaries. The twin planes were mechanically moved between exposures.
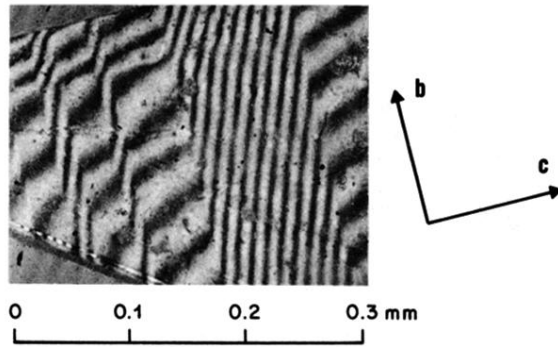
FIG. 4. Reflection interference pattern from the (100) surface of an *a*-type-twinned NdP_5O_{14} crystal showing the equal inclination angles of alternate regions separated by two twin boundaries.

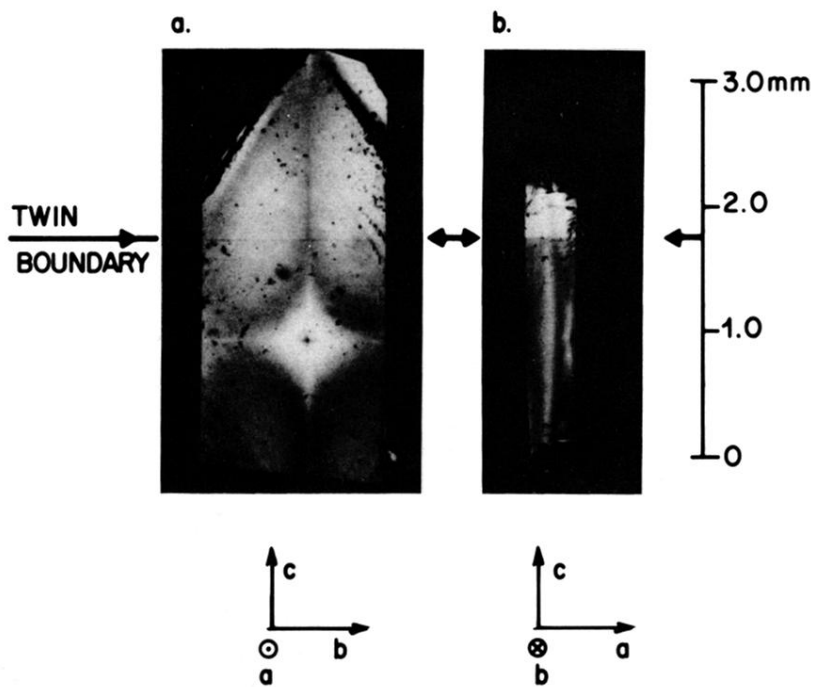FIG. 5. Transmission of light for a cleaved NdP_5O_{14} crystal containing one a-type twin boundary observed between crossed polarizers. (a) Viewing along [100] showing no significant contrast between the two regions. (b) Viewing along [010] showing the different extinction orientations of the two regions.
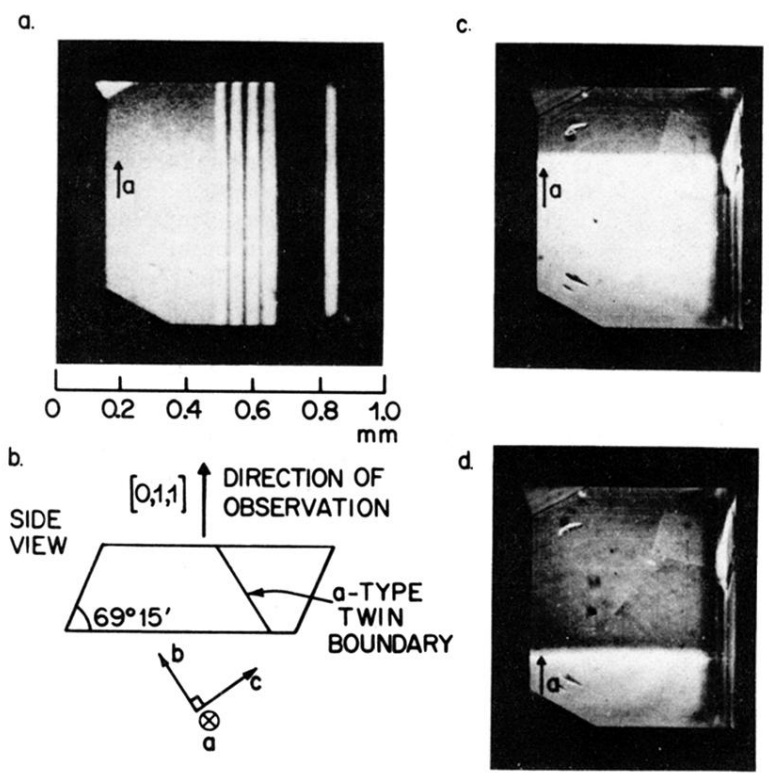
a.

0   0.2   0.4   0.6   0.8   1.0
                                mm

b.

SIDE
VIEW

$[0,1,1]$ ↑ DIRECTION OF
              OBSERVATION

69°15′ ⟍   a-TYPE
                        TWIN
                        BOUNDARY

b
c
⊗
a

c.

d.

FIG. 6. Transmission of light for two $Nd_{0.5}La_{0.5}P_5O_{14}$ crystals containing one a-type and one b-type twin, respectively, observed normal to the (011) plane between crossed polarizers. (a) Crystal with a-type twin showing a striped pattern revealing the smooth change in optical thickness across the twinned region. (b) Schematic orientation of the crystals. The a-type twin observed in Fig. 6(a) is also shown. The b-type twins in Figs. 6(c) and 6(d) would be in the plane of the paper. (c), (d) Crystal with b-type twin showing the different extinction orientations of the two regions. The twin plane was moved mechanically between exposures.