Dislocation-Containing Planar Faults in Metastably Retained Hexagonal Barium Titanate

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Planar faults, designated F_1 – F_6 and F_a , with intermixed dislocations in metastably retained hexagonal-BaTiO₃ ceramics, were found and analyzed by transmission electron microscopy. Only faults with one, two, and four extra c -layers parallel to the basal plane have been identified. Fault vectors $\mathbf{R}_{F_1} = \mathbf{R}_{F_a} = 1/6[0221]$, $\mathbf{R}_{F_2} = \mathbf{R}_{F_3} = 1/3[0111]$, $\mathbf{R}_{F_4} = 1/3[0112]$, and $\mathbf{R}_{F_1} = \mathbf{R}_{F_5} = \mathbf{R}_{F_6} = \mathbf{R}_{F_a} = 1/6[0221]$ were determined adopting the $2\pi\mathbf{g} \cdot \mathbf{R} = 0$ (or $2\pi n$) criteria in combination with high-resolution imaging. Further, the embedded dislocations were half-partials with Burgers vectors $\mathbf{b} = 1/3\langle 10\bar{1}0 \rangle$ determined by the $\mathbf{g} \cdot \mathbf{b} = 0$ effective invisibility criteria in conjunction with the eligible fault vectors. A rotation by 60° about the c -axis was found between fault segments with $\mathbf{R}_{F_1} = 1/6[0221]$ and $\mathbf{R}_{F_1} = 1/6[0221]$ located on either side of a partial. Basal dislocations with $\mathbf{b}_B = 1/3\langle 12\bar{1}0 \rangle$ have dissociated into two prism-plane Shockley half-partials with $\mathbf{b}_{Pr} = 1/3\langle 1\bar{1}00 \rangle$ by glide in the fault plane (0002) according to $1/3\langle 12\bar{1}0 \rangle \rightarrow 1/3\langle 1\bar{1}00 \rangle + 1/3\langle 0\bar{1}10 \rangle$. The fault segment F_1 encompassed by two half-partials is an extrinsic complex stacking fault.

I. Introduction

HIGH-TEMPERATURE¹ hexagonal (h -) BaTiO₃ ($P6_3/mmc$, No. 194) have been retained metastably at room temperature with acceptor doping,^{2,3} for example, Mg^{2+} or Fe^{3+} , substituting for the Ti^{4+} site, or by hot pressing^{4,5} in low oxygen partial pressures. The 6H-polytype (i.e., h -BaTiO₃) is not ferroelectric; another hexagonal phase ($P2_1$ (No. 17)) only becomes ferroelectric at ~ 77 K (-196°C).⁶ Although doped with acceptor-oxide Ga₂O₃, h -BaTiO₃ has been explored⁷ for microwave dielectrics; unlike the tetragonal phase, its practical applications are still limited. Fault vectors ($\mathbf{R}_F$) in the h -BaTiO₃ ceramics prepared by hot pressing in a graphite die assembly were analyzed and how these faults were generated in low oxygen partial pressures has been proposed.⁵ It was suggested that the stacking faults described as interleaved c -BaTiO₃ lamellae embedded in h -BaTiO₃ matrix are growth faults due to local oxygen excess accommodated by extra corner-sharing c -layer(s). The faults consisted of extra c -layers relative to the $(chc)_1(chc)_2$ (or equivalently (CBC)(ABA)) stacking sequence of 6H-polytype.^{3,5} They contained no dislocations.⁵ Here, we report on an analysis con-

centrating on planar faults, intermixed with dislocations, which have been divided into several segments. With dislocations running across the faults, the fault fringe contrast was distinctive on either side of a dislocation. A fringe contrast of alternate visible-invisible bands in strong-beam images had appeared among the fault segments.

Eibl *et al.*⁸ have reported the presence of both perfect and partial dislocations in h -BaTiO₃ ceramics sintered at 1440°C in air, followed by annealing at 1400°C in an N_2 – H_2 atmosphere. Similar to hexagonal close-packed metals (A3 hcp), and D0₁₉ intermetallics, the unit lattice translation vectors in h -BaTiO₃ are $1/3\langle 11\bar{2}0 \rangle$, $\langle 10\bar{1}0 \rangle$, and $[0001]$. Perfect dislocations with $\mathbf{b} = 1/3\langle 11\bar{2}0 \rangle$ and a pair of Shockley partials with $\mathbf{b}'$ and $\mathbf{b}'' = 1/3\langle 1\bar{1}00 \rangle$, all lying in the basal plane, were identified. Dissociation of the former into a pair of Shockley half-partials, often found in hexagonal systems, has also occurred in h -BaTiO₃ by the classical reaction

$$\mathbf{b} \rightarrow \mathbf{b}' + \mathbf{b}''$$

$$1/3[\bar{1}2\bar{1}0] \rightarrow 1/3[\bar{1}100] + 1/3[01\bar{1}0] \quad (1)$$

Some of the perfect dislocations not lying in the basal plane had climbed.⁸ The Shockley half-partials were found to lie within widely extended planar stacking faults (EPSF). The fault vector of the portion of the stacking fault bordered by partial dislocations D_4 and D_5 in Eibl *et al.*⁸ was $\mathbf{R}_F = 1/3[\bar{1}101]$ while that of the other portion was $\mathbf{R}_F = 1/3[\bar{1}101]$, such that the fault vectors have opposite components in the basal plane: $[1\bar{1}00]$ and $[\bar{1}100]$. It was concluded⁸ that the two fault vectors differed only in the components parallel to the basal plane, i.e. the prismatic components $1/3\langle 10\bar{1}0 \rangle$ of the general form: $\mathbf{R}_F = (n/6)[0001] + 1/3\langle 10\bar{1}0 \rangle$,⁵ by vectors other than the lattice vectors. In addition, the two segments of stacking fault, separated by one of the Shockley half-partials, lie in two distinct basal planes (i.e., $(000l)$ where $l = 1$ – 6) separated by $(1/2)\mathbf{c}$ where $\mathbf{c} = [0001]$.

In this study, the planar faults in hot-pressed h -BaTiO₃ found to be embedded with partial dislocations were determined for their type, fault vectors,⁵ and the intermixed dislocations for Burgers vectors by the transmission electron microscopy (TEM). All dislocations were half-partials with $\mathbf{b} = 1/3\langle 10\bar{1}0 \rangle$, consistent with those reported before by Eibl *et al.*⁸ Relationships between Burgers vectors and fault characteristics are discussed.

II. Experimental Procedure

A commercial hydrothermal BaTiO₃ powder⁵ (K-PlusTM) of an average particle size of ~ 120 nm purchased from Cabot[®] (Boyertown, PA) was used in this study. The powder was stoichiometric and contained major impurities of residual carbon

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(<1000 ppm), soluble chloride (<250 ppm), and strontium (<500 ppm), according to the manufacturer. Samples were hot pressed at 1300°C for 2 h by a uniaxial pressure of ~ 25.8 MPa in a graphite die assembly using a Fujidempa (Osaka, Japan) multipurpose furnace equipped with a graphite resistance-heating element whose temperature was monitored by a W-Re thermocouple and a two-color pyrometer. Hot-pressed samples, while being kept in the graphite die, were furnace cooled to room temperature.

As-hot-pressed disks of 15 mm diameter were lightly polished to remove graphite and boron nitride (BN) coating from the graphite spacers that were inserted between the graphite punch and BaTiO₃ samples before hot pressing. Thin foils for TEM were prepared by the conventional technique of cutting with a diamond-embedded saw, grinding, and polishing to an ~ 30 μm thickness, and 1 μm of surface roughness before dimple grinding to an ~ 10 μm thickness in the center region. The thin sections were then Ar⁺ ion-beam thinned (DuoMill™, Gatan, Pleasanton, CA) to electron transparency. Observations were made in a JEOL™ AEM3010 (Tokyo, Japan), with a double-tilting stage of $\pm 45^\circ$ for the x -tilt and $\pm 30^\circ$ for the y -tilt, operating at 300 kV.

III. Results

Stacking faults were observed in almost all h -BaTiO₃ grains. Grains were spherulitic in shape although not grown abnormally.^{2,9}

(1) General Observations—Fringe Patterns

Selecter area diffraction patterns were indexed⁵ corresponding to the standard triangles of the stereographic projection. Seven sets of faults are discernible from one of the h -BaTiO₃ grains imaged in strong beam bright field (BF) (Fig. 1). Spot streaking suggests that the foil contains planar defects inclining to the beam direction at deviation parameter $s_g \neq 0$ (Figs. 1(a) and (b)).^{5,10} Only six of them, designated as F_1 – F_6 , have been fully

characterized. Faults F_5 and F_6 were visible (Fig. 1(a)) when imaged with $g = \bar{1}01\bar{4}$. Faults F_5 and F_6 are partly superimposed in projection, producing a complex fringe pattern that remains symmetrical in BF. They are entirely similar to faults F_B and F_C in a previous report.⁵

Four dislocations, d_1 – d_4 , were detected in the field of view (Fig. 1(a)). Two of them, d_1 and d_2 , with the respective Burgers vectors $\mathbf{b}_{d_1}$ and $\mathbf{b}_{d_2}$, lie within fault F_1 , thus dividing F_1 into three segments, F_1 – F'_1 – F_1 , which show alternate fringe contrast. Using $g = \bar{1}01\bar{4}$, the inner segment F'_1 was visible (indicated by (O)) while the rest of the fault, on both sides of F'_1 , was invisible (indicated by (X)), i.e., fault contrast was reversed across both dislocations d_1 and d_2 . Nevertheless, the fringe patterns revealed by a strong-beam BF image were symmetrical and complementary (for $s_g = 0$) about a foil center.¹¹ Extreme fringe contrast was both dark in BF image, and the dark–dark (D–D) fringe pattern of F'_1 is indicated in Fig. 1(a). The fringes were however asymmetrical in CDF image where the extreme fringe contrast became dark–bright (D–B). The contrast characteristics are similar to those reported⁵ before, which allows us to conclude as of the α -type fault¹¹ with fault vectors ($\mathbf{R}_F$), thus directly related to the lattice displacement vectors of hexagonal crystals.

Fault F_7 is bordered by dislocation d_3 , as indicated in Fig. 1(c). Dislocation d_4 with Burgers vector $\mathbf{b}_{d_4}$ sits in the foil alongside with fault F_3 . Causing no change in the fringe pattern, it is not in contact with the fault. Nevertheless, its presence indicates that h -BaTiO₃ was plastically deformed during hot pressing by the movement of dislocations $\mathbf{b}_{d_4} = 1/3[10\bar{1}0]$.

Faults F_1 – F_4 are invisible (X) while F_5 and F_6 remain visible (O) both from $g = \bar{1}01\bar{4}$ and $110\bar{4}$. All these faults are invisible from $g = \bar{2}110$ and visible from $g = 2\bar{1}\bar{1}4$, accordingly (Figs. 1(a)–(d)). They are of a wedge shape as the foil thickness continuously decreases toward the left of the image.

(2) Determination of Fault Vectors for Faults F_1 – F_6 and F_a

The eligible fault vectors for F_1 – F_6 were determined by the three-step procedure⁵ described previously: (1) application of the invisibility criteria under diffraction contrast and under

Fig. 1. Contrast analysis for faults F_1 – F_6 using (a) $g = \bar{1}01\bar{4}$, (b) $g = 110\bar{4}$, (c) $g = \bar{2}110$, and (d) $g = 2\bar{1}\bar{1}4$ (bright-field image, transmission electron microscopy).

Fig. 4. Fault F_a interrupted by partial dislocation d_a : (a) bright-field image showing two segments F_a and F'_a ; (b) and (c) are high-resolution images taken from either side of partial d_a ; (d) and (e) are higher magnifications of the local regions of (b) and (c).

moved in the (0002) fault plane by glide, leaving the $\mathbf{c}$ -component of the fault vectors unchanged.

The unit lattice translation vectors are possible Burgers vectors for perfect dislocations. We have in h -BaTiO₃ the basal dislocation, $\mathbf{b}_B = 1/3\langle 11\bar{2}0 \rangle$, the prism-plane dislocation, $\mathbf{b}_{Pr} = \langle 10\bar{1}0 \rangle$, and the pyramidal dislocation, $\mathbf{b}_{Py} = 1/3\langle 20\bar{2}1 \rangle$. Similar to hcp A3 metals, $\langle 11\bar{2}0 \rangle$ are the close-packed directions in h -BaTiO₃. Basal slip along the close-packed directions, $1/3\langle 11\bar{2}0 \rangle$ (0001), is the easiest slip system. However, unlike the configurations reported by Eibl *et al.*⁸ who found basal dislocations $\mathbf{b}_B$ further dissociated into two prism-plane half-partials (by Eq.

(1)), only dissociated $\mathbf{b}_{Pr}$ have been identified in hot-pressed samples, i.e., d_1 , d_2 coplanar with fault F_1 , and d_3 , d_4 coplanar with F_7 and F_3 , respectively. Perfect basal dislocations with $\mathbf{b}_B = 1/3\langle 11\bar{2}0 \rangle$ that would indicate possible shear in this direction have not yet been identified in hot-pressed h -BaTiO₃.

However, Shockley half-partials, d_1 and d_2 , originated from the dissociation of basal dislocations $\mathbf{b}_B = 1/3\langle 11\bar{2}0 \rangle$ by the reaction $\mathbf{b}_B \rightarrow \mathbf{b}_{d_1} + \mathbf{b}_{d_2}$:

$$1/3[1\bar{2}10] \rightarrow 1/3[1\bar{1}00] + 1/3[0\bar{1}10] \quad (2)$$

As basal dislocations had subsequently dissociated into two prism-plane half-partials (by Eq. (2)), they were observed in the present samples. Shear still occurs principally along $\langle 11\bar{2}0 \rangle$ in h -BaTiO₃, only that such basal dislocations induced plastically had dissociated. Therefore, plastic deformation took place predominantly through the easiest slip system $1/3\langle 11\bar{2}0 \rangle$ (0002) in h -BaTiO₃ when a powder of ~ 120 nm was hot pressed at 1300°C.

The dissociation of basal dislocations into partials d_1 and d_2 with $\mathbf{b}_{d_1}$ and $\mathbf{b}_{d_2}$ takes place by glide in (0002), the B-layer in (CBC) stacking sequence, as the $\mathbf{c}$ component of faults F_1 and F'_1 is the same. The fault segment encompassed by partials d_1

Table II. True Directions, Type of Dislocations and the Relevant Slip Plane

Dislocation	True direction $\mathbf{u}_i$	Burgers vector $\mathbf{b}$	Slip plane	Dislocation type
d_1	$[0\bar{1}10]$	$1/3[1\bar{1}00]$	(0002)	Mixed (60°)
d_2	$[1\bar{2}10]$	$1/3[0\bar{1}10]$	(0002)	Mixed (30°)
d_3	$[10\bar{1}0]$	$1/3[10\bar{1}0]$	(0002)	Screw
d_4	$[1100]$	$1/3[10\bar{1}0]$	(0002)	Mixed (60°)
d_a	$[2110]$	$1/3[10\bar{1}0]$	(0002)	Mixed (30°)

Fig. 5. Stacking sequence of the unfaulted *h*-BaTiO₃ and those of the five-, six-, seven-, eight-, and nine-layer faults with shear on (0002), including faults (a) inclined to the left, and (b) inclined to the right.⁵

and d_2 is therefore a complex extrinsic stacking fault (CESF). The complex fault enveloped by partials d_1 and d_2 is fault segment F'_1 that contains one extra *c*-layer with fault vector $\mathbf{R}_{F'_1} = 1/6[20\bar{2}1]$ (Fig. 2(a)). The fault vectors of F_1 and F'_1 belong to two crystallographically distinctive groups of $1/6\langle 02\bar{2}1 \rangle$, whose projections on (0002) together with $\mathbf{b}_{d_1}$ and $\mathbf{b}_{d_2}$ are shown schematically in Fig. 6.

Fig. 6. Dissociation of basal dislocation with $\mathbf{b}_B = 1/3[1210]$ to prism-plane partial dislocations ($\mathbf{b}_{pr}$) with $\mathbf{b}_{d_1} = 1/3[1100]$ and $\mathbf{b}_{d_2} = 1/3[0110]$ in the (0002) plane.

Compared with the stacking fault energy $\gamma_{SF} \approx 108 \text{ mJ/m}^2$ of *t*-BaTiO₃,¹⁵ the larger separation (d) between a pair of half-partials, d_1 and d_2 , of $\sim 500 \text{ nm}$ (Fig. 1(a)) indicates a significantly lower γ_{SF} in *h*-BaTiO₃. However, this value cannot be estimated at present as no shear modulus for *h*-BaTiO₃ is available in the literature. Partial dislocations separated by $d \approx 120 \text{ nm}$ found by Eibl *et al.*,⁸ being much narrower, may not lie in the same plane but in planes differing by $(1/2)c$ indicate a climb dissociation of $\mathbf{b}_B$ into half-partials (nevertheless, similarly, by Eq. (2)). Fault segments on both sides of the partials D_4 and/or D_5 in Eibl *et al.* would have distinctive fault vectors with a characteristic stacking sequence containing different extra *c*-layers. This necessitates climb of partial dislocations upon dissociation in prism planes at the interface of the two stacking faults. One of a pair of half-partials migrated by a mixed glide in (0002) and climb in (1010) has indeed been determined.¹⁶

The glide of d_1 with $\mathbf{b}_{d_1} = 1/3[1100]$ on (0002), the fault plane of F_1 , has effectively rotated $\mathbf{R}_{F_1} = 1/6[02\bar{2}1]$ by 60° clockwise to $\mathbf{R}_{F'_1} = 1/6[20\bar{2}1]$. A complex stacking fault is created extrinsically in fault segment F'_1 with $\mathbf{R}_{F'_1}$. Partial dislocation d_2 with $\mathbf{b}_{d_2} = 1/3[0110]$ then restores $\mathbf{R}_{F'_1}$ back to $\mathbf{R}_{F_1}$ in a similar fashion. Thus, bordering between faults F_1 and F'_1 is an interface characterized by half-partial $\mathbf{b}_{d_1} = 1/3[1100]$ (Fig. 6). Analogously, the interface across F'_1 and F'_a is generated by $\mathbf{b}_{d_2} = 1/3[0110]$. Consequently, the configuration of F_1 is resumed and $\mathbf{R}_{F_1}$ restored after the trailing half-partial, $\mathbf{b}_{d_2}$. Fault segments F_1 – F'_1 – F'_a separated by dislocations d_1 and d_2 did not have identical shear vectors (Table I) due to the displacement of $B \rightarrow A$ (for F_1) versus $B \rightarrow C$ (for F'_1) in (0002) (Fig. 5). Nevertheless, the total number of nine layers in F_1 and F'_1 remains unchanged across half-partials d_1 and d_2 , i.e., $(R_5 + L_4)$ to $(L_5 + R_4)$, from a right-inclined fault (F_1 in Fig. 2(a)) to a left-inclined one (similar to that of F'_a) in Fig. 4(e). Dissociation of perfect basal dislocation $\mathbf{b}_B$ by a pure glide in (0002) has resulted in an extended separation between half-partials, such as that observed for F_1 – F'_1 at $d \approx 500 \text{ nm}$ (Fig. 1(a)). Indeed, a separation of $d > 320 \text{ nm}$ for d_a and its pair partial (which should have been located left of d_a) (Fig. 4(c)) if not milled off) was estimated from a lower magnification strong-beam image. When dissociation occurs by climb, such as that found by Eibl *et al.*⁸ or a

mixed mechanism,¹⁶ half-partials would have separated the initial fault into two fault segments consisting of different extra *c*-layers, i.e. differing in the *c* component of fault vectors $\mathbf{R}_{F_{(n)}}$. Planar faults in *h*-BaTiO₃ intersected by glide-dissociated half-partials and divided into two or more segments of dissimilar fault vectors (Table I) but of similar stacking sequence (*chc*)*c*(*chc*) are found with F_a and F'_a (Figs. 4(d)–(e)). Whether climb dissociation⁸ of $\mathbf{b}_B$ generated by plastic deformation occurs at all may be investigated by determining the fault vector for each of the fault segments. Climb of partials must occur whenever faults with different extra *c*-layers are encountered in dislocation dissociation. Fault vectors of such segments suggest that the intersecting dislocations have climbed by $(n/6)[0001]$ from one segment to the next, for example $1/2[0001]$ reported by Eibl *et al.*⁸ and Wu *et al.*¹⁶ If there are dislocations intersecting between a five-layer fault, e.g. F_1 and a six-layer fault, e.g. F_2 , climb of $\mathbf{b} = 1/3[1\bar{1}00]$ by $1/6[0001]$ must occur upon crossing from $\mathbf{R}_{F_1} = 1/6[0221]$ to $\mathbf{R}_{F_2} = 1/3[10\bar{1}1]$, although such dislocations were not observed here. Other combinations with different extra *c*-layers in stacking faults are therefore possible. This has consequently produced discrepant separations between half-partials, such as $d \approx 500$ nm determined in the present study and ~ 120 nm reported by Eibl *et al.*⁸ and ~ 47 – 195 nm by Wu *et al.*¹⁶

V. Conclusions

Eligible fault vectors determined for faults in metastably retained *h*-BaTiO₃ with dislocations embedded were similar to those without the intersecting dislocations. Only three of the six prismatic components of $\langle 10\bar{1}0 \rangle$ were eligible for type II faults F_1 – F_6 , and F_a (with $\mathbf{R}_{F_{(n)}}$ fault vectors) analyzed here.

During hot pressing, the Shockley half-partials with $\mathbf{b}_{Pr} = 1/3\langle 0\bar{1}10 \rangle$ were dissociated from basal dislocations with $\mathbf{b}_B = 1/3\langle 1120 \rangle$ by glide in (0002) and/or climb in prism planes of *h*-BaTiO₃. The interface dislocation with $\mathbf{b}_{Pr} = 1/3\langle 0\bar{1}10 \rangle$ has effected a 60° rotation of fault segment F_1 with $\mathbf{R}_{F_1}$ to F'_1 with $\mathbf{R}_{F'_1}$ about $[0001]$, and then from F'_1 back to F_1 by another rotation of 60° successively. Fault segments F_1 – F'_1 and F'_1 – F_1 are

bordered by a pair of half-partials, d_1 and d_2 , one leading and the other trailing. The fault segment F'_1 enveloped by half-partials is a CESF.

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