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Transmission Electron Microscopy Study on the Annealing of $YBa_2Cu_3O_{7-\delta}$

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T.E.M. Studies on the Annealing of $YBa_2Cu_3O_{7-\delta}$

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Abstract

Annealing in oxygen-rich atmosphere at intermediate temperatures ($400^o C$ - $600^o C$) has proved necessary to provide the appropriate microstructure for superconducting $YBa_2Cu_3O_{7-\delta}$. The symmetry of the orthorhombic phase requires that if more than one type of twin plane is present within a grain, a distorted region should exist inside the multiple twinned grain. This distorted region hinders the tetragonal - to - orthorhombic transformation, and may account for some retained tetragonal phase inside an otherwise orthorhombic grain. A physical model is presented describing the formation of such regions and their eventual transformation into low angle grain boundaries after long annealing. Extended annealing at intermediate temperatures apparently leads to the formation of planar faults in off-stoichiometric samples. Transmission electron microscope image contrast and energy dispersive x-ray analyses of highly defective regions suggests these defects are $CuO_x (x = 1, 2)$ extra layers, which result from the slight copper enrichment in the $YBa_2Cu_3O_{7-\delta}$ used here. These extra layers tend to form near grain boundaries or free surfaces, where oxygen is readily available.

I Introduction

The discovery of the so-called high-temperature superconductors by Bednorz and Muller [1] and the subsequent discovery of superconductivity in $YBa_2Cu_3O_{7-\delta}$ at even higher temperatures [2] has promoted considerable interest in the study of its properties. This material has been produced either as single crystals or polycrystalline pellets. The crystal structure of the high-temperature superconducting phase has been accurately determined by neutron diffraction [3]; tetragonal at high temperatures, and orthorhombic at low temperatures and higher oxygen contents. In polycrystalline materials the critical temperature and field, as well as the critical current density, have been shown to depend strongly on the microstructure, particularly grain boundaries and grain orientation, and therefore on the processing conditions. One of the most critical steps in the processing of these materials is the final annealing, which is carried out to achieve the desired oxygen stoichiometry ($\delta \rightarrow 0$).

Different workers have proposed different annealing conditions, which are not totally consistent with each other, to optimize the superconducting properties of $YBa_2Cu_3O_{7-\delta}$ [4,5,6,7]. According to Ginley et al. [5] the tetragonal phase can be made orthorhombic upon annealing at temperatures as low as 300^oC, and therefore higher temperatures only improve the kinetics of oxygenation. Umakoshi et al. [4] have found that annealing at temperatures below 500^oC or above 600^oC produced samples with the highest critical temperatures. Johnson et al. [7], on the other hand, have found optimum properties when annealing was carried out at 550^oC. Part of the disparity among those results can be attributed to differences in the composition of the original powders, and to the difficulty of controlling the oxygen content.

Second phases commonly exist in grain boundaries of polycrystalline ceramics, and may result from slight overall or local deviations from the exact stoichiometry, or liquid phase sintering [8] if the processing temperatures are high, and from contamination of the powder. An example of second phase segregation at grain boundary in $YBa_2Cu_3O_{7-\delta}$ has been given by Schrott et al. [9], who used photoemission spectroscopy to detect $BaCO_3$ at the grain boundaries. The presence of second phases in $YBa_2Cu_3O_{7-\delta}$ leads to deterioration of the superconducting properties.

In addition to second phases, the presence of extended defects should also have a negative effect on the superconducting properties of $YBa_2Cu_3O_{7-\delta}$. Several types of such defects have been identified in this material. Twin boundaries [10,11] with a (110) mirror plane, in which the a and b axis are interchanged across the boundary, are always present in polycrystalline $YBa_2Cu_3O_{7-\delta}$. These are formed in order to accomodate the strain energy generated as a consequence of the tetragonal-to-orthorhombic phase transformation. Zandbergen et al. [11] have also shown the existence of [100] 90^o twins with twin boundaries predominantly along (100) planes, although sintering at high temperatures inhibits their formation.

Another commonly observed extended defect results from the insertion of extra $CuO_x (x = 1, 2)$ layers parallel to the (001) plane [12,13]. The schematic representation of such defective regions, provided originally in reference [12], is useful to show the possible positions of the extra layer. An extra CuO layer can exist either between a CuO and a BaO layer (Figure 1 (a) or (b)), resulting in a packing sequence [12,13]

$$CuO_2 - Y - CuO_2 - BaO - CuO^* - CuO^* - BaO - CuO_2 - Y - CuO_2$$

or between a Y and a BaO plane (Figure 1 (c)), resulting in the packing sequence [13]

CuO - BaO - CuO_2 - Y - CuO_2^* - CuO_2^* - BaO - CuO - BaO - CuO_2

The asterix indicates the location of the extra layer. Planar faults are common in perovskite-like materials. They have been reported to occur, for instance, in $SrTiO_3$ with excess Sr [14], where the excess Sr is accommodated as inserted SrO layers between blocks of perovskite structure.

Planar faults of the type described in the previous paragraph should be bound by dislocations if they terminate within the crystal. Work that has demonstrated the existence of the extra $CuO_x(x = 1, 2)$ [12,13] has not indicated the existence of these dislocations. Ikeda et al. [15] have reported TEM results of analysis of dislocations in $YBa_2Cu_3O_{7-\delta}$ with possible Burgers vectors [100] and/or [010]. This result has not been confirmed by high resolution electron microscopy.

The present work addresses two major points. First, it demonstrates that despite the ability of reoxygenated tetragonal $YBa_2Cu_3O_{7-\delta}$ to produce the orthorhombic superconducting phase at temperatures as low as $200°C$ [5], higher temperatures are required for kinetic reasons and to produce a microstructure that results in the desired properties. Second, it shows that $CuO_x(x = 1, 2)$ double layers bound by dislocations are created during long annealing of samples containing excess copper. These faults can occur either at grain boundaries or inside grains. An explanation based on the minimization of broken bonds is given to justify the apparent existence of defects with two distinct displacement vectors. According to this, the displacement vector associated with the extra layer depends on the type of domain where the defect originally nucleated.

II Experimental

The $YBa_2Cu_3O_{7-\delta}$ used in this investigation was prepared from powders made by the freeze-drying method [7]. In this process a solution of salts is prepared, frozen rapidly, and then freeze-dried to remove the solvent. Freeze drying is carried out at low temperature and high vacuum to allow sublimation of the solvent, rather than melting. The resulting powder has a large surface area although it may be agglomerated. A large surface area is very desirable to improve sintering kinetics and produce a higher density. This powder was calcined at temperatures of approximately 800^oC, and sintered at 890^oC for several hours.

The pellets were annealed in flowing oxygen at the conditions described below:

- 450^oC for 20 hours and furnace cooling
- 550^oC for 2 hours and furnace cooling
- 700^oC for 8 hours and furnace cooling
- 700^oC for 32 hours and furnace cooling
- 750^oC for 20 hours and furnace cooling
- 750^oC for 20 hours followed by annealing at 450^oC for 20 hours and furnace cooling

Transition temperatures of the annealed material were above 90 K. The microstructure of the samples before and after each heat treatment was characterized by transmission electron microscopy with a Phillips 301 microscope. Composition analysis was carried out in a Phillips 400 electron microscope equipped with a energy dispersive X-ray spectrometer. Both microscopes were equipped with double-tilt specimen holders, so that contrast analysis of the extended defects could be performed.

III Results and Discussion

III-1 Evolution of Microstructure during Annealing

The stable phase of $YBa_2Cu_3O_{7-\delta}$ at high temperatures is the tetragonal one, and this is the phase that should exist after the sintering operation. Lattice constants of this phase at room temperature have been determined to be $a = b = 0.385\,nm$ and $c = 1.17\,nm$ [16,17], although slight variations of these values may result from deviations in the oxygen stoichiometry [18]. The tetragonal phase transforms into the orthorhombic phase when the material is cooled below about $700^o C$, the exact temperature depending on the oxygen content of the sample [19]. The orthorhombic phase, with lattice constants $a = 0.382\ nm$, $b = 0.389\ nm$ and $c = 1.168\ nm$ [17], results from the alignment of oxygen vacancies along the [100] direction [19,20].

The change in lattice constants associated with the T $\rightarrow$ O transformation is accommodated by the subdivision of each grain in parallel plates that are (110) twin related 90^o domains [20]. Figure 2 is an electron micrograph of such domains in a sample that was annealed in oxygen for 8 hours at $700^o C$ and slowly cooled to room temperature. Although Figure 2 may suggest that the domain widths are uniform in samples that have been annealed for extended periods of time, this was not found to occur even when $YBa_2Cu_3O_{7-\delta}$ was annealed for over 30 hours. An approximate uniformity was found to exist within each individual grain, but not among different grains.

Figure 3 is an electron micrograph taken from a $YBa_2Cu_3O_{7-\delta}$ pellet quenched from the sintering temperature and then annealed at 550^oC for 2 hours. It shows that when a twin domain is excessively wide in comparison with the neighboring ones, a new domain, twinned with respect to the parent one, is produced at the grain boundary and grows inwards. Since there is no composition difference between the two domains, the driving force for the formation of the new one should be the reduction of free energy associated with elastic strain relaxation. Minimization of elastic strain by formation of twin related domains is a common phenomenon in phase transformations of metallic alloys and ceramics [21]. The models proposed so far to describe the structure of the twin boundaries suggest a deficiency [22] or an excess [10] of oxygen atoms with respect to the bulk concentration. Therefore, the nucleation of these new domains at the grain boundaries is understandable, as grain boundaries could act as good oxygen sinks or vacancy sources or vice-versa.

Consistent results have not been obtained concerning the optimum annealing conditions for producing $YBa_2Cu_3O_{7-\delta}$ with high transition temperatures. Slow cooling of the $YBa_2Cu_3O_{7-\delta}$ from temperatures at which the tetragonal phase is stable to room temperature has been suggested for producing materials with superconducting properties similar to materials that have been subjected to long annealing [4]. Other researchers [7] disagree with those results, and claim the need for a post-sintering low-temperature annealing in order to achieve the appropriate oxygen stoichiometry. Here we compare microstructures of $YBa_2Cu_3O_{7-\delta}$ that have been slowly cooled from 750^oC to room temperature with some that have been slowly cooled from 750^oC to 450^oC, annealed at this temperature for 20 hours, and finally cooled to room temperature. In both cases, the samples were kept at 750^oC for 20 hours. The thermodynamically stable forms of $YBa_2Cu_3O_{7-\delta}$ at 750^oC and 450^oC under 1 atm oxygen pressure are tetragonal and orthorhombic respectively [19]. However, before making this comparison, it is necessary to explain the possible microstructures.

As is suggested above, and in agreement with the model proposed by Jou and Washburn [22], growth of the orthorhombic domains starts from the grain boundaries of the parent tetragonal phase. However, due to the symmetry of the tetragonal phase, it is equally likely that those domains are twinned with respect to the (110) or the $(1\bar{1}0)$. Therefore it is possible that the two sets of twin related domains are formed at the early stages of the transformation, and then grow towards the center of the grain. Similar microstructures resulting from this type of growth have been observed by others [10,23].

During the T $\rightarrow$ O phase transformation the twin planes are expected to be invariant. Since (110) and $(1\bar{1}0)$ are orthogonal in the tetragonal phase and not orthogonal in the orthorhombic phase, twinning along these two planes within a grain results in distortion in the region where the two different sets of domains meet. This distortion is a consequence of the 0.5^o rotation around [001] that is required to maintain $(110)_T$ // $(110)_O$. The same 0.5^o rotation, but in the opposite direction, is required if the same domain is such that $(1\bar{1}0)_T$ // $(1\bar{1}0)_O$. Therefore, if a particular domain is bound by $(110)_O$ and $(1\bar{1}0)_O$, there must be a region within it that is slightly distorted (about 1^o). This distortion can be accommodated either by elastic strain or by retention of some tetragonal phase within the orthorhombic phase. Ultimately, it may lead to the formation of a low-angle grain boundary as discussed below.

A schematic representation of the microstructure resulting when a tetragonal grain is transformed to the orthorhombic structure with twinning along two orthogonal planes is shown in Figure 4. When enough time and oxygen are provided such that the two sets of twin related domains meet each other, low angle grain boundaries are produced. These can be twist boundaries if the boundary plane is $\{110\}$, or tilt boundaries if the boundary plane is $\{001\}$, as shown in Figure 4 (a) and (b) respectively. As for the case where the domain walls do not meet, represented in Figure 4 (c), the intermediate region is likely to be untransformed tetragonal phase.

The microstructure of $YBa_2Cu_3O_{7-\delta}$ that was annealed at 750^oC and furnace cooled was observed by TEM, and some grains showed a pattern similar to the one described in Figure 4 (c), grain A in Figure 5 for example. In the adjacent grain B, in the same figure, there is only one twin plane, and apparently the transformation was complete. Samples of $YBa_2Cu_3O_{7-\delta}$ that had been subjected to the long low temperature anneals, i.e. 400^oC - 600^oC, did not show this microstructure, and the domain walls either terminate at grain boundaries or at another domain wall, as is suggested in Figures 3 (a) and (b).

Regions of retained tetragonal phase, shown in 4(c), and perhaps the small angle grain boundaries, shown in 4(a) and 4(b), have a degrading effect on the superconducting properties of $YBa_2Cu_3O_{7-\delta}$. Their effect on the ability of the $YBa_2Cu_3O_{7-\delta}$ to transport current can be understood as that of non-superconducting regions dispersed inside superconducting material. Deutscher and Muller [24] have suggested that twin boundaries and grain boundaries act as Josephson junctions, with such boundaries being non-superconducting. The microstructures resulting from incomplete transformation and described in the previous paragraphs can, similarly, produce Josephson junctions internally in the grains. This can explain previously reported results [25] that provided some evidence of magnetic flux pinning within grains of orthorhombic $YBa_2Cu_3O_{7-\delta}$. Since the London penetration depth of $YBa_2Cu_3O_{7-\delta}$ is of the order of the size of the unit cell [9], the annealing treatment becomes very important for eliminating gaps such as the ones shown in Figure 4(c). Directional cooling from the sintering temperature may therefore be an interesting means of hindering the formation of such insulating junctions within $YBa_2Cu_3O_{7-\delta}$ grains.

Annealing of orthorhombic $YBa_2Cu_3O_{7-\delta}$ apparently also promotes homogenization of this phase. Composition and microstructure of samples that were quenched from the sintering temperature were compared to samples that were annealed at 550^oC for 2 hours. The quenched samples contained primarily tetragonal $YBa_2Cu_3O_{7-\delta}$, as evidenced by the lack of twinning, whereas the annealed samples were fully transformed into the orthorhombic structure. An unexpectedly large difference was found between the composition of the phases in the two sets of samples.

The $YBa_2Cu_3O_{7-\delta}$ used in this study is slightly copper-rich and yttrium-poor. Yang et al. [26] have reported that a small copper enrichment, a stoichiometric coefficient between 3 and 3.5, is not detrimental to the superconducting properties of $YBa_2Cu_3O_{7-\delta}$. In addition, the excess copper acts as a fluxing agent promoting grain growth and crystal orientation alignment. Energy dispersive X-ray analyses (EDX) have shown that the excess copper in the samples used here was either copper oxide or excess Cu in the $YBa_2Cu_3O_{7-\delta}$. The average composition of the metal ions in the $YBa_2Cu_3O_{7-\delta}$ phase in the quenched and annealed samples, as measured by EDX, is shown in Table I, as well as the concentration that would be expected according to the stoichiometric coefficients 1-2-3. It is obvious from Table I that the annealing at 550^oC helped to homogenize the sample and allowed an increase in the Y content of the $YBa_2Cu_3O_{7-\delta}$ phase. Annealing for much longer periods did not bring the Y content to the expected stoichiometry value.

Figure 6 shows an example of the heterogeneities that were found in the quenched samples. It consists of an unreacted yttrium-copper oxide grain surrounded by a copper oxide grain, which in turn is immersed in the off-stoichiometric $YBa_2Cu_3O_{7-\delta}$. The EDX spectra of the regions marked in the micrograph are also provided, and the corresponding compositions are presented in Table II. These types of heterogeneities were never found in the annealed samples. In fact, the only phase other than $YBa_2Cu_3O_{7-\delta}$ found in the annealed samples was copper oxide.

III-2 Planar Defects in $YBa_2Cu_3O_{7-\delta}$

As shown in the previous section, the $YBa_2Cu_3O_{7-\delta}$ used in this study is slightly copper-rich and yttrium-poor. Deviations from the ideal stoichiometry may lead to the formation of extended defects, such as planar faults, during the high temperature treatments. Therefore, these $YBa_2Cu_3O_{7-\delta}$ samples were suitable for studying the formation of the $CuO_x(x = 1, 2)$ double layers that have been reported elsewhere [12,13]. Planar defects have been associated with regions close to free surfaces, to which oxygen can diffuse easily. However, these reports have not accounted for the change in composition resulting from or required for the formation of such defects.

Planar faults, which produce image contrast similar to stacking faults, were observed in samples that had been annealed for extended periods of time. Examination of the same material prior to the heat treatment did not show such defects. The alternating bright-dark fringe images arise because there is an abrupt change in the phases of the incident and diffracted electron waves as they encounter the defect. Such faults displace the matrix planes on either side. The total relative displacement between the two sides of the fault is referred to here as **R**.

The distribution of these faults in $YBa_2Cu_3O_{7-\delta}$ was not uniform. They were usually located near or at grain boundaries as shown in Figure 7, although in a few instances these faults could be seen in the interior of grains. These faults were also sometimes found near the edges of the thin-foil used for transmission electron microscopy, but this is believed to be an artifact resulting from the increased surface area available in these regions. Energy dispersive X-ray analysis was used to probe the concentration of the metallic ions along the regions where the incidence of such defects were high. Table III shows the concentration of Cu, Ba and Y at the positions A, B, and C indicated in Figure 8(a). A slight increase in Cu concentration was noticed in region B. Although the increase was just about the accuracy of the instrument, the same trend was found in other boundaries where the concentration of planar faults and the associated dislocations were high. Such an increase in copper content was not observed in defect-free grain boundaries. These findings agree with the results of others [12,13] that described these faults as extra (CuO_x) layers. Figure 7(a) and 8(b) show that these faults are bound by dislocations with Burgers vectors in the [010] or [100] direction. The role of grain boundaries in the formation of the planar defects is to provide the necesssary pathways for oxygen diffusion.

These planar faults were found to have a {001} habit plane. Initial contrast analyses have indicated that the displacement vector associated with these defects were either $\mathbf{R} = 1/6[031]$ or $\mathbf{R} = 1/6[301]$, in agreement with the models proposed by Zandbergen et al. [12] and Matsui et al. [13]. Figure 7 shows two bright field images obtained from the same region of a

$YBa_2Cu_3O_{7-\delta}$ thin foil with two different operating reflections. The image contrast analysis by itself was not able to differentiate whether these faults consisted of extra CuO or CuO_2 layers because the displacement vector associated with the two types of faults are nearly the same. The possibility of formation of each type of defect is discussed in the following paragraphs.

CuO extra planes may, in principle, be associated with both displacement vectors $\mathbf{R} = 1/6[301]$ and $\mathbf{R} = 1/6[031]$, as shown in Figure 1. However, in order for the former $\mathbf{R}$ to occur, the Cu ions in the adjacent CuO layers should have some unsatisfied bonds. On the other hand, when an extra CuO layer is inserted so that the resulting displacement vector is $\mathbf{R} = 1/6[031]$, all the bonds in the adjacent CuO layers are satisfied. Therefore it is expected that the latter cofiguration, shown in Figure 1(b), is the most stable and should prevail. CuO_2 extra planes have similar displacement vectors, 1/6[301] and 1/6[031], although due to the spacial configuration of the Cu ion and its surrounding O, both displacements are equally likely. In all cases, the planar faults are bound by dislocations with Burgers vectors parallel to [100] or [010].

The effect of these defects on the properties of $YBa_2Cu_3O_{7-\delta}$ has not been clarified yet. One should expect, however, degrading effects, due to the strain fields around such defects and to the excess charge associated with them. Both enhance electron scattering and therefore should increase resistivity.

IV Conclusions

Annealed $YBa_2Cu_3O_{7-\delta}$ has been studied by transmission electron microscopy. It was found that annealing at temperatures around 500^oC for extended periods of time is required in order to allow total transformation inside the grains of the orthorhombic phase. The T $\rightarrow$ O transformation is apparently easier and faster when the new phase consists of domains that are twin related with only one twin plane. When the orthorhombic phase contains two twin planes inside a single grain, elastic strain, resulting from small rotations around the c-axis necessary to maintain the twin plane invariants, appears to hinder the transformation. When an orthorhombic domain becomes substantially wider than adjacent ones, a new domain, twin-related to the parent one, forms at a grain boundary and grows towards the center of the grain. Comparisons between quenched and annealed $YBa_2Cu_3O_{7-\delta}$ have demonstrated that an annealing treatment at about 500^oC is useful not only to provide the correct oxygen stoichiometry, but also to improve the homogeneity of the material.

Prolonged annealing of copper-rich $YBa_2Cu_3O_{7-\delta}$ leads to the formation of extra $(CuO_x, x = 1, 2)$ layers normal to the c-axis. These faults are formed primarily at grain boundaries or free surfaces (e.g. pores), which provide a sufficient supply of oxygen. These faults are bound by dislocations with Burgers vectors in the direction of [100] and/or [010].

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Table I

Concentration of metal ions in $YBa_2Cu_3O_{7-\delta}$ (atomic %)

Species	Stoichiometric $YBa_2Cu_3O_{7-\delta}$	Annealed $YBa_2Cu_3O_{7-\delta}$	Quenched $YBa_2Cu_3O_{7-\delta}$
Y	17	13	8-9
Ba	33	33	34
Cu	50	54	57

Table II

Concentration of metal ions in Figure 5 (atomic %)

Species	Position C	Position D	Position A
Y	27.8	8.9	0.6
Ba	6.3	34.1	2.3
Cu	65.9	57.0	97.1

Table III

Concentration of metal ions in Figure 6 (atomic %)

Species	Position A	Position B	Position C
Y	11.9	11.1	11.8
Ba	34.7	33.3	34.1
Cu	53.3	55.6	54.0

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Figure Captions

Figure 1: Likely configuration of planar faults resulting from excess copper. (a) $\mathbf{R} = 1/6[03\bar{1}]$, extra CuO plane; (b) $\mathbf{R} = 1/6[30\bar{1}]$, extra CuO plane; (c) $\mathbf{R} = 1/6[30\bar{1}] = 1/6[03\bar{1}]$, extra CuO_2 plane.

Figure 2: Bright (BF) and dark field (DF) images of $YBa_2Cu_3O_{7-\delta}$ annealed in flowing oxygen for 8 hours and slowly cooled to room temperature. The twin related domains are characteristic of the orthorhombic phase.

Figure 3: Bright field electron micrograph of $YBa_2Cu_3O_{7-\delta}$ after quenching from the sintering temperature and annealing at 550^oC for 2 hours. Twin related domains originated at the grain boundaries to relax strain energy.

Figure 4: Schematic representation of the likely microstructures that may result from twinning along (110) and $(1\bar{1}0)$ in the orthorhombic $YBa_2Cu_3O_{7-\delta}$. (a) A low angle twist boundary results inside a domain. (b) A low angle tilt boundary results inside a domain. (c) Incomplete transformation results in disordered regions inside the grain.

Figure 5: Bright field image of $YBa_2Cu_3O_{7-\delta}$ annnealed in flowing oxygen at 750^oC and slowly cooled to room temperature. Grain A shows the microstructure expected in partially transformed materials, whereas grain B shows that expected in fully transformed materials.

Figure 6: Bright field image on $YBa_2Cu_3O_{7-\delta}$ quenched from sintering temperature. EDX spectra of the letterd regions are included to show the non-uniform composition. See also Table II.

Figure 7: Planar faults (P) near a grain boundary and bound by dislocations (d) in $YBa_2Cu_3O_{7-\delta}$ annealed at 750^oC for 20 hours followed by annealing at 450^oC for 20 hours.

Figure 8: Bright field images of $YBa_2Cu_3O_{7-\delta}$ quenched and annealed for

2 hours at $550^o C$. Two distinct diffraction conditions were used to highlight the existence of faults in region B (a), which are bound by dislocations (b).

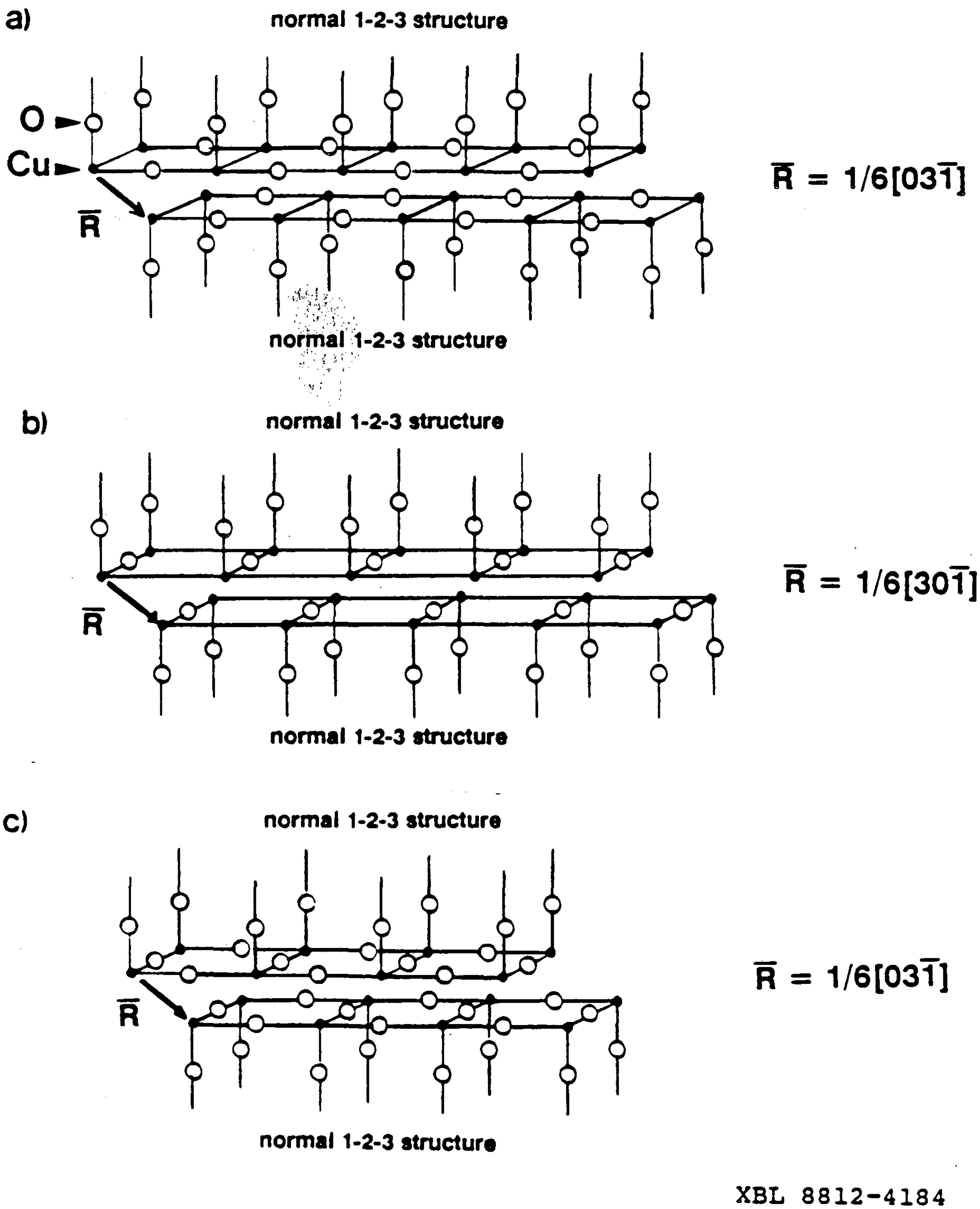

a)
normal 1-2-3 structure
O
Cu
R̄
R̄ = 1/6[03$\bar{1}$]
normal 1-2-3 structure
b)
normal 1-2-3 structure
R̄
R̄ = 1/6[30$\bar{1}$]
normal 1-2-3 structure
c)
normal 1-2-3 structure
R̄
R̄ = 1/6[03$\bar{1}$]
normal 1-2-3 structure
XBL 8812-4184

Fig 1

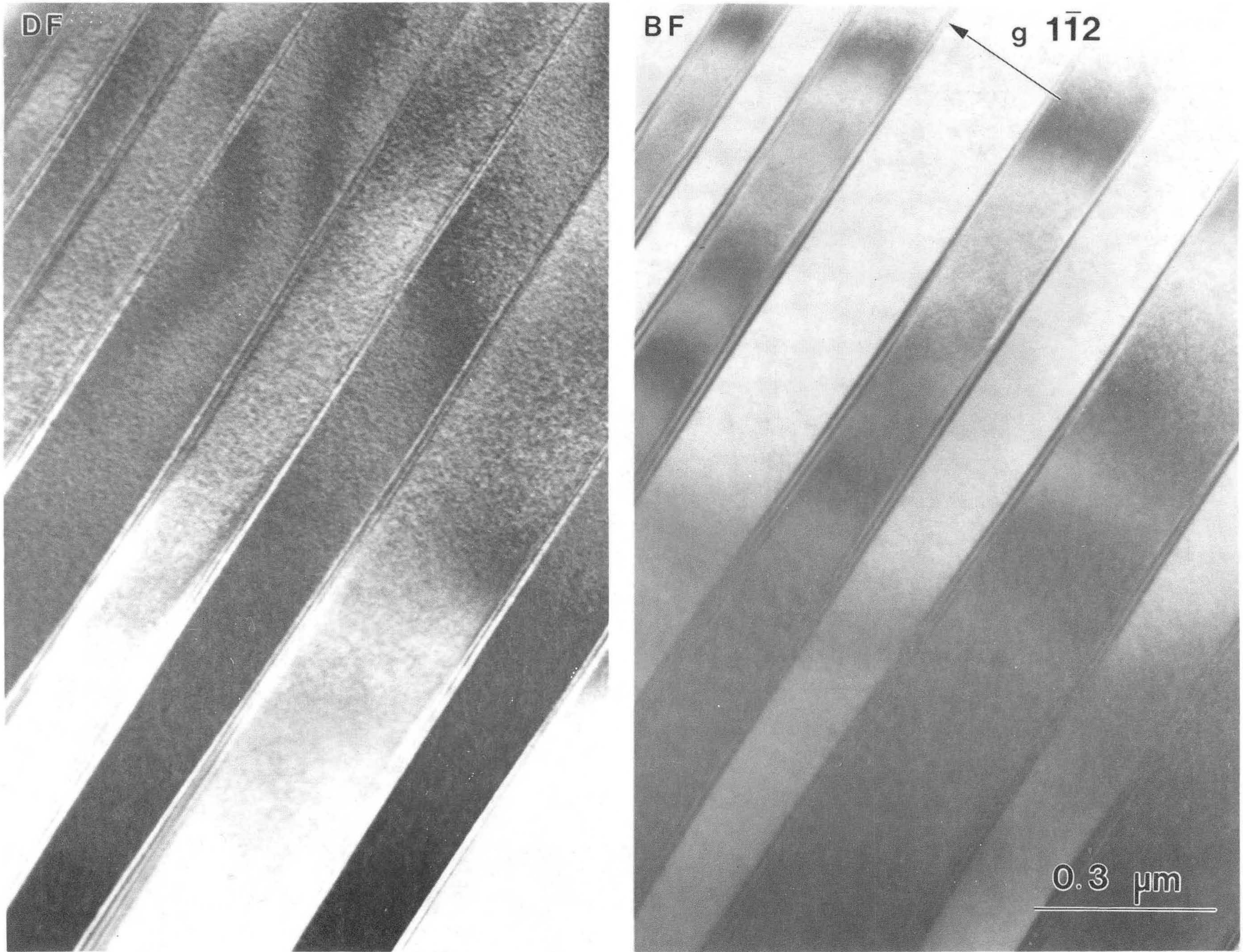
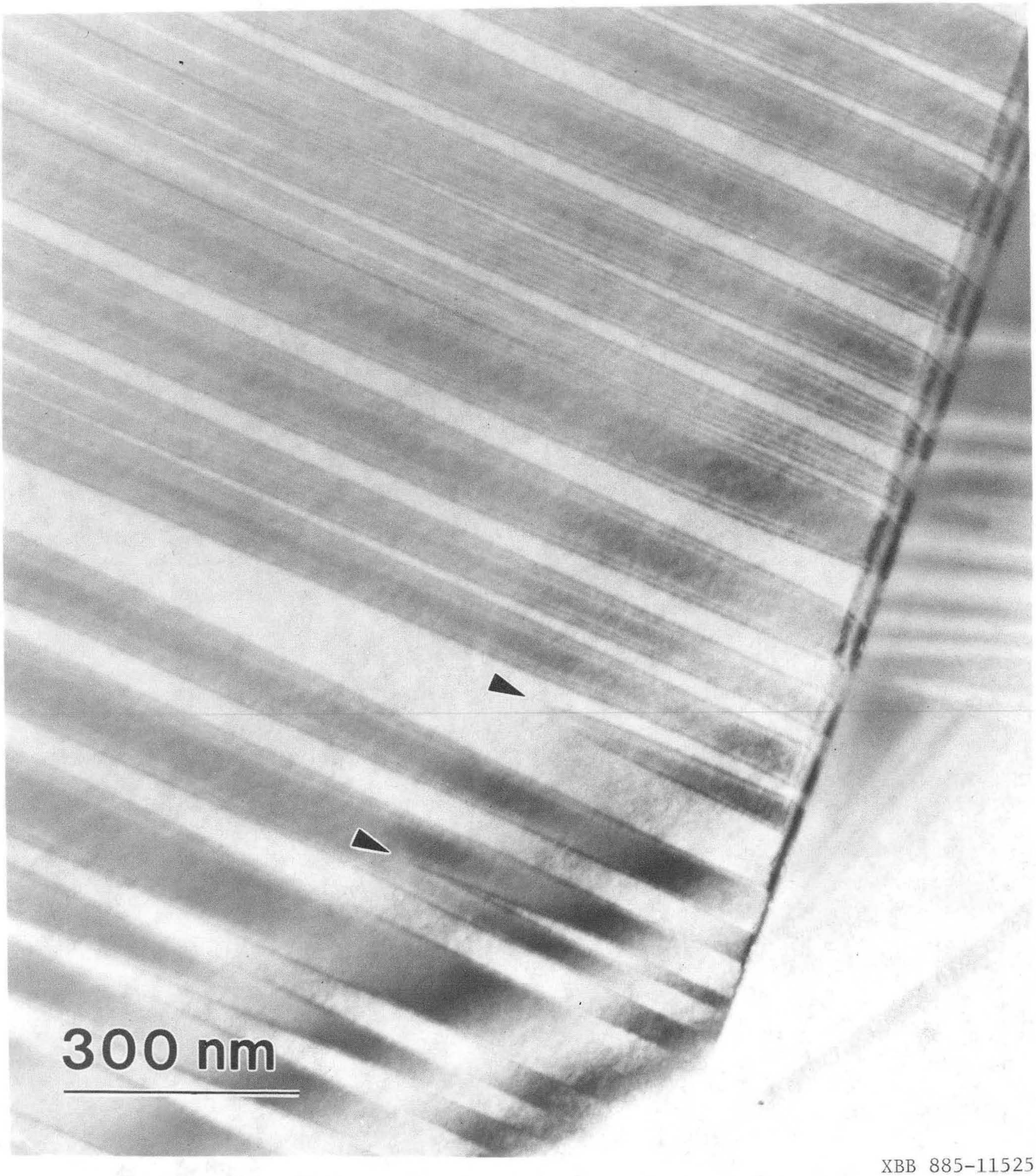


Fig 2



XBB 885-11525

Fig 3

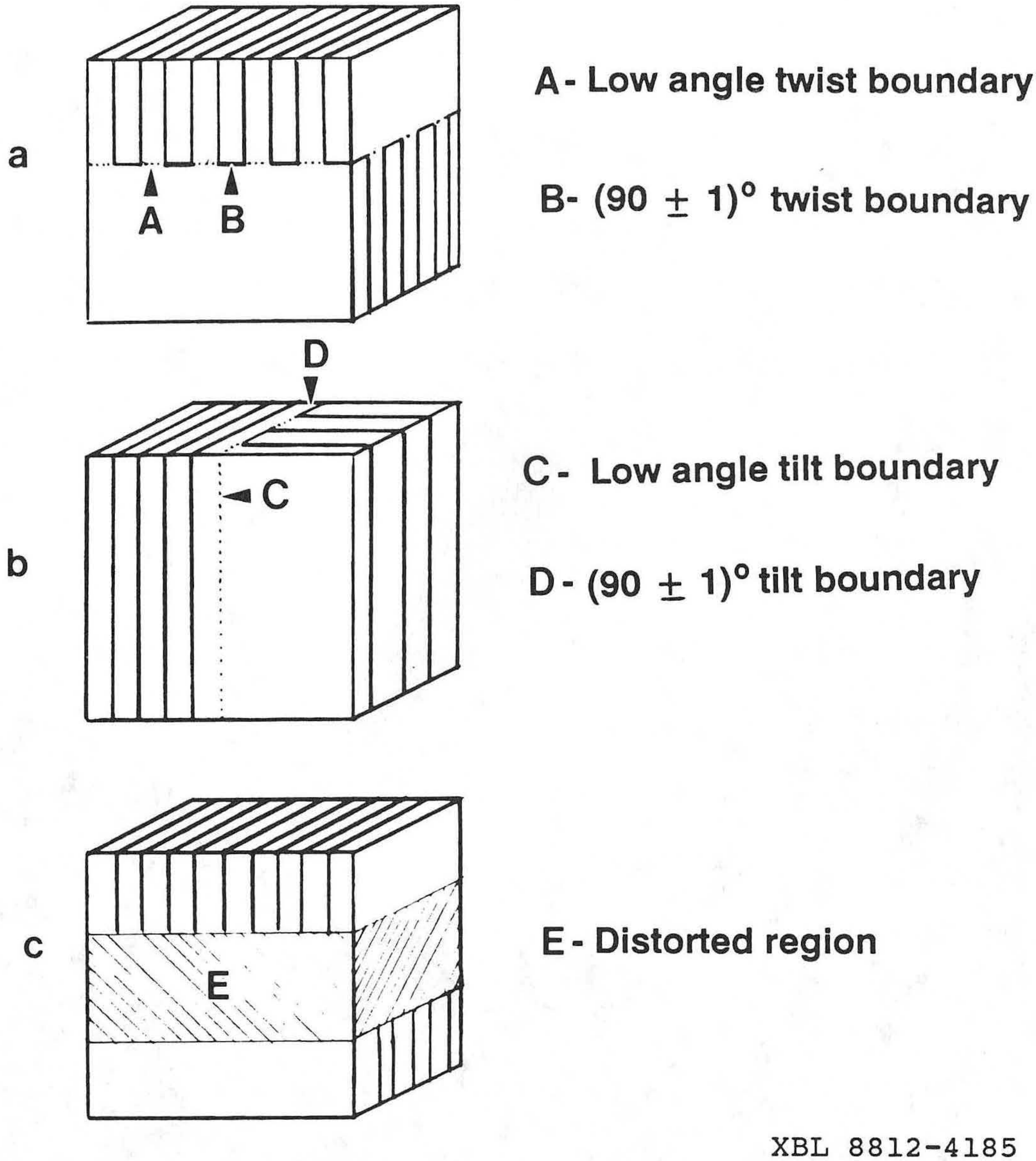
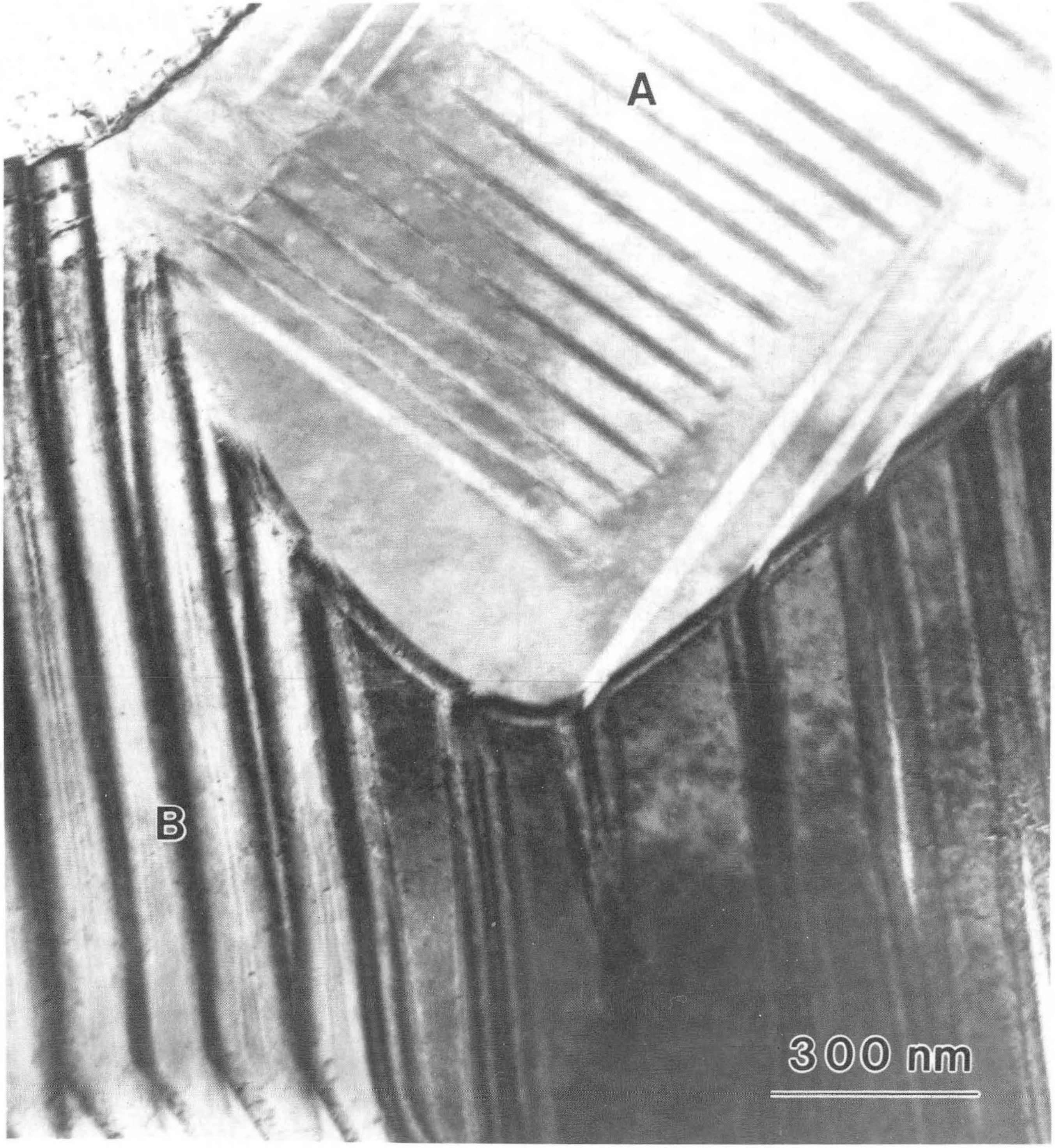
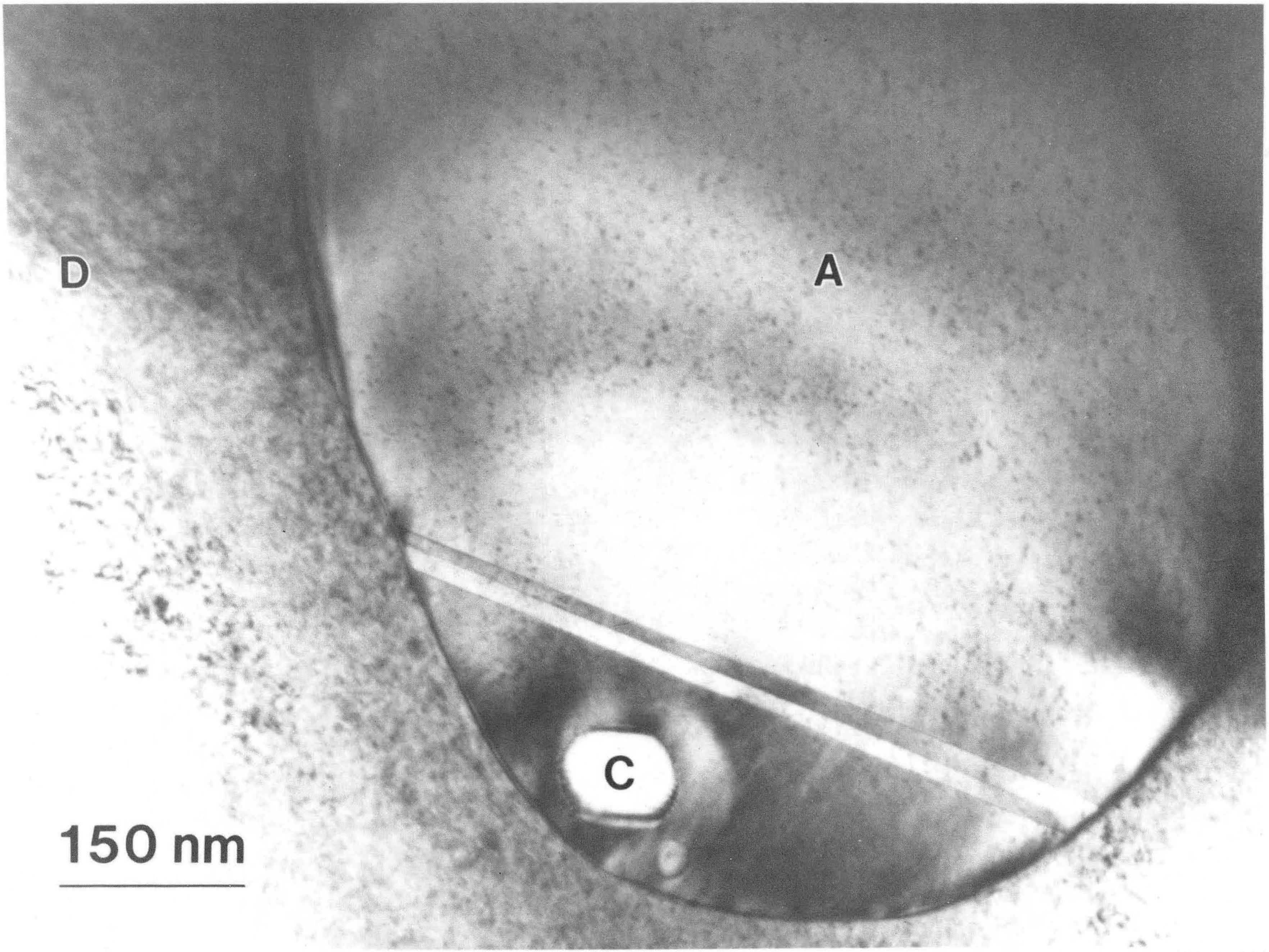

a
A
B
A- Low angle twist boundary
B- (90 ± 1)° twist boundary
D
C
b
C- Low angle tilt boundary
D- (90 ± 1)° tilt boundary
E
c
E- Distorted region
XBL 8812-4185

Fig 4



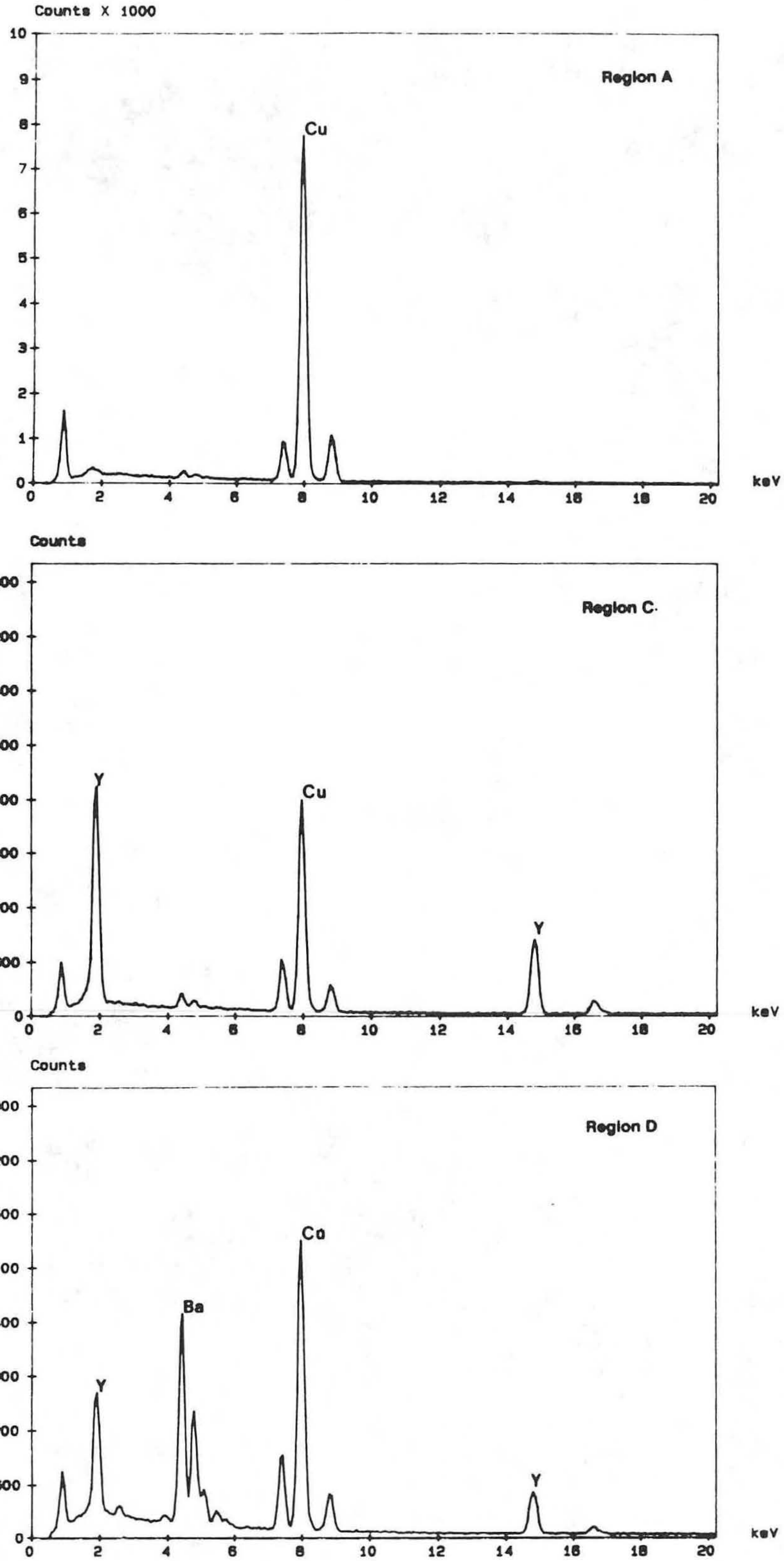
XBB 889-9210A

Fig 5



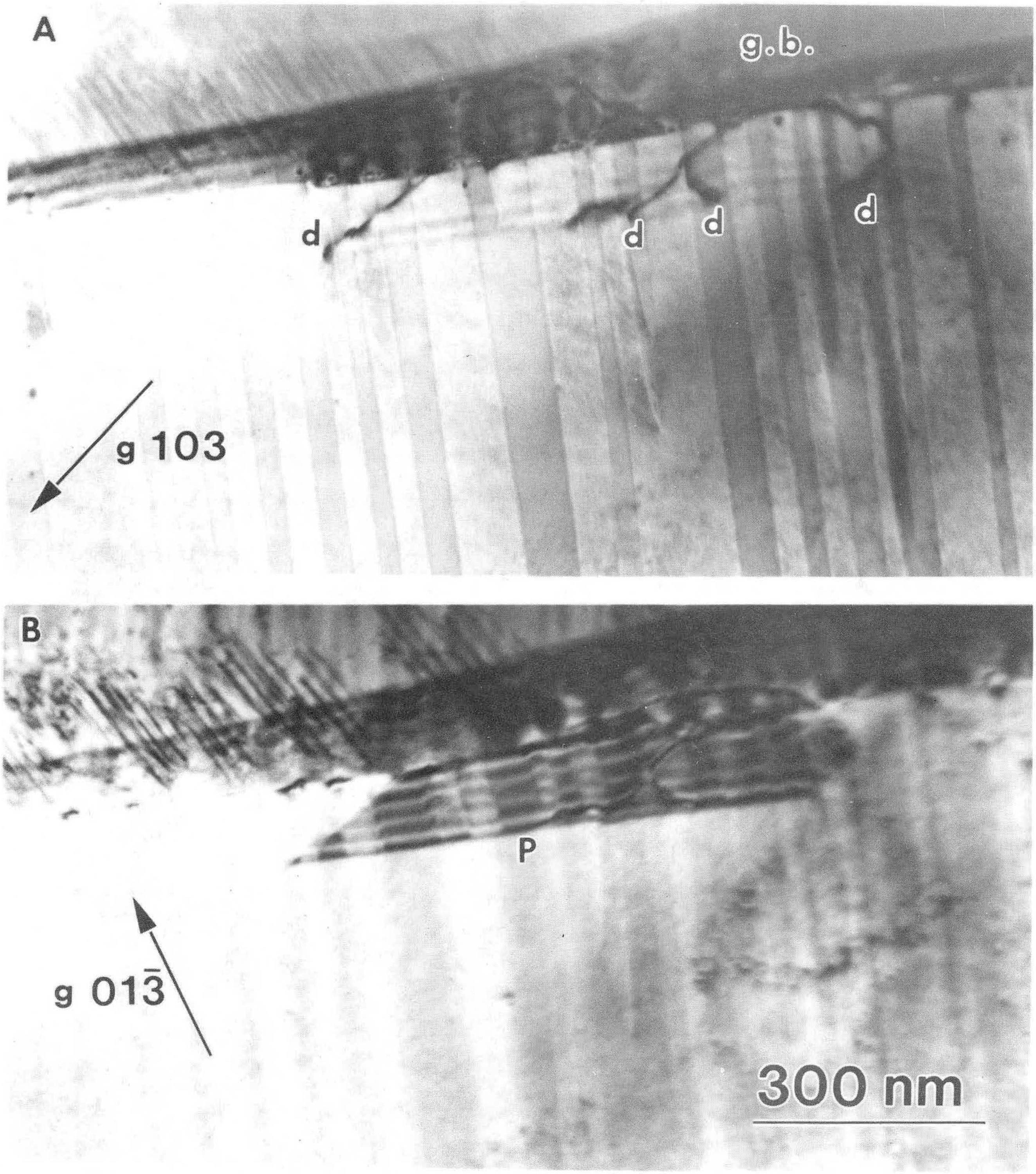
XBB 889-11524

Fig 6



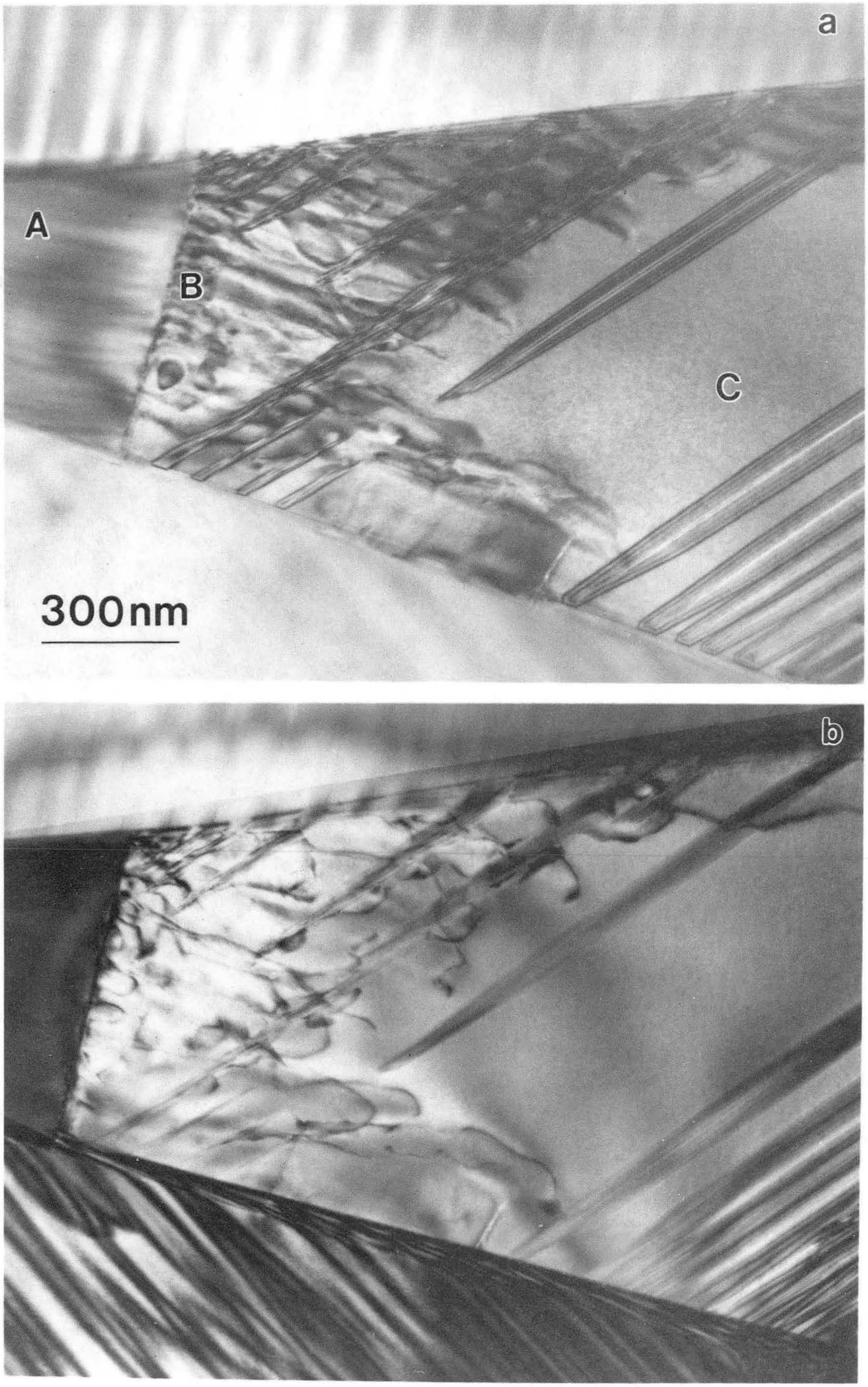
XBL 8812-4183

Fig 6 cont.



XBB 880-11527

Fig 7



XBB 880-11526

Fig 8

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