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IDENTIFICATION OF INTERSTITIAL VS VACANCY TYPE DISLOCATION LOOPS IN ION IMPLANTED SILICON

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ABSTRACT

A quick method of distinguishing between vacancy type and interstitial type dislocation loops is described which is applicable to foils near ⟨111⟩ orientation and loops in the size range which gives rise to double-arc contrast. This method utilizes the Kikuchi Line pattern and some general rules concerning electron diffraction contrast from small loops. With this method, small prismatic loops in As^+ implanted silicon have been shown to be of interstitial type.

1. INTRODUCTION

Because of the growing application of ion-implantation to the manufacture of semiconductor devices, radiation damage in these materials is of practical as well as theoretical interest.

One of the known effects of the radiation damage which accompanies ion implantation is the formation of dislocation loops during post implantation heat treatment. To better understand the nature of the radiation damage and the changes that take place on subsequent heating it is necessary to identify these loops as either vacancy or interstitial type.

It has been pointed out[1,2,3] that for large inclined loops of known slope (usually determined by slip trace or high angle tilting method), it is easy to distinguish between the two types by tilting experiments that cause the diffraction contrast image to flip from inside to outside of the dislocation line. However, for small loops this method is not applicable. In this paper a very convenient method of distinguishing between small vacancy and interstitial loops is described.

For As^+ (1×10^{14} nvt) implanted silicon, the perfect loops formed after annealing at 800°C are approximately 100 Å in diameter. They give rise to double-arc type diffraction contrast. The two beam electron diffraction contrast image has an out of contrast line which lies along one of the $\langle 110 \rangle$ directions in the loop plane. The Burgers vectors for this kind of loop are inclined to the loop plane, lying along the $\langle 110 \rangle$ direction that is perpendicular to the out of contrast line. The details of double-arc contrast from dislocation loops in F.C.C., B.C.C. and D.C. crystals were first described by G. Thomas et al.[7]

In this paper, some simple rules are presented concerning contrast from small loops which enable the determination of loop type when the Burgers vector of the loop is inclined to the plane of the loop. Also an easy method of determining the sign of the diffraction vector, $\vec{g}$, for foil orientation near [111] has been described using the Kikuchi line pattern. This permits a quick and reliable determination of the interstitial or vacancy character of loops in any specimen that gives a Kikuchi line pattern.

Because of relatively great foil thickness, the Kikuchi pattern is usually well developed for silicon electron microscope specimens.

In this paper, the method has been applied to specimen containing loops that had already been identified as interstitial type by previously established procedures.

2. DIFFRACTION CONTRAST FROM SMALL LOOPS

The diffraction contrast technique for loop type determination was first applied to loops formed during the deformation of magnesium oxide.[1] Latter it was widely used in determining loop types in quenched aluminium[2,3] and neutron irradiated molybdenum.[10]

This procedure has been described in detail by Groves and Kelly,[1] Ruedl et al.[9] and Mazey et al.[3] The principle is shown in Fig. 1. It is purely a geometrical problem to distinguish between the two types once the inclination of the loops relative to the direction of the diffraction vector, $\vec{g}$ vector, and the conditions for inside and outside contrast are known. The method is apparently not applicable to loops that lie parallel to the plane of the foil.

We follow the FS/RH (perfect crystal) convention of P. B. Hirsch et al.[11] in defining Burgers vectors. (i.e. Assume the positive direction along the dislocations is always into the paper for the edge of the loop which is farthest to the right (Fig. 1).) Stated below are some general rules for contrast from loops.

1. $(\vec{g}\cdot\vec{b}) < 0$ For outside contrast (keeping s always positive)

   $(\vec{g}\cdot\vec{b}) > 0$ For inside contrast (keeping s always positive)

2. $\vec{b}\cdot\hat{n} > 0$ For vacancy type.

   $\vec{b}\cdot\hat{n} < 0$ For interstitial type.

In assigning the plane normal $\hat{n}$, we adopt the convention that it always makes an obtuse angle with the beam direction.

These rules can be applied to loops that lie on the foil plane provided that the Burgers vector is inclined to it.

Following these rules, the sense of the Burgers vector of a loop is determined if the $\vec{g}$ vector is known. The plane normal is fixed according to the convention, if the beam direction through the foil is known. Once the senses of the Burgers vector and the plane normal are known, loop type is uniquely determined by the second rule.

Some confusion has arisen concerning how to uniquely determine the $\vec{g}$ vector and the plane normal. Completely incorrect results in loop type determination can be found in the literature.[12,13]

3. METHOD OF CORRELATING THE $\vec{g}$ VECTOR WITH THE FOIL ORIENTATION

The Kikuchi pattern gives the orientation unambiguously and has long been used as a convenient road map for finding poles and for determinating the sense of s (the deviation parameter from the exact Bragg condition). But no one to the knowledge of the authors has ever pointed out the fact that it also uniquely correlates the indices of $\vec{g}$ vectors to the foil orientation. A more complete discussion will be published elsewhere.[8]

The method we used for foil orientations near $\langle 111 \rangle$ was to orient a Thompson tetrahedron on the Kikuchi map with [111] pointing up and with its corners pointing away from the $\langle 112 \rangle$ poles. Then index all $\vec{g}$ vectors assuming the [111] orientation is pointing upward. Or equivalently, we can reverse the Thompson tetrahedron and with its corners pointing toward $\langle 112 \rangle$ poles index all $\vec{g}$ vectors as if $[\bar{1}\bar{1}\bar{1}]$ is up. (Fig. 2) These are the only two possibility and if the conventions and rules previously described are followed both lead to the same result.

4. SPECIMEN PREPARATION

One {111} high resistivity p-type silicon slice was implanted with $2\times10^{14}/cm^2$ 100 KeV phosphorus ions at room temperature and one slice of low pax {111} p-type silicon was implanted with $1\times10^{14}/cm^2$ 100 KeV arsenic ions at room temperature. Both implantations were carried out by the Fairchild Camera and Instrument Corporation.

The implanted slices were then cut into discs 2.2 mm in diameter and annealed in high vacuum (10^{-6} Torr) for half an hour to one hour. They were then chemically thinned in one part A solution (2.5 gram iodine and 1100 cc CH_3COOH) and two parts solution B (1 HF + $3HNO_3$) for electron microscopy observations.

5. EXPERIMENTS

A. P^+ Implanted Si after $\frac{1}{2}$ Hour Annealing at 750°C:

For phosphorous implanted silicon, there were many bar like defects along ⟨110⟩ extending through the foil in the thin area like those found in boron ions implanted silicon.[5] In a dark field picture with $s>0$, the top and bottom of the foil can be determined by the method first pointed out by G. Thomas et al.[7] The Thompson tetrahedron could be either oriented with $[\bar{1}\bar{1}\bar{1}]$ up or with [111] up in exactly the same way as with the Kikuchi pattern as shown in Fig. 3.

From the sequence pictures in Fig. 4, it is clear that no matter which foil orientation we assumed, once a self consistent system is used the three sets of main double-arc dislocations A, B, C, are all interstitial in nature as reported elsewhere.[4,6]

0 0 0 0 3 9 0 0 7 9 7

The relationships are as follows:

Table 1.

loops	Foil orientation	$(\vec{g}\cdot\vec{b})$	$\vec{g}$	$\vec{b}$	$\hat{n}$	$\hat{b}\cdot\hat{n}$	loop type
A	$\bar{1}\bar{1}\bar{1}$	outside	$20\bar{2}$	$\frac{a}{2}[011]$	$\bar{1}\bar{1}\bar{1}$, $\bar{1}\bar{1}1$, $1\bar{1}\bar{1}$, $\bar{1}1\bar{1}$	-	interstitial
B	$\bar{1}\bar{1}\bar{1}$	inside	$20\bar{2}$	$\frac{a}{2}[110]$	$\bar{1}\bar{1}\bar{1}$, $\bar{1}\bar{1}1$, $1\bar{1}\bar{1}$, $\bar{1}1\bar{1}$	-	interstitial
C	$\bar{1}\bar{1}\bar{1}$	outside	$02\bar{2}$	$\frac{a}{2}[101]$	$\bar{1}\bar{1}\bar{1}$, $\bar{1}\bar{1}1$, $1\bar{1}\bar{1}$, $\bar{1}1\bar{1}$	-	interstitial
A	111	outside	$20\bar{2}$	$\frac{a}{2}[\bar{1}\bar{1}0]$	111, $11\bar{1}$, $1\bar{1}1$, $\bar{1}11$	"-"	interstitial
B	111	inside	$20\bar{2}$	$\frac{a}{2}[0\bar{1}\bar{1}]$	111, $11\bar{1}$, $1\bar{1}1$, $\bar{1}11$	-	interstitial
C	111	outside	$2\bar{2}0$	$\frac{a}{2}[\bar{1}0\bar{1}]$	111, $11\bar{1}$, $1\bar{1}1$, $\bar{1}11$	-	interstitial

B. As^+ ($1\times10^{14}/cm^2$) Implanted Si After One Hour Annealing at 800°C

The defect structure in As^+ implanted silicon has no bar-like defects so that the Kikuchi map method must be used in determining loop type. All loops proved to be of interstitial type as in P^+ implanted Si. (Fig. 5)

6. DISCUSSION AND SUMMARY

A simple method for determining loop type in foils near [111] orientation has been described and it has been shown to give the same results as previously used more cumbersome methods by applying it to phosphous and arsenic implanted silicon.

The advantages of this method over others are:

(a) Only small angle tilting is necessary to know the sense of the Kikuchi pattern near $\langle 111 \rangle$. No high angle tilting[3] or dark field top-bottom determinations[6,7] are needed.

(b) Only plus $\vec{g}$ and minus $\vec{g}$ bright field photographs are required.

This method is particularly useful in ion implantation studies with orientation near $\langle 111 \rangle$ because silicon usually gives very clear Kikuchi patterns. Even without a clear Kikuchi pattern, the second Laue zone method[14] can be used to reveal the sense of the Kikuchi pattern which is all that is needed.

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FIGURE CAPTIONS

Fig. 1. Relationship of diffraction contrast and associated $\vec{g}$, $\vec{b}$, $\vec{n}$... The Burgers vectors defined here are following FS/RH (perfect crystal) method as described in Ref. 11.

Fig. 2. Correlation of Kikuchi pattern with respect to $\vec{g}$ and $\vec{n}$.

Fig. 3. Dark field picture of P^+ (2×10^{14} nvt) implanted Si annealed at 750°C for 1/2 hr. Top-bottom (T-B) of bar-like defects and correlation of the Kikuchi map with the {111} tetrahedron are also shown.

Fig. 4(a-d). Sequent S.A.D. and bright field pictures of P^+(2×10^{14} nvt) implanted Si annealed at 750°C for 1/2 hr.

Fig. 5(a-c). Sequent S.A.D. and bright field pictures of As^+(1×10^{14} nvt) implanted Si annealed at 800°C for one hour.

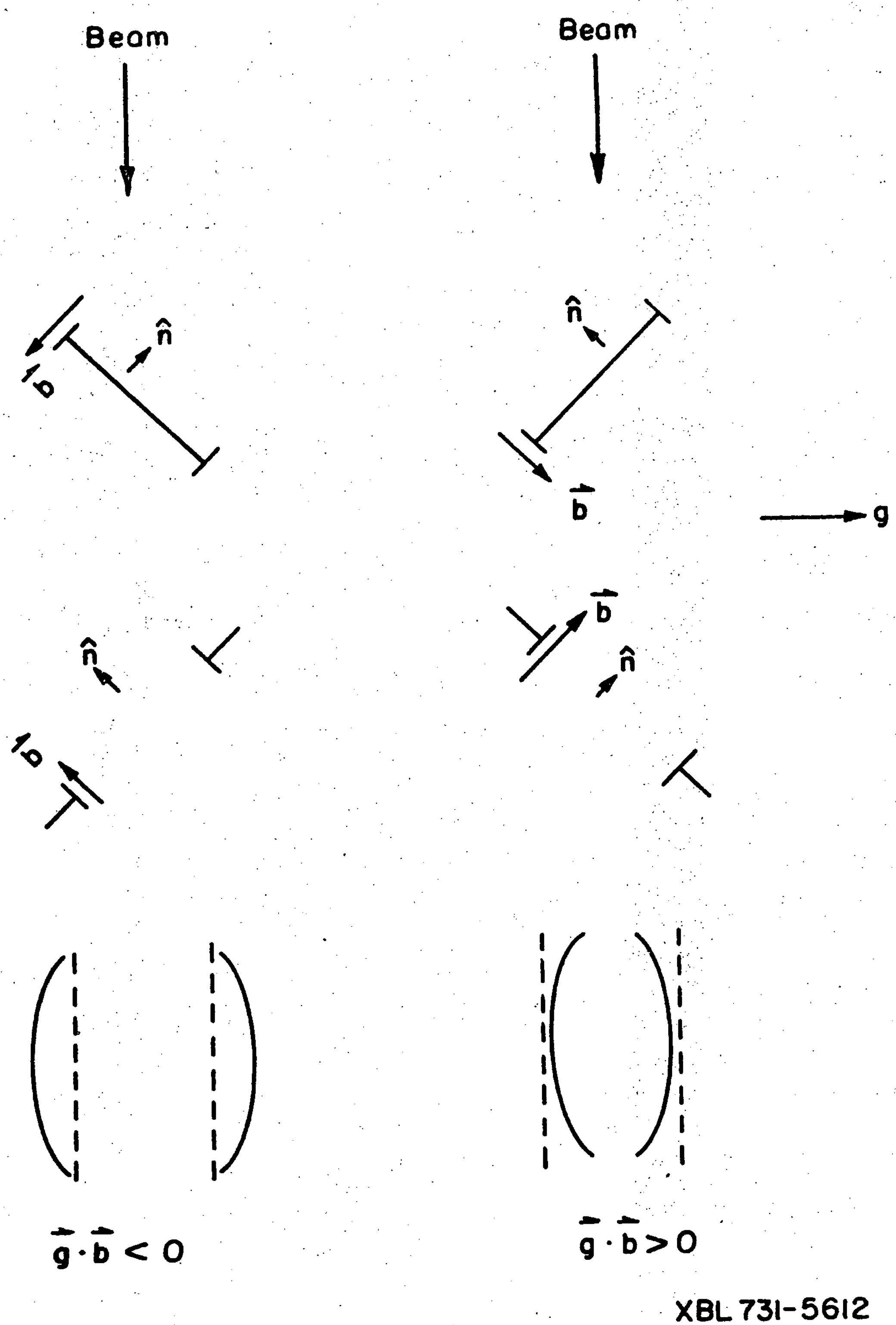
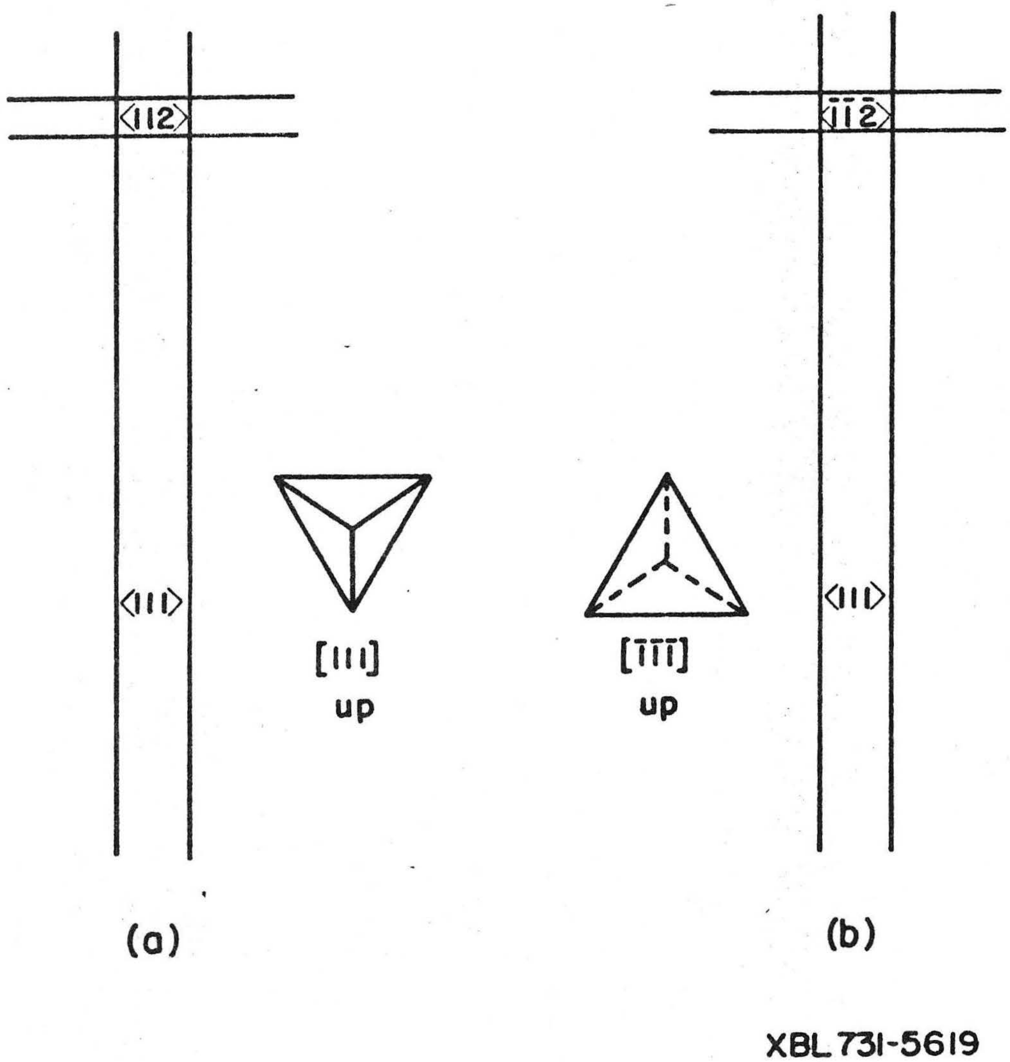
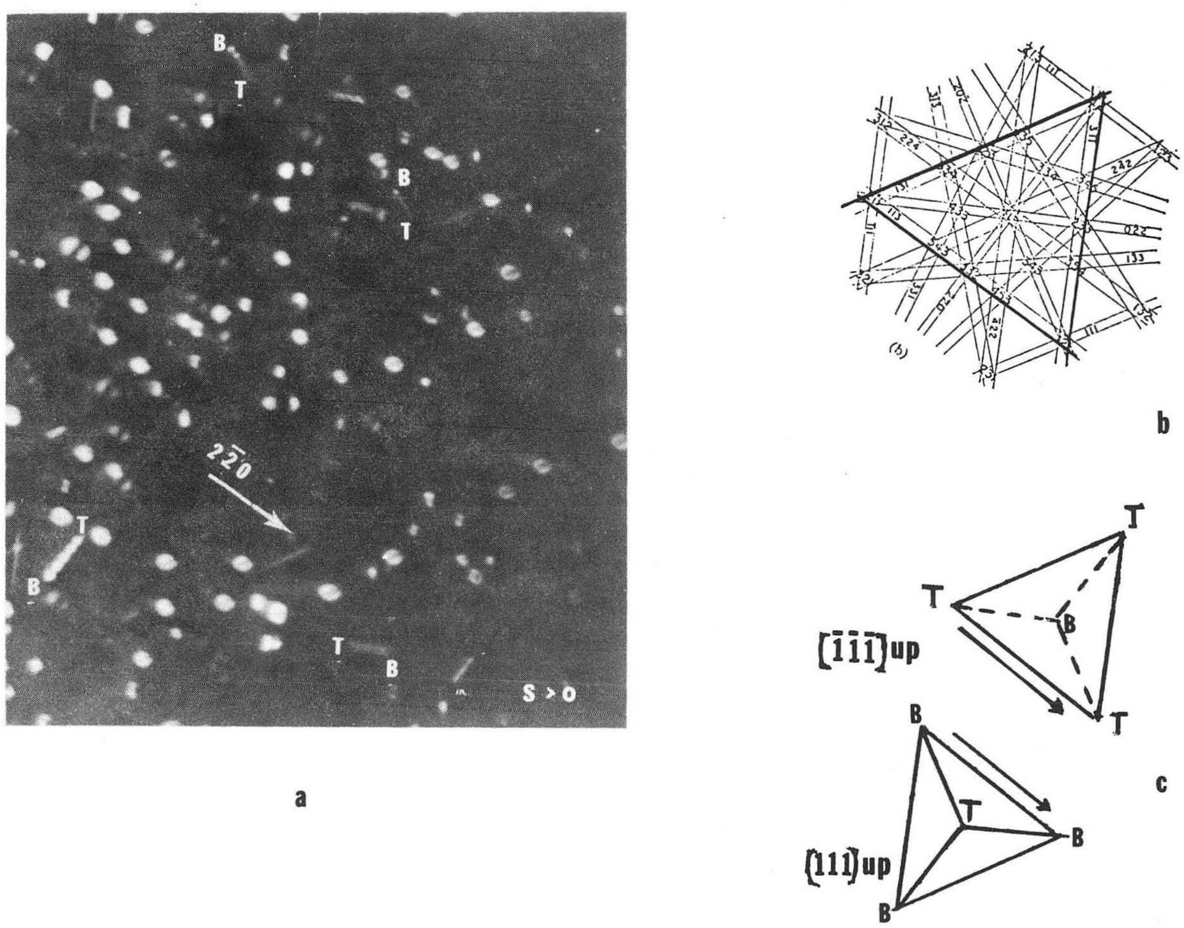

Beam
Beam
n̂
b
n̂
b
g
n̂
b
b
n̂
$\vec{g}\cdot\vec{b} < 0$
$\vec{g}\cdot\vec{b} > 0$
XBL 731-5612

FIG. 1



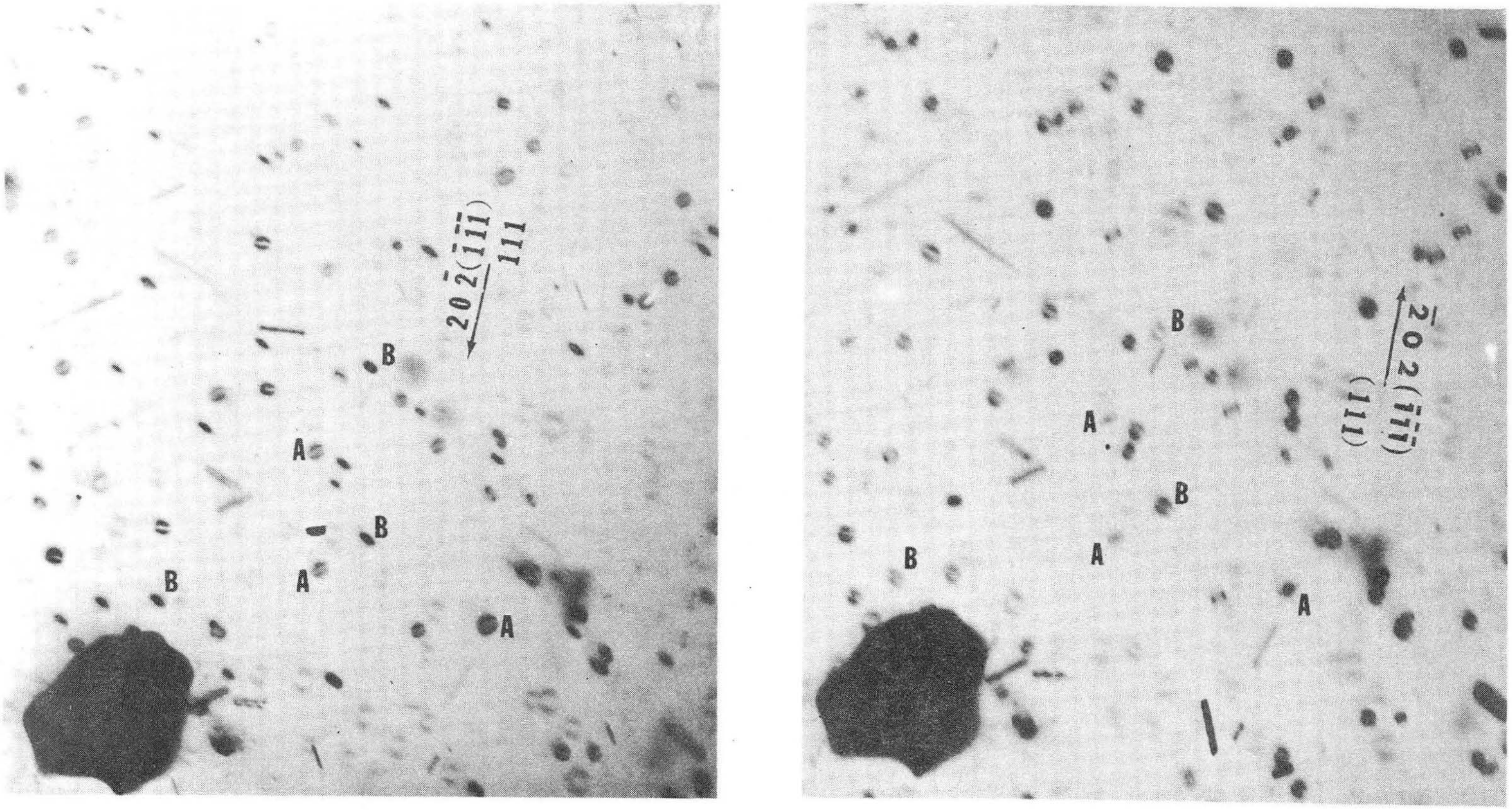
$\langle 112 \rangle$
$\langle \bar{1}\bar{1}\bar{2} \rangle$
$\langle 111 \rangle$
[111]
up
$[\bar{1}\bar{1}\bar{1}]$
up
$\langle 111 \rangle$
(a)
(b)
XBL 731-5619

FIG. 2



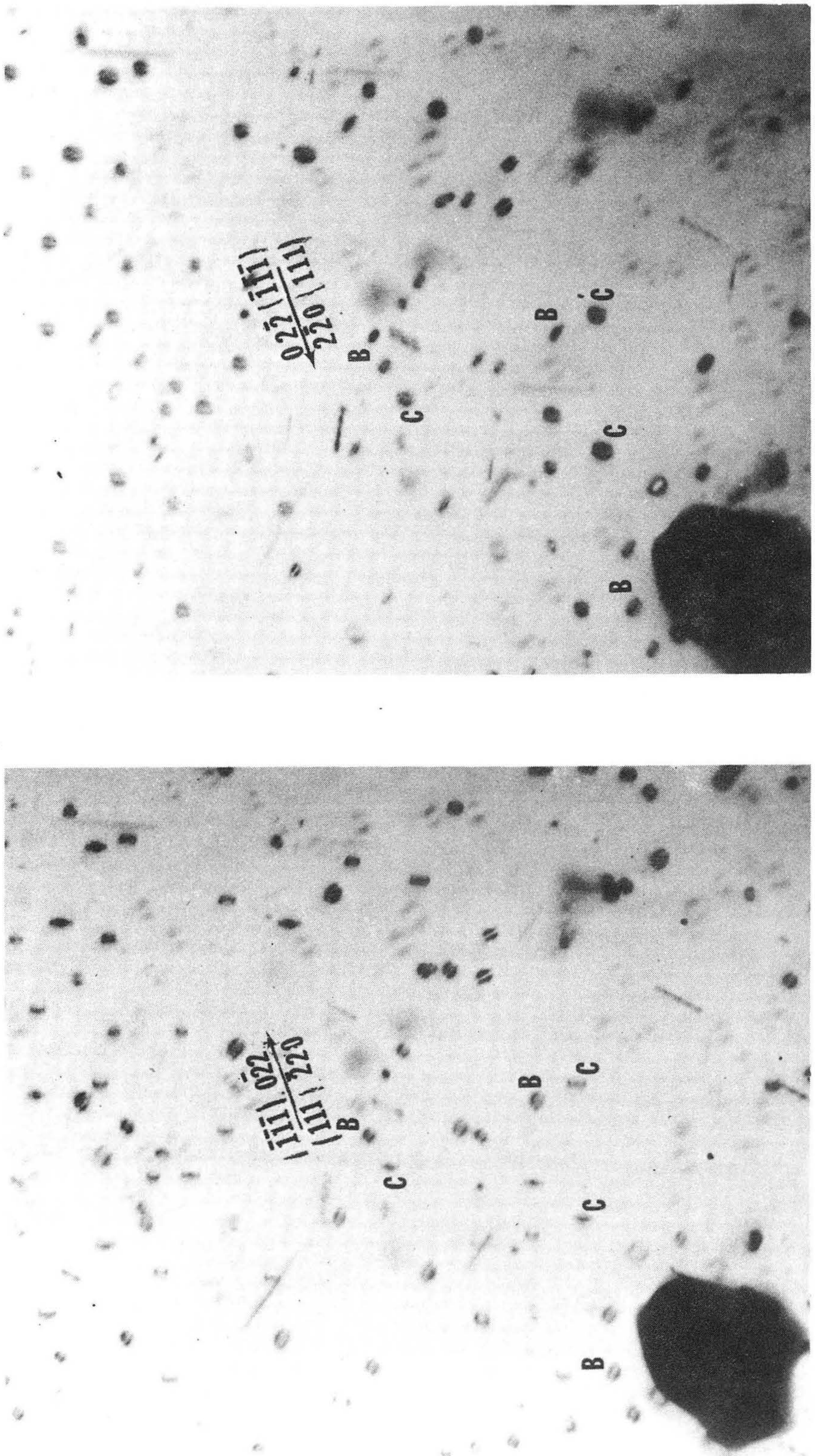
XBB 736-3978

Fig. 3



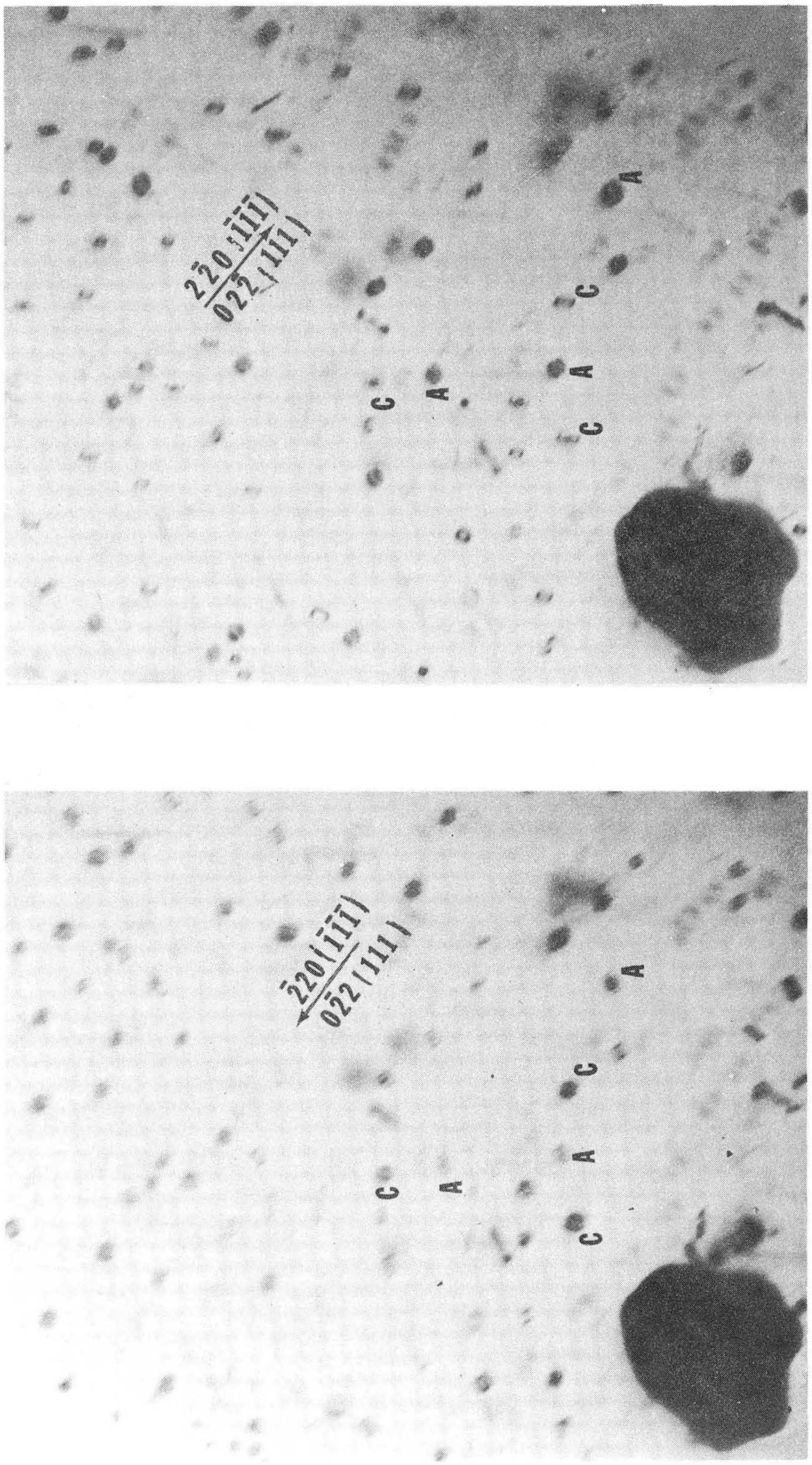
XBB 7212-6229

Fig. 4a



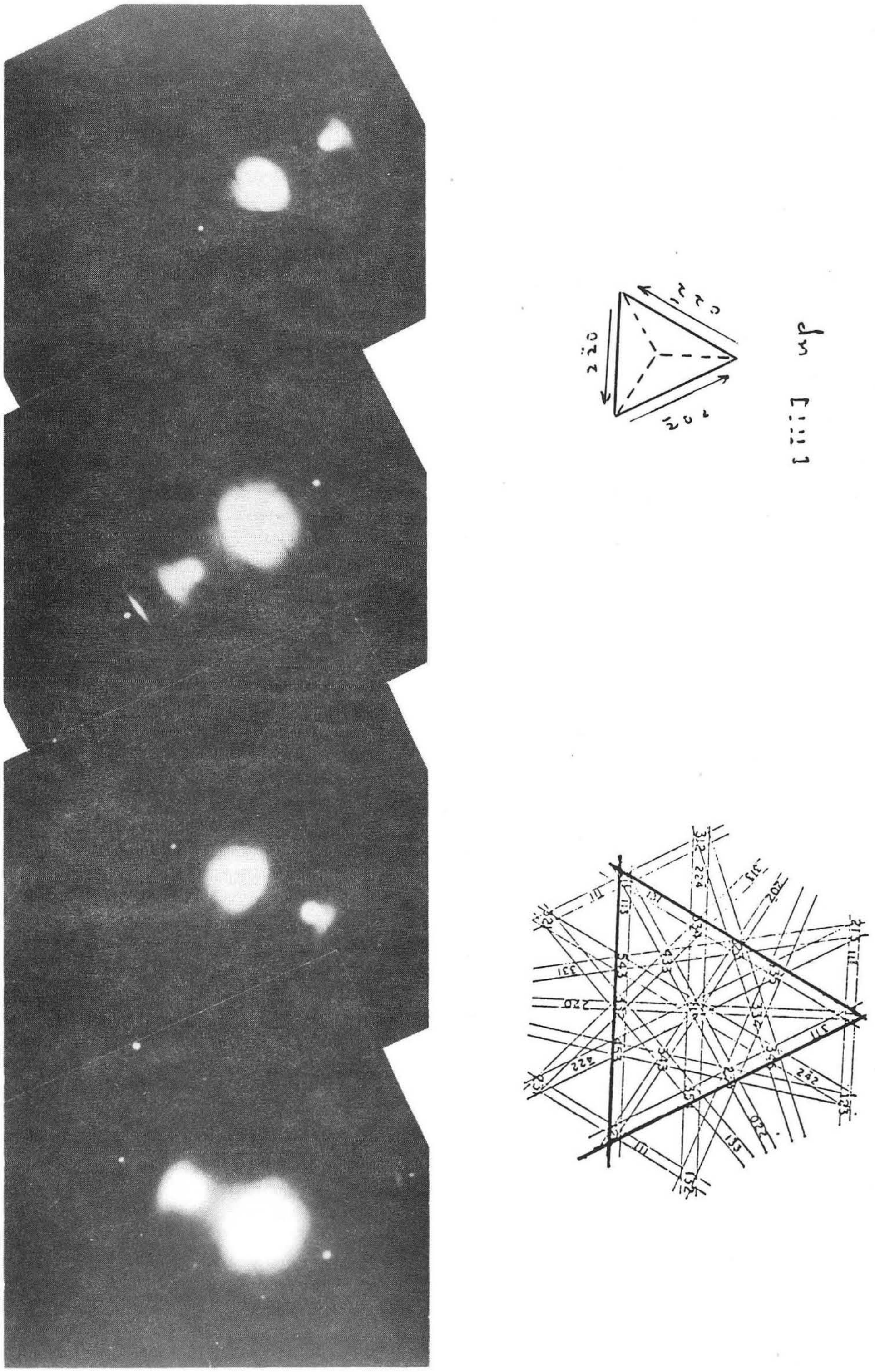
XBB 7212-6226

Fig. 4b



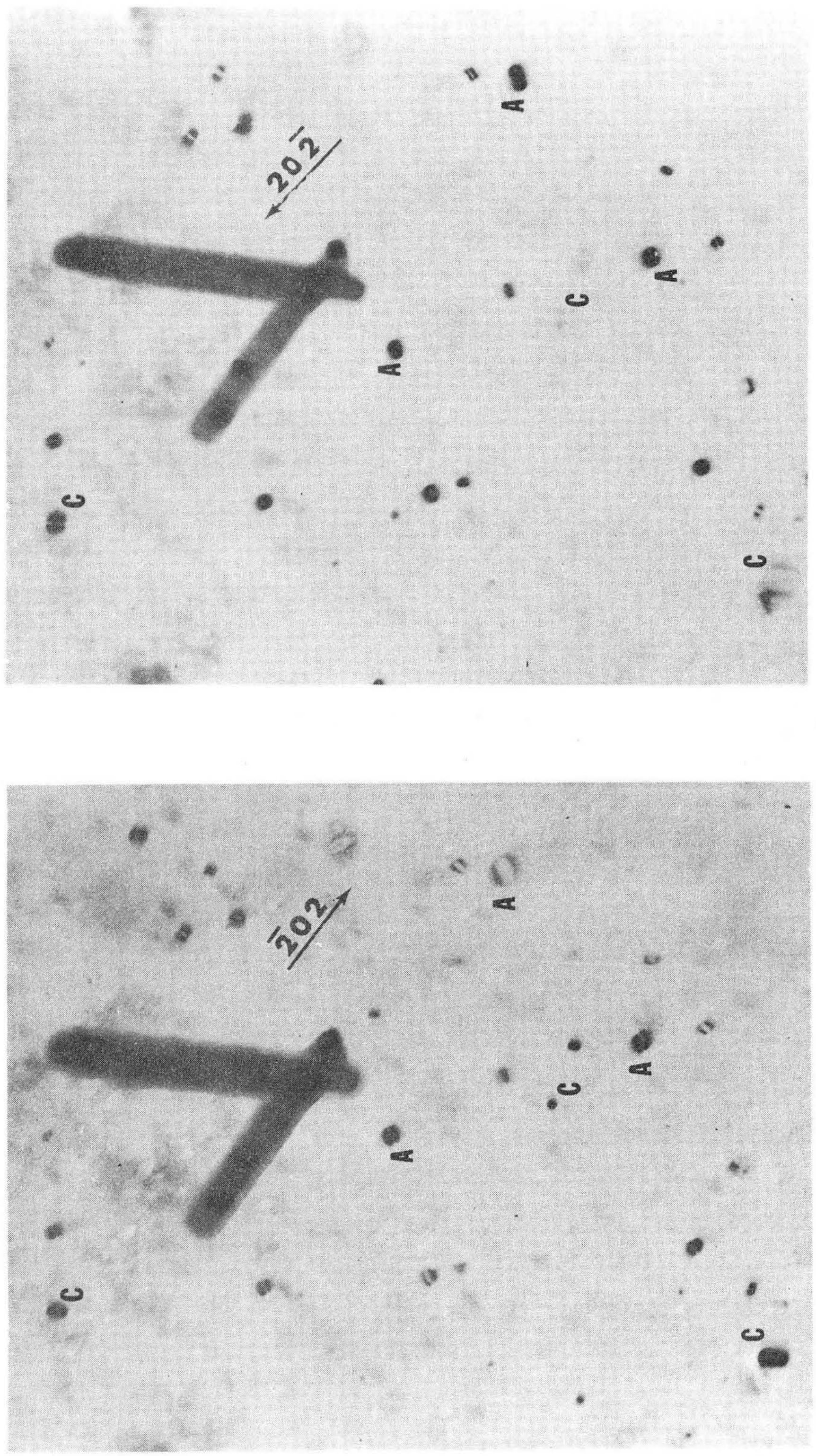
XBB 7212-6224

Fig. 4c



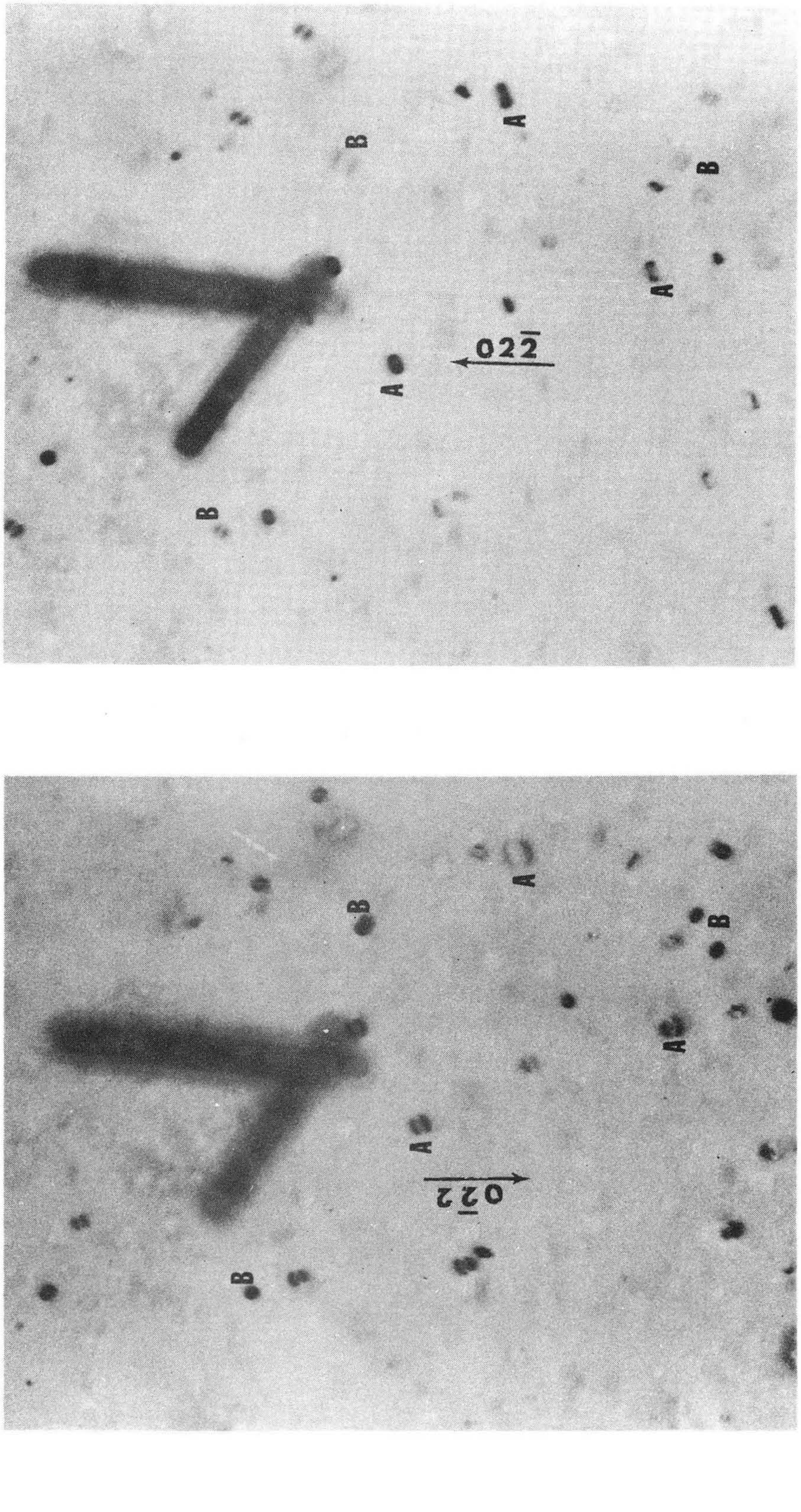
XBB 7212-6234

Fig. 5a



XBB 7212-6225

Fig. 5b



XBB 7212-6227

Fig. 5c