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ABSTRACT

Contrary to a common assumption that the Kikuchi line spacing should be the same as that of the corresponding Bragg spot pattern if diffraction geometry is properly considered, experimental results obtained by using high purity silicon single crystal specimens at 100 kV show that the Kikuchi line spacing can be different from that of the corresponding Bragg spot pattern depending on the specimen thickness.

This result is explained on the basis of dynamical scattering of Kikuchi electrons.

I. INTRODUCTION

It is well known that in transmission electron diffraction experiments for most crystals the diffraction pattern consists of Bragg spots as well as Kikuchi lines (or bands). Kikuchi (1928) interpreted these lines as the result of subsequent Bragg rescattering of inelastic electrons. An electron beam which is incident on an inelastic scattering center is scattered and the angular intensity distribution of these inelastically scattered electrons is then peaked in the direction of the incident beam and decreases monotonically with increasing scattering angle. Variations in this intensity distribution can occur because these electrons on traveling through the rest of the crystal in certain directions satisfy the Bragg law of diffraction and this results in an excess or deficiency in the number of inelastic electrons in these directions. The subsequent

Bragg rescattering after the generation of these inelastic electrons produces a pair of cones of rays with a semi-vertex angle of $90^{\circ}-\theta_B$ (where θ_B is the Bragg angle), along which the excess or deficient electrons occur. Associated with each (hkℓ) diffraction is a corresponding pair of cones of rays which intersects the viewing plate or screen and produces a pair of hyperbola, which can be closely approximated to straight lines due to the small values of θ_B for fast electrons. When kinematical diffraction conditions are satisfied for the incident elastic beam, it can be easily shown that the line closer to the transmitted Bragg spot, or the (000) line, is deficient in electrons, whilst the other line of the pair, the (hkℓ) line consists of an excess of electrons. The spacing between the two lines of the pair corresponding to the angular separation of the two associated cones of rays, is usually that corresponding to $2\theta_B$, and is considered to be invariant(e.g. for review see Thomas, 1969).

Although the geometry and contrast (excess or deficiency of electrons) were explained by this simple theory for the two beam kinematical case for elastic electrons, more intricate situations arise if dynamical scattering conditions are satisfied or many beam interactions occur. Some of the more important features due to these effects include the presence of a band structure between the lines, and its changing from an excess to a deficiency in electrons in the simultaneous many beam case with decrease in accelerating voltage or increase in specimen thickness (Boersch 1937 and Pfister 1953). Subsidiary maxima of Kikuchi lines and bands have been observed by many authors, as described by Uyeda et al. (1954).

The interchange from a deficient to an excess of electrons and vice versa between the pair of (000) and (hkℓ) lines was recently investigated by Thomas and Bell (1968). We present in this paper a new effect which is dynamical and unusual, viz., the Kikuchi line spacing variations with respect to specimen thickness.

II. EXPERIMENTS

Careful experiments were performed using high purity silicon single crystals. Thin foils were prepared in the usual way by chemical polishing. A Siemens Elmiskop 1A electron microscope was used, operated at 100 kV.

Original observations were made during experiments designed to study Kossel-Mollenstedt fringes in the elastic beams, hence a fully focused condenser II lens and a 400 μm condenser aperture were used. Later experiments to determine that the convergent nature of the beam was not responsible for the observed behavior of the Kikuchi pairs involved using less focused or fully defocused illumination. The use of convergent illumination proved to be an advantage in that foil thicknesses could be determined from the spacings of the Kossel-Mollenstedt fringes (Bell and Thomas 1969).

The smallest available intermediate aperture, 10 μm in diameter, was centered and used to obtain selected areas with the smallest variations in thickness, since small-angle wedge-shaped regions were found to yield Kossel-Mollenstedt fringes in the diffraction patterns. The smallest-bore standard projector pole piece was used to give maximum camera constant such that correctly exposed photographic records could be obtained in which the fringes were well resolved. The Miller indices of operating reflections were determined by comparing the rel-vector lengths to those of (111) or (220) reflections taken under identical conditions.

Upon obtaining a diffraction pattern, the foil was then tilted until a high index (hkℓ) diffraction spot was strongly excited with its (hkℓ) Kikuchi line passing through or near the center of the spot, i.e. two-beam conditions were satisfied for the elastic electrons. After the first desired result was recorded, a new selected area was obtained by translating the specimen and, if necessary, performing very small tilting adjustments to maintain diffracting conditions. At no time during a series were any changes made in lens currents. The only parameter being significantly altered was the foil thickness.

III. RESULTS

The series of photographs shown in Figure 1 were obtained using a (511) reflection of a silicon foil. The Kossel-Mollenstedt fringes are clearly visible inside the limits of the image of the condenser aperture. It is particularly easy to determine in Figure 1A that these fringes extend well outside the condenser aperture image, a region forbidden to elastic electrons. Hence these figures contain additional proof that the inelastic electrons behave dynamically in a manner closely similar to the elastic electrons. However, the interesting feature in these images is that the dark Kikuchi line associated with the (000) spot is in all cases at a distance from the diffracted line greater than would be the case if the angles between the cones of inelastic radiation were $2\theta_B$, the angle between the centers of the elastic rel-spots. Although slight reversals may occur, the general trend is that the black line approaches the white line as the foil thickness is increased. Using the Kossel-Mollenstedt fringes, the foil thickness for Figure 1a–e are respectively 1.8, 2.6, 3.3, 3.9 and 4.0 times the extinction distance for the (511) reflection. The calculated value for this extinction distance at 100 kV, including Debye-Waller effects, is 2500 Angstroms. Hence the thicknesses range from 4500 Angstroms to one micron.

Figure 2 illustrates that it is possible to cause the dark Kikuchi line to pass through and beyond the (000) spot by increasing the foil thickness, in this case for the (733) reflection. Figure 3 shows the simultaneous excitation of two high-index reflections. The spacing of the (440) Kikuchi pair is slightly smaller, and that of the $(1\bar{1}5)$ pair is considerably larger, than their corresponding rel-vector lengths. Ignoring simultaneous interactions amongst the inelastic electrons, this result indicates that both the thickness and the particular diffracting planes are important parameters in determining the degree of discrepancy between[8] Kikuchi pair spacing and that of the corresponding spots.

Figure 4 shows the behavior of the dark Kikuchi line with increasing thickness when fully defocused illumination is used to excite an (822) reflection in silicon. Kikuchi line spacings larger or smaller than the spot spacing can still be obtained.

IV. SUMMARY AND DISCUSSION

Contrary to the common assumption that the spacing of black and white Kikuchi lines should always be the same as that of the corresponding reciprocal lattice points, the present results have shown, for the first time, that this assumption is not always true. The spacing of these lines is in fact dependent upon the specimen thickness. Furthermore, the degree of discrepancy for a given thickness appears to be dependent upon the particular diffracting planes.

Since Kikuchi patterns are produced by inelastic electrons of smaller kinetic energy than the elastic electrons, it might be expected that the Kikuchi angles (Bragg angles for Kikuchi electrons) should always be somewhat larger than the corresponding Bragg angles for elastic electrons. However, this effect alone cannot explain the experimental results presented in this paper a) because this energy discrepancy is usually too small to produce any observable difference between the corresponding Kikuchi angles and the Bragg angles, and b) because of the observed results that the apparent Kikuchi angles are sometimes smaller than the appropriate Bragg angles. It may be concluded at this point that the observed specimen thickness dependent Kikuchi angle variations are due to dynamical effects involving at least four beams (two elastic beams and two inelastic beams). The effect can be expected to be even more profound if higher accelerating voltages are used to achieve a many beam dynamical case.

A theory explaining in detail this effect as well as that of the Kikuchi line intensity reversals and Kikuchi band formation will be presented in another paper. For purposes relevant to the experimental observations presented in this paper, the one parameter that is responsible for the behavior of the Kikuchi line associated with the transmitted spot is the anomalous absorption parameter. Just as for the elastic beams this parameter produces asymmetry in the bright-field rocking curve, so also does it produce asymmetry in the inelastic intensity distribution of the (000) Kikuchi line. The dark line observed is an intensity minimum whose position depends upon the foil thickness and the anomalous absorption length. Since this anomalous absorption length is not a constant parameter of the crystal but varies with the diffracting planes, the behavior of the lines in Figure 3 can be explained.

Although the results shown in this paper indicate that Kikuchi line spacings for high-index reflections may vary considerably if strong elastic beams are present and may not be suitable for measurements to obtain data such as lattice parameter variations or electron wavelength calibrations, crystal parameters such as the anomalous absorption length might be obtainable when sufficient data on thickness and line spacings are gathered.

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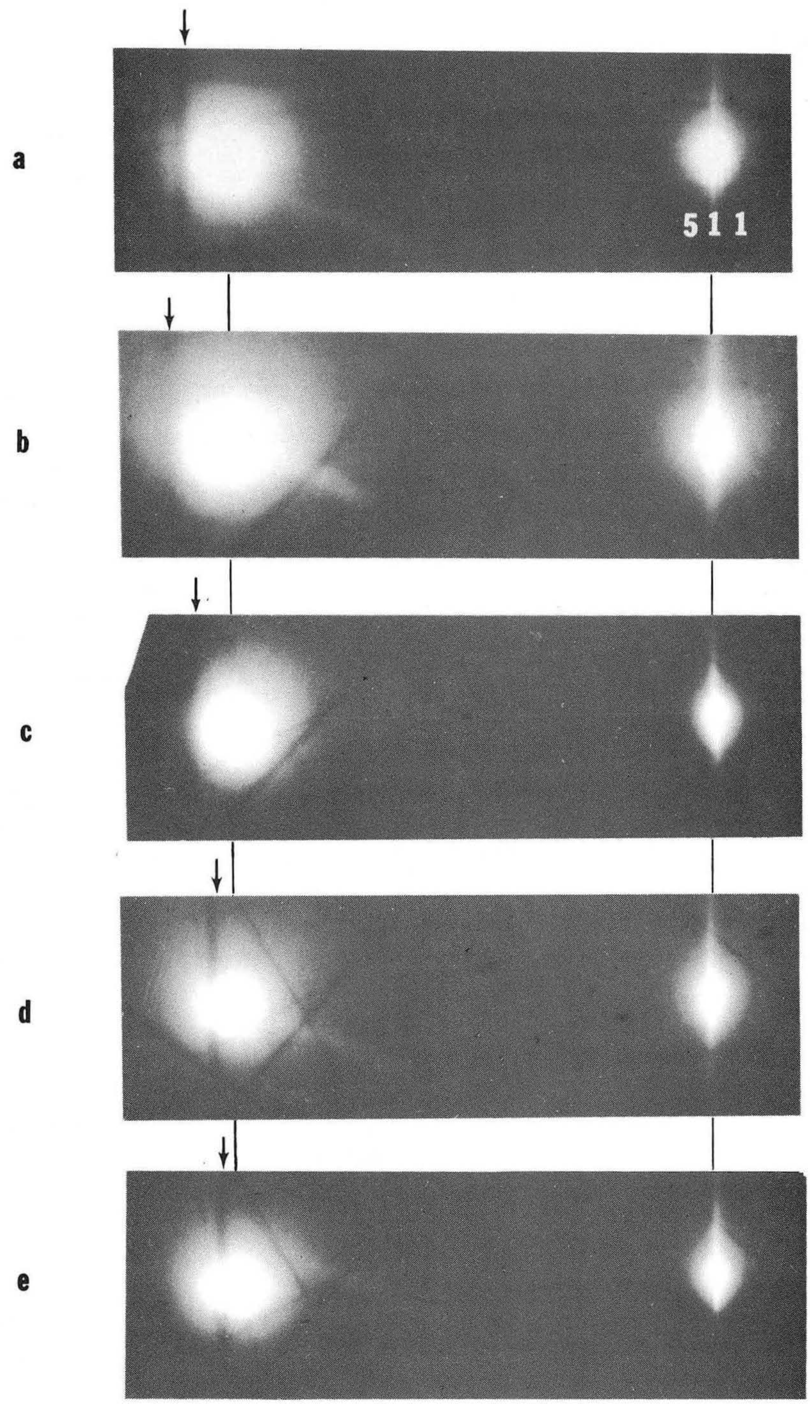
FIGURE CAPTIONS

Fig. 1. Observed silicon (511) Kikuchi line spacing variations with specimen thickness. The series of pictures are arranged vertically in order of increasing thickness, a-e. In all cases the Kikuchi line spacing is larger than that of the corresponding (511) Bragg spot pattern.

Fig. 2. Observed silicon (733) Kikuchi line spacing variations with specimen thickness. In case (a) the Kikuchi line spacing is larger than that of the (733) Bragg spot pattern and in cases (b) and (c) the Kikuchi line spacings are approximately equal to and smaller than that of the (733) Bragg spot pattern respectively. The diffraction patterns were obtained by moving through a wedge-shaped part of the foil.

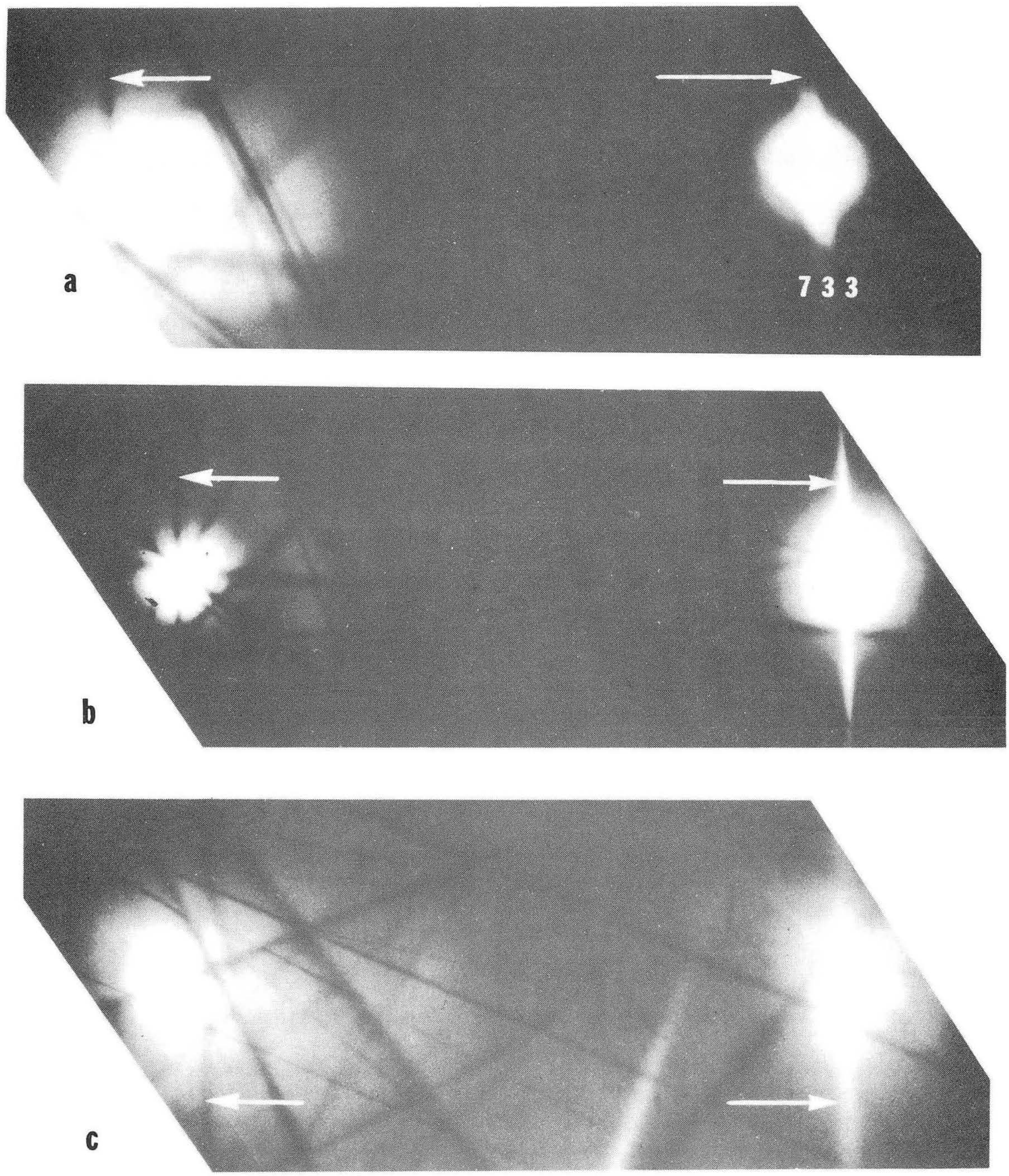
Fig. 3. In this observation, the (440) pair and the $(1\bar{1}5)$ pair of Kikuchi lines occurred simultaneously. It can be seen that the (440) line spacing is barely smaller than that of the (440) Bragg spot pattern while the $(1\bar{1}5)$ line spacing is considerably larger than that of the $(1\bar{1}5)$ Bragg spot pattern.

Fig. 4. A demonstration (with silicon (822)) to show that the Kikuchi line spacing variations with specimen thickness, a-c, occurs even when a conventional parallel incident beam is used.



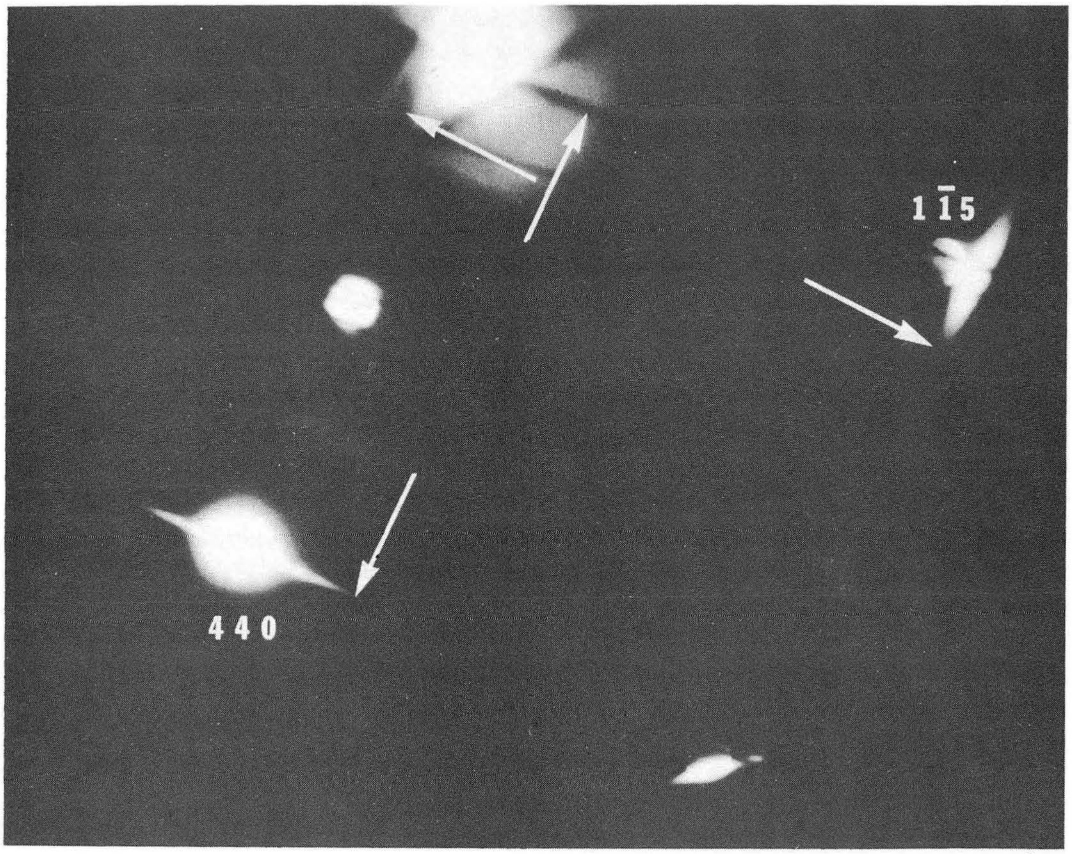
XBB 713-983

Fig. 1



XBB 701-57b

Fig. 2



XBB 713-985

Fig. 3

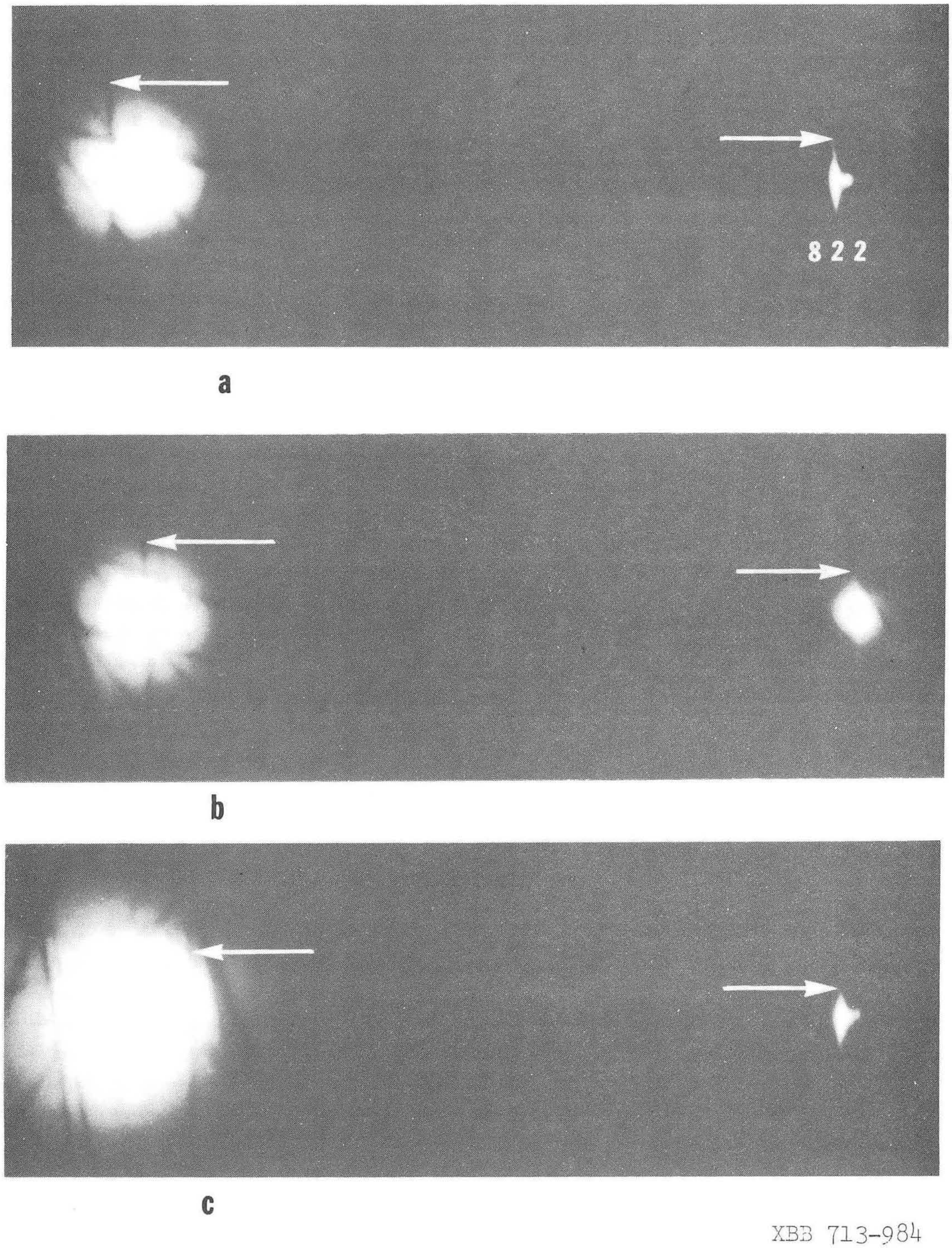


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Fig. 4

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