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November 1987

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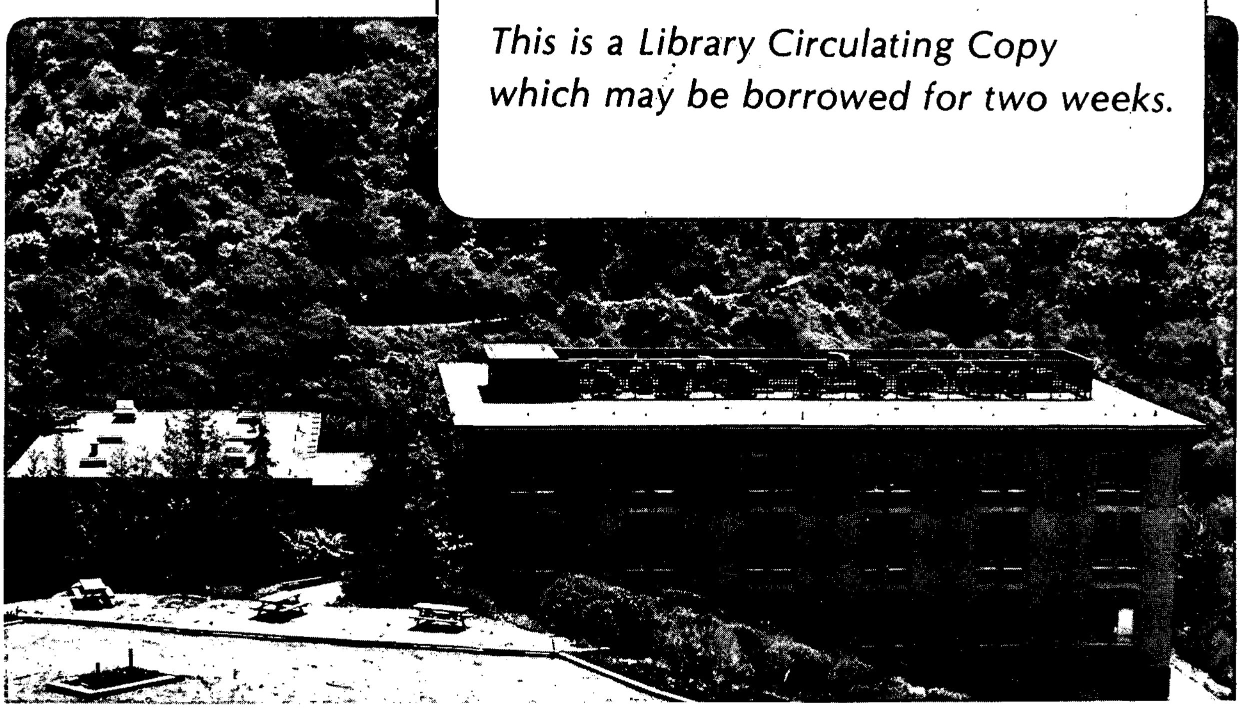

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SIMULATION AND PROCESSING OF HIGH RESOLUTION IMAGES OF DEFECTS AND INTERFACES IN SUPERCONDUCTING $YBa_2Cu_3O_7$

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INTRODUCTION

The superconducting performance, in particular the critical current, of polycrystalline $YBa_2Cu_3O_7$ will depend strongly on the structure of grain boundaries and the nature and number of defects in the microstructure. To characterize the atomic structure of defects and grain boundaries in these materials, a high resolution electron microscopy study has been initiated. Emphasis has been placed on the technique of phase contrast imaging with corresponding computer simulation and processing for optimum interpretation of results.

EXPERIMENTAL

High resolution electron microscopy was carried out in the Berkeley Atomic Resolution Microscope [1] equipped with a ±40° biaxial, double-tilt-lift goniometer operating at 1000 kV.

For determination of the structural changes due to reaction with air, $YBa_2Cu_3O_7$ was finely powdered and suspended in pure ethanol. A few droplets of the suspension were put on a carbon-coated holey films over Cu grids and left in air for one hour.

For analysis of the structural details of the grain boundaries, 92% dense material was thinned to 10μm by dry grinding with 320 mesh paper and subsequent ion beam thinning using an Ar beam at 4 kV and an incidence angle of 15° while the sample was liquid-nitrogen cooled. The thinned samples were left in the vacuum of the ion beam thinning equipment until they could be transferred into the electron microscope.

Image processing was carried out using SEMPER software [2] on images digitized via an Eikonix™ camera. The simulation of images was performed using the CEMPAS simulation software developed at the National Center for Electron Microscopy [3].

EXPERIMENTAL RESULTS

Figure 1 shows the result of different types of image processing by spatial averaging. The planar defects running horizontally across the experimental image of Fig 1a are those found in $YBa_2Cu_3O_7$ when it is exposed to air. In Fig. 1b, the image was translationally averaged over 10 unit cells along the defect planes. In Fig. 1c, additional averaging was performed by using operations of rotational and mirror symmetry. Rotation averaging was done by adding the image of figure 1b with this same image after rotating it over 180° about the two-fold axis located between each two neighboring Cu atoms of the $(CuO)_2$ double layer. In a similar way, the rotation-averaged image was further averaged with another formed by reflection in a mirror perpendicular to the defect planes.

It is noted that the processed images show the symmetries expected of the normal 123 structure (see Fig. 1f), but the experimental image lacks both 2-fold and mirror symmetry, even in the perfect matrix structure away from the defects. This suggests that the specimen was either misoriented or mechanically unstable during imaging. Since no additional deviations from either two-fold or mirror symmetries are observed at the defects, it follows that spatial averaging should improve the information retrieval from the defect images as well. Finally, a calculated image and the model used for the calculation are given in Figs 1e and 1f. Details of the calculation are given in ref [4].

Figure 2a shows a high resolution image of a grain boundary. The upper grain is in near-perfect [100] orientation and has a very sharp (001) interface plane whereas the lower grain is of high-index orientation, close to [441], and has a high-index interface plane. Because the lattice periodicities of both grains are incommensurate along the grain boundary, spatial averaging cannot be carried out across the entire image using a single operation of translational symmetry. The compromise is to process each grain individually. Figures 2b and 2c result from averaging over 21 units using the translation vector of the [100]-oriented grain and 12 units of the [113]-oriented grain, respectively.

Figure 3 shows a high resolution image of a relatively rare grain boundary in which the interface plane is not an (001) plane of either bordering grain. Two interesting features are observed. First, the upper grain in [110] orientation shows strong changes in image contrast due to deformation of the lattice caused by decomposition [5]. Because of the corresponding loss of translational symmetry, image enhancement by lattice averaging is not possible. Second, the stepped interface of the [110] oriented grain is closely followed by the lower grain, with no apparent amorphous material at the grain boundary.

DISCUSSION

The study of the atomic structures of planar defects and grain boundaries is complicated by the fact that they must remain in an edge-on orientation relative to the direction of the electron beam and in addition at least one of the grains must be in an exact zone-axis orientation to provide structural information. Furthermore the specimen must be thin enough in the region of interest to allow direct interpretation by application of such models as the weak phase object or linear image [3]. Together these restrictions make both a large-angle goniometer stage and meticulous specimen preparation procedures mandatory.

The electron microscope study of defects and grain boundaries in $YBa_2Cu_3O_7$ is further complicated by the continual formation of new defects due a surfacenucleated decomposition reaction [5]. The formation of such defects is aggravated by ion-beam thinning, even when the specimen is cooled to liquid nitrogen temperature. In order to avoid such decomposition artefacts, it is necessary to study $YBa_2Cu_3O_7$ using freshly-cleaved wedge-shaped crystals, mounted between folding grids (preferably made of a refractory metal) and inserted into the microscope as soon as possible.

In addition to specimen-induced difficulties, there are other problems associated with image formation because the region of an internal interface is susceptible to defocus-induced effects [6]. In particular, the proximity of signals from contiguous grains may result in image periodicities that come from the interference of both grains. This problem is most serious when there is grain overlap of course, but even for properly-oriented interfaces, such effects can occur at large defocus values. The most useful image for the majority of studies is recorded at Scherzer defocus [6], because it provides the largest passband of spatial frequencies within the resolution limit of the microscope. Images recorded at different defocus values can still be interpreted, but only by matching to simulated images that account for all specimen and microscope parameters.

The above results show that image processing can be carried out in several ways simply by exploiting the symmetry of the specimen. Both macroscopic (rotation and reflection) and microscopic (translation) elements of symmetry can be used, and the relationship between actual and processed images is discretely preserved. However, another method involves averaging in Fourier space. A digitized image can be Fourier transformed and the spectrum examined for its periodic (crystalline) and non-periodic (damaged, noise) components. The crystalline information can then be separated by multiplying the Fourier transform by a filter function, such that all information contained within the periodic spectral points is either maintained (to image the crystal) or deleted (to identify the noise, or amorphous regions of the specimen, etc.). The final image is obtained by performing an inverse Fourier transform.

The main advantage of the first method, averaging in real space, is that it is exactly clear what has been averaged, whereas for filtering in Fourier space a translation of the filtering operation to the real image has to be made. For instance, when reciprocal lattice points are masked to very sharp delta functions, the processed image looks perfect even if it does contain defects! Non-periodic information about local changes in structure can only be retained in the Fourier-processed image by including some of the non-"Bragg" scattering in the inverse transformation process.

ACKNOWLEDGEMENTS

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Figure captions

Fig. 1. (a) Digitized high resolution image of $YBa_2Cu_3O_7$ in [010] orientation showing two planar defect layers; (b) translationally-averaged; (c) rotationally and reflection-averaged; (d) simulated image; (e) projected structure with Cu shown as filled circles, Ba large open and Y large filled circles, anions are omitted. (f) shows the structure with the aions included.

Fig. 2. (a) Digitized high resolution image of grain boundary with upper grain in [100] orientation, lower grain in [441]; (b) and (c) are processed images. Note the bending of (104) lattice planes at the interface region.

Fig. 3. High resolution image of a sharply-faceted grain boundary. The upper grain shows large local structural changes due to insertion of extra CuO layers.

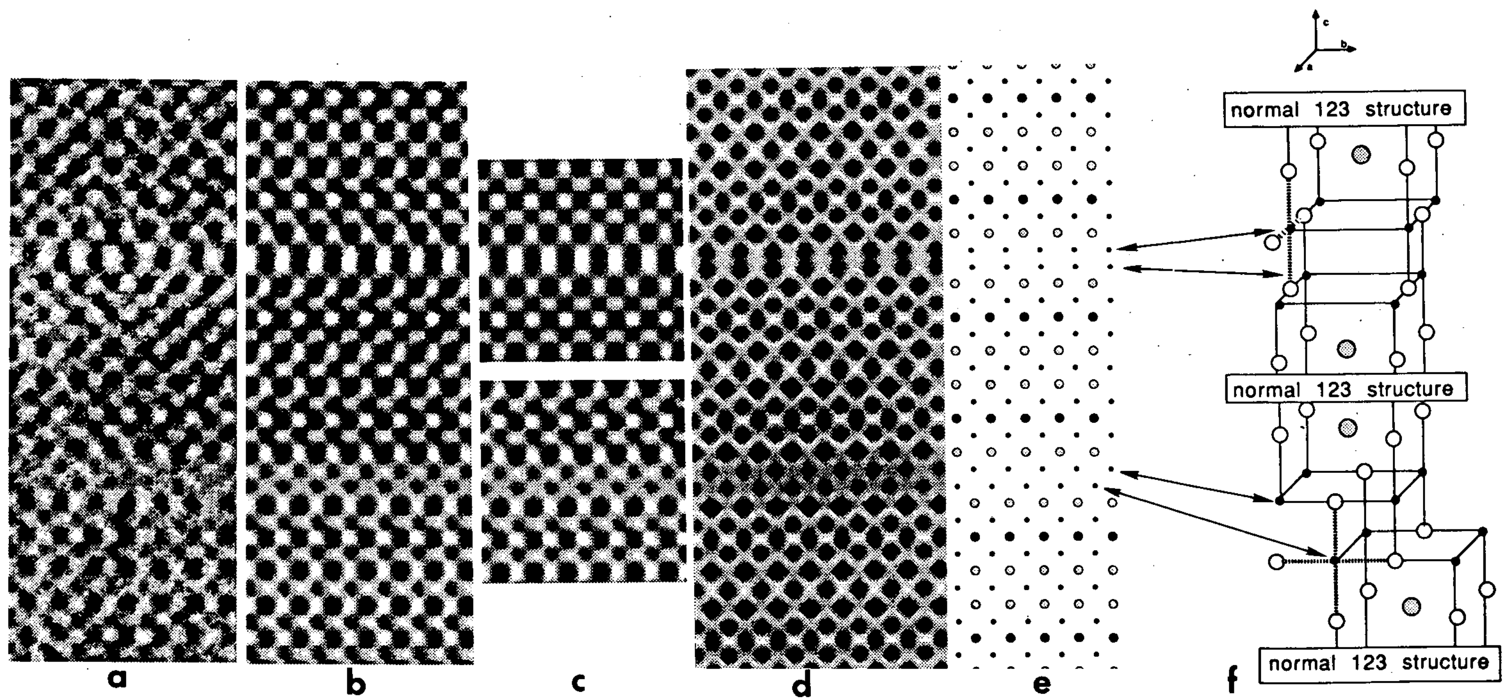

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Fig. 1

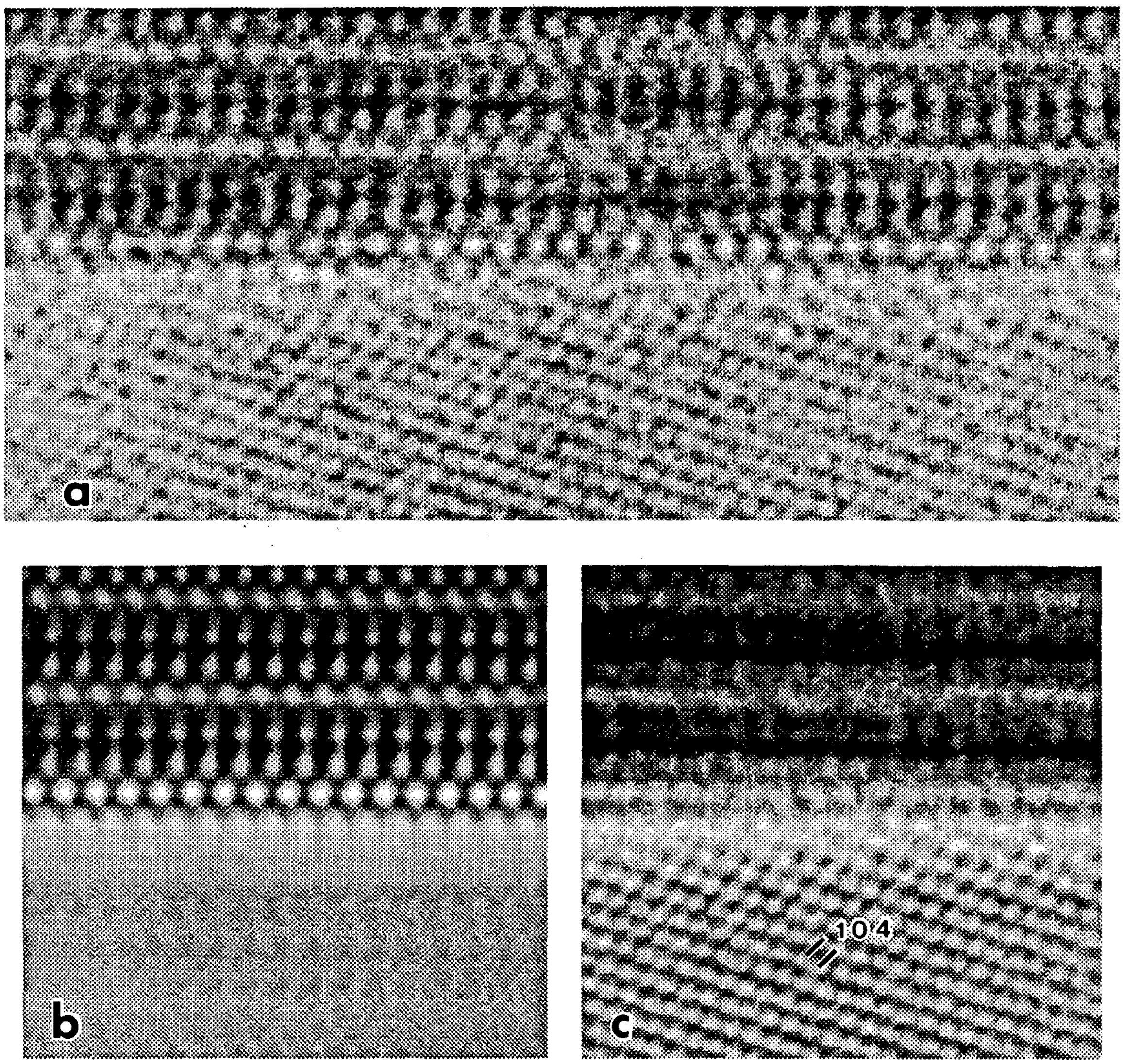

Fig. 2

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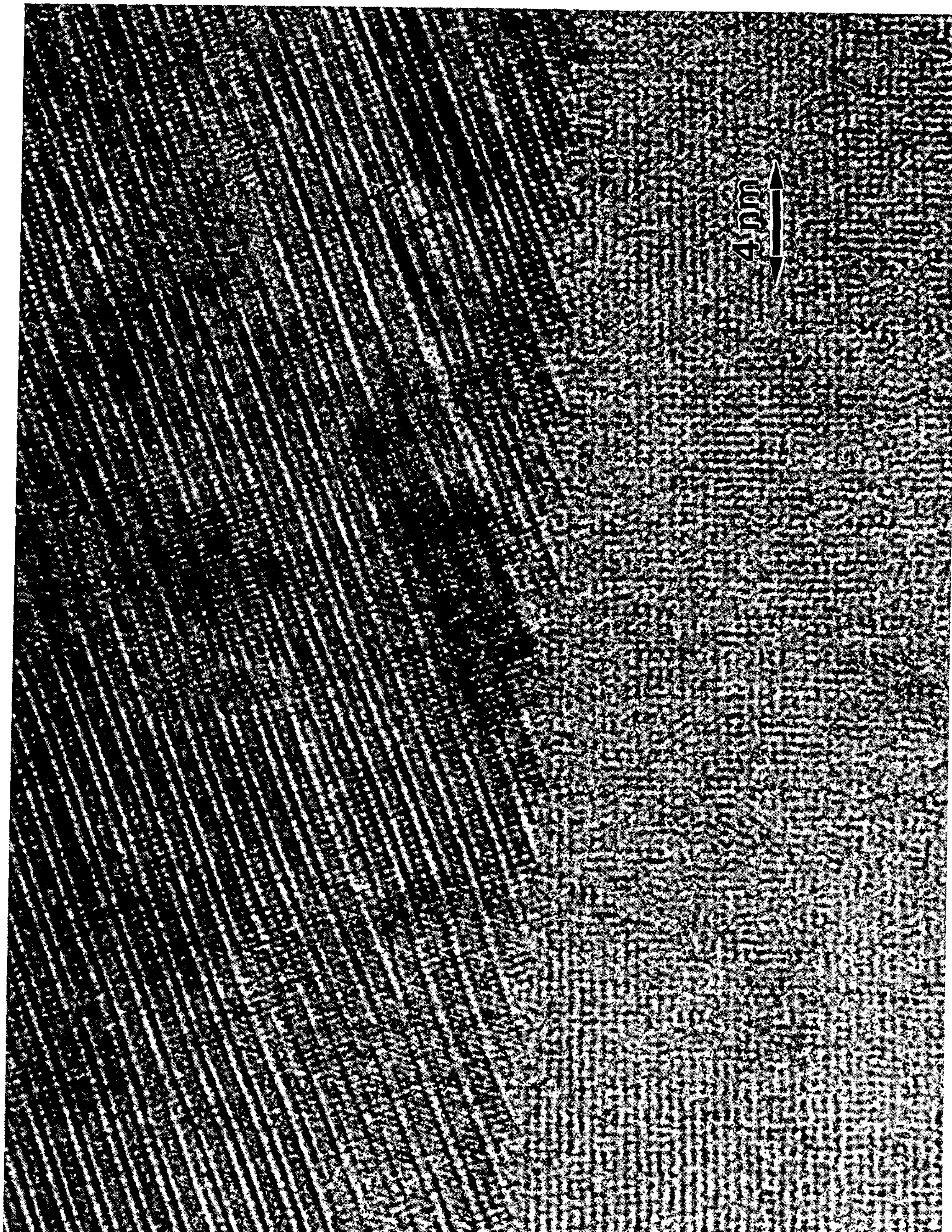

Fig. 3

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