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March 1984

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RESOLUTION AT ITS CURRENT LIMIT: APPLICATIONS OF THE BERKELEY ARM

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INTRODUCTION

The Atomic Resolution Microscope (ARM) is a high voltage electron microscope manufactured by JEOL, Ltd. to achieve a point-to-point resolution consistently at or better than 0.18 nm over its entire 400 kV to 1000 kV accelerating potential range. Both the history of the project [1] and details of the instrument [2] have been previously reviewed. Now located within the National Center for Electron Microscopy, the ARM is made available by the Department of Energy for use by all microscopists submitting an approved proposal [3]. The purpose of this paper is to highlight some of its recent and planned applications which exploit its superior resolution.

OPERATION

The ARM accepts common 3mm diameter specimens which have been thinned to electron transparency by standard techniques and screened for suitability in another TEM. Three cartridges can be simultaneously loaded into the specimen airlock and individually withdrawn by a turret mechanism for rapid insertion into the top-entry goniometer stage. In addition to $\pm$ 40° biaxial tilting, the stage also has a height (z) control to alter specimen position along the optic axis over a 2mm range within the objective lens. Once in position, focusing and astigmatism correction are carried out while viewing the image directly on the screen phosphor. Through-focus series are facilitated by infinite-turn, continuously variable potentiometer control of the objective lens current, with a minimum focus increment of 3 nm. In addition, the stigmator coils are fed by six independent CPU channels, permitting different reference settings to be stored in memory for rapid recall. Operation is further simplified by digital readout of all relevant lens and deflector coil excitation values at the console. Specific data concerning film number, operator code, magnification or camera length, and a 14 character text assigned by a user terminal are also recorded in the margin of each negative, of which there are 50 per magazine.

APPLICATIONS

<u>METALS AND ALLOYS</u> The most obvious candidate specimens for the ARM are the metallic elements and alloys which prefer close-packed arrangements of atoms. Included in the first group of ARM proposals are a number of investigations of the defect structures, particularly at interfaces, in these materials. One completed study [4] of precipitation in the Mo-Hf-N system is represented in Figure 1. The microstructure is a dispersion of HfN plates having the NaCl structure in a matrix of bcc Mo with a (001) habit. The precipitate plates are related to the matrix by the Bain correspondence which for this combination of lattice parameters yields an invariant plane strain manifested as a simple extension normal to the precipitate habit plane. The large volume change of about 40% between parent and product phases must be accommodated by lattice vacancies. Therefore the results shown in Figure 1 are highly significant. The image reveals that

(1) the precipitate-matrix interface is atomically smooth and (2) the precipitate contains no internal faulting. Consequently it can be concluded that the vacancies participating in the reaction condensed in double, not single, layers. Single layers would have resulted in internal faulting. Here the structural evidence is clearly at the atomic level.

IONIC SOLIDS The results in Figure 2 show another outstanding advantage of the ARM for probing larger unit cell, ionically-bonded materials. By image contrast variations alone, the distinctions between anion and cation sites are clearly revealed. In this figure the $BaTiO_3$ crystal is viewed along a cube axis and compared with a computer-simulated image for the same experimental conditions. Future investigations will employ the ARM to investigate local variations in stoichiometry (chemical disorder) as well as structural disorder of similar ionic solids, imaging interstitial as well as lattice occupancy sites.

SEMICONDUCTORS The largest single class of materials to be explored in the ARM fall into the electronic materials category, where again structural and compositional variations will be monitored at the atomic scale. A particularly enticing possibility is suggested by the computer simulated images of Figure 3 which show that the ARM is capable of distinguishing atomic species in GaAs. Experiments exploring the nature of antisite defects have already been proposed.

CATALYSTS The higher voltage range of the ARM is expected to be an advantage in the direct imaging of atomic structure of Zeolites. Proposals here again range from crystal structure determination to the types of structural disorder (e.g. intergrowths) which accompany Zeolite synthesis methods, where the higher voltage of the ARM would induce less ionization damage.

SUMMARY

With a demonstrated point-to-point resolution of 0.16nm (Figure 4) and a theoretical point-to-point resolution of 0.13 nm, the ARM currently establishes the operational limit of existing microscopes. The sample of applications presented here shows the manner in which this instrument will be applied to the characterization of both structural and compositional discontinuities in solids at atomic dimensions. These remain areas of critical need and provide ample stimulus for further improvement of instrumental resolution.

ACKNOWLEDGEMENTS

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2. R. Gronsky and G. Thomas in 41st Annual Proc. Electron Microscopy Soc. Amer., Phoenix, G.W. Bailey, (ed.) p. 310 (1983).
3. A User's Guide is available upon request from M. Moore, National Center for Electron Microscopy, Lawrence Berkeley Laboratory, Berkeley, CA 94720.
4. K.H. Westmacott and U. Dahmen, Precipitate Plate Growth Mechanisms, Scripta Met. (submitted).

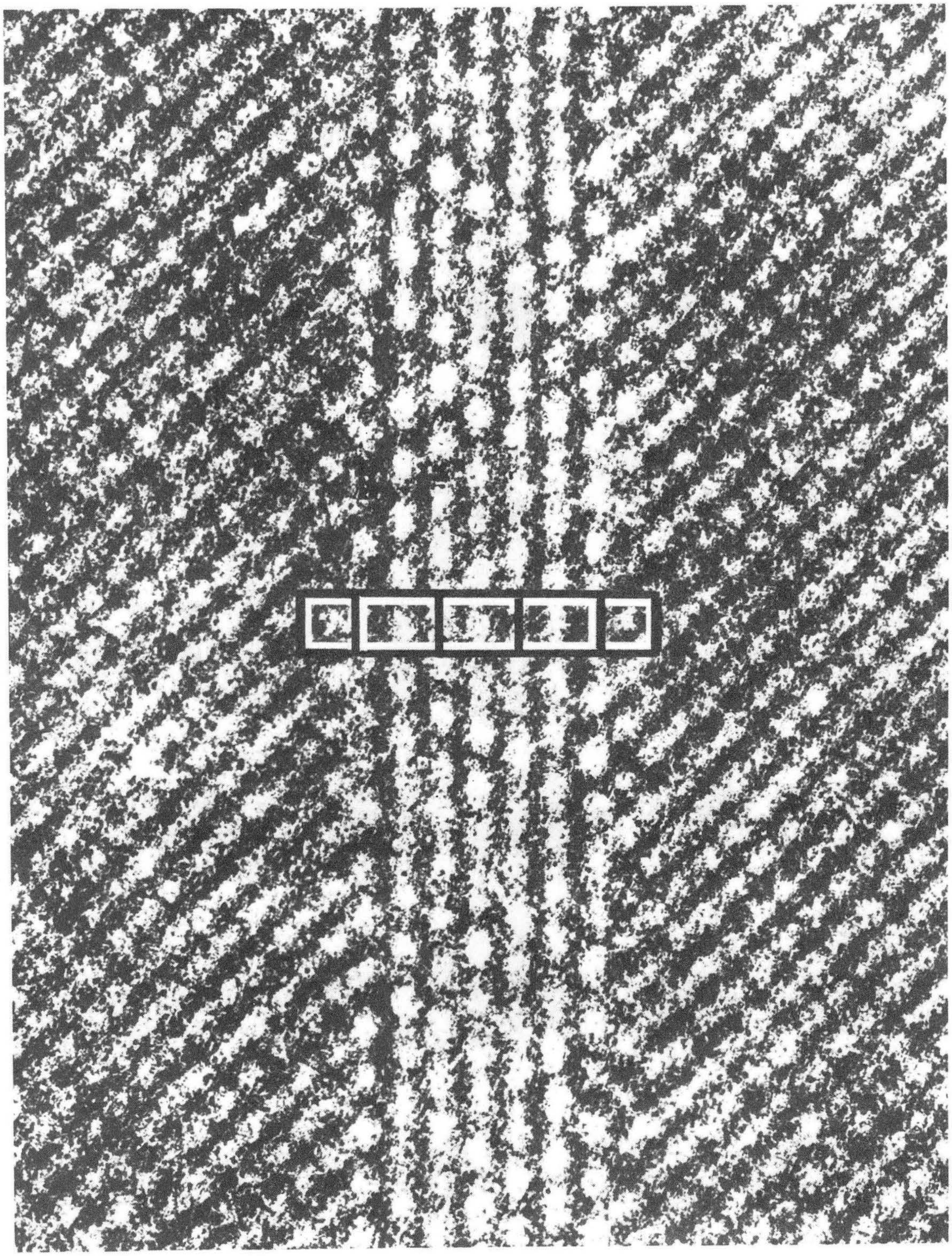
FIGURE CAPTIONS

Fig. 1 Atomic resolution image of HfN precipitate plate in matrix of Mo, [010] orientation, Scherzer defocus (ARM). The Mo unit cell is 0.31 nm on edge.

Fig. 2 (a) Model of ionic positions in [001] projection of $BaTiO_3$ (b) experimental image taken on the ARM at 1MeV and (c) computed simulation showing perfect image match at a defocus of 9nm and thickness of 16nm.

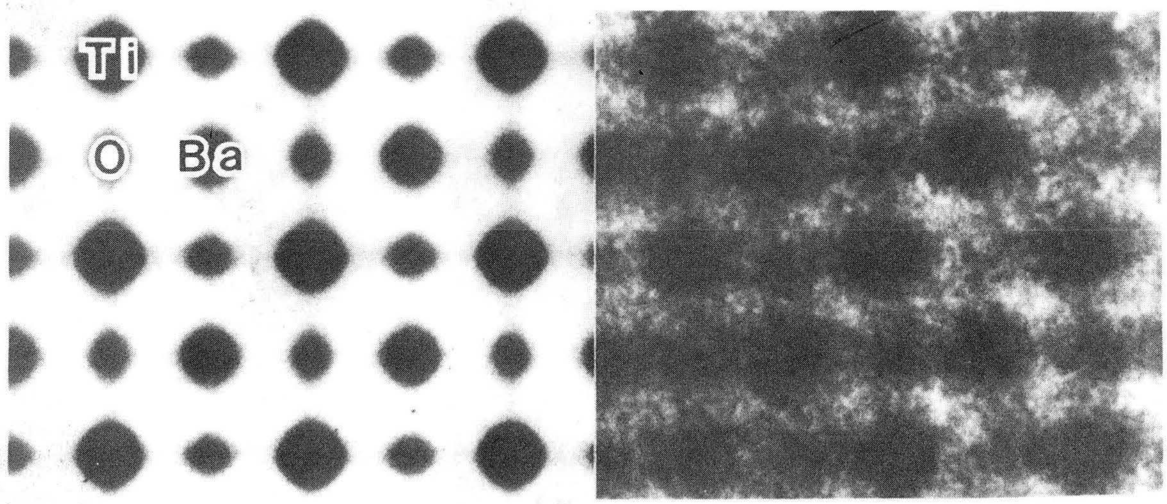
Fig. 3 Projected atomic potential and simulated image of GaAs in [011] orientation, defocus 87 nm, thickness 12 nm, 1 MeV (ARM).

Fig. 4 Optical diffractogram resolution test of the ARM at 1 MeV, Scherzer defocus. Calibration spots are from crystalline Au, diffuse scattering from amorphous Si.



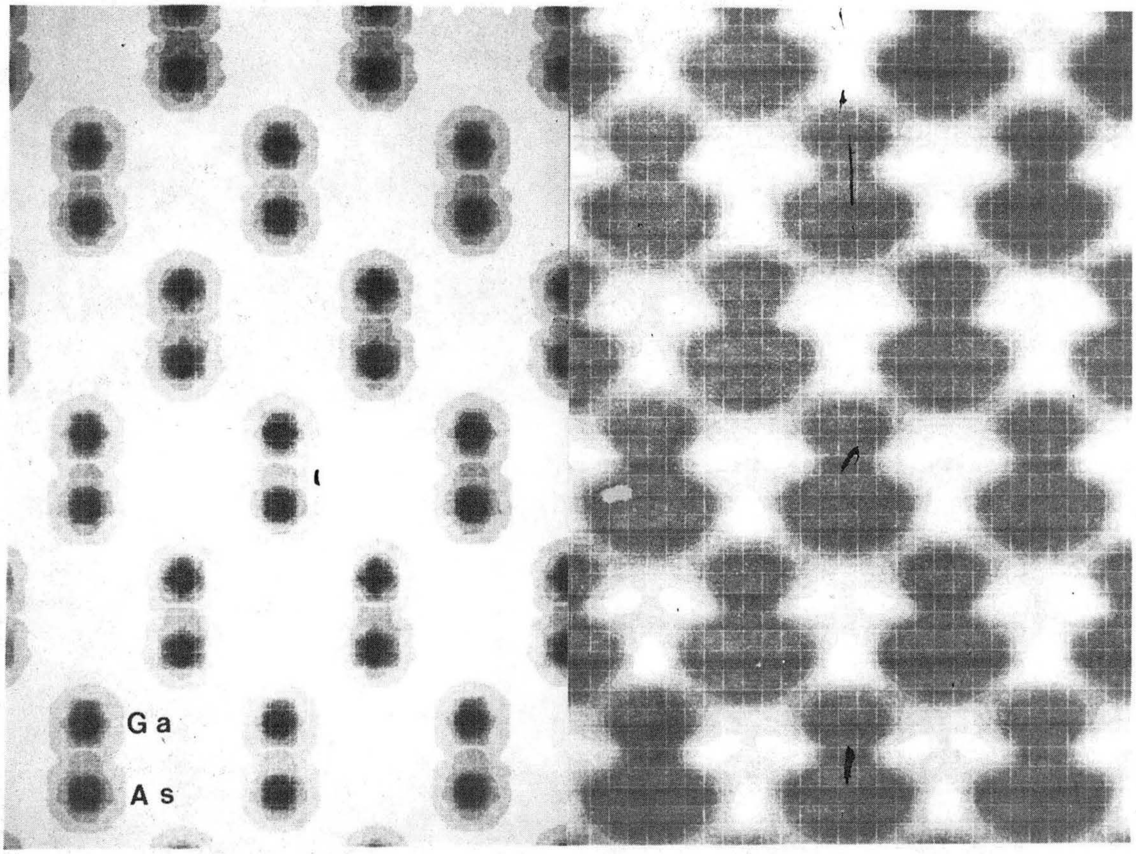
XBB 839-7867

Fig. 1



XBB 844-2691

Fig. 2(c)



XBB 844-2693

Fig. 3

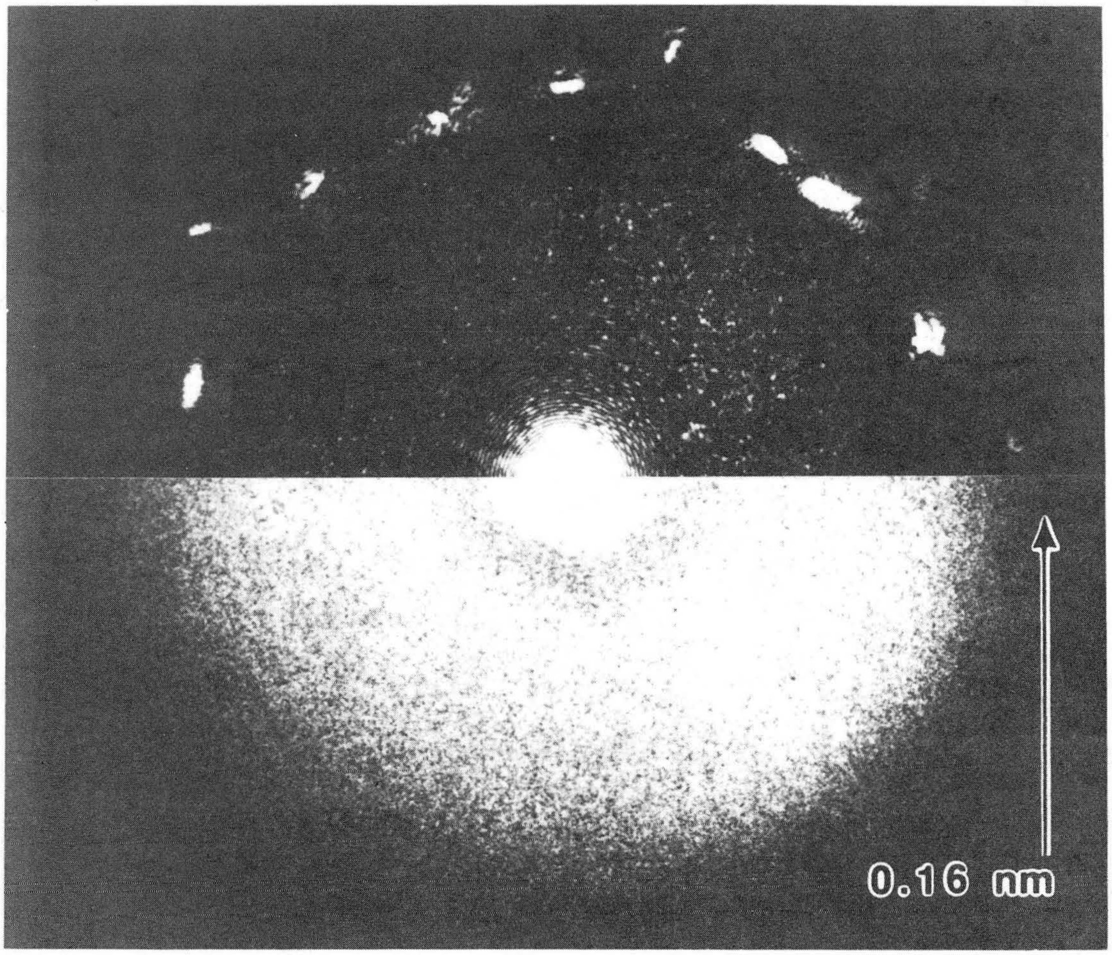


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Fig. 4

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