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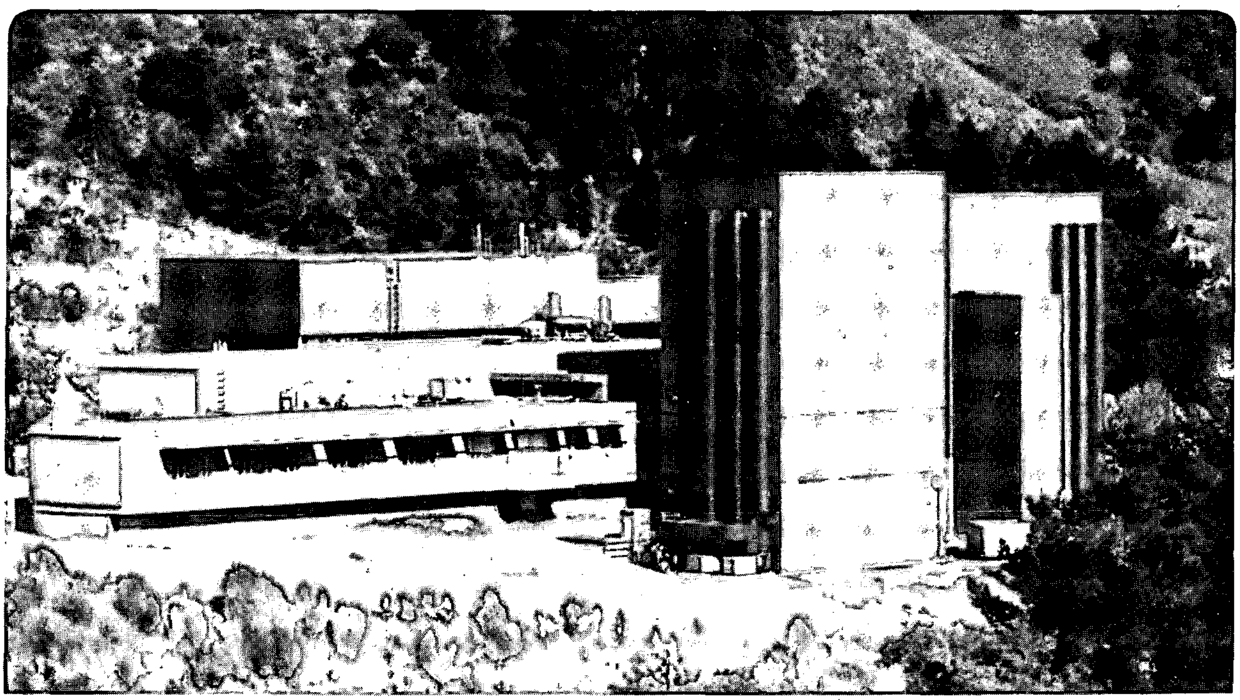

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HIGH VOLTAGE / HIGH RESOLUTION STUDIES OF METAL AND SEMICONDUCTOR INTERFACES

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ABSTRACT

The application of high resolution transmission electron microscopy to the study of homo- or hetero-phase interface structures requires specimens that meet stringent criteria. In some systems the necessary geometric imaging conditions are established naturally, thus greatly simplifying the analysis. This is illustrated for a diamond-hexagonal/diamond-cubic interface in deformed silicon, a Σ99 tilt boundary in a pure aluminum bicrystal, and a germanium precipitate in an aluminum matrix.

INTRODUCTION

The strength of materials is strongly influenced by the presence of interfaces such as grain or interphase boundaries. Consequently, fundamental studies of the structure of interfaces will ultimately lead to a better understanding of the mechanical behavior of materials.

In recent years the availability of transmission electron microscopes with sub-2Å point resolution has allowed atomic resolution imaging of interfaces to be extended to the close-packed metals. The purpose of this short contribution is 1) to emphasize the importance of optimizing the specimen geometry and quality to ensure attainment of the necessary contrast and resolution of interfacial structure and 2) to present brief summaries of three recent high resolution studies of interfaces in metals and semiconductors. Although not all the examples were specifically designed as an interface study, the work provides a clear illustration of three different types of interface, a heterophase interface in a semiconductor (Si), a homophase interface (grain boundary) in a metal (Al), and a heterophase metal/semiconductor interface (Al-Ge).

EXPERIMENTAL DETAILS

An ideal specimen for high resolution imaging is one that is thin (<100Å) flat (not bent) and clean (with a minimum covering of contamination). Most unsuccessful attempts to obtain an interpretable image are attributable to failure to fulfill one or more of these conditions. For the successful imaging of an interface at atomic resolution further stringent geometrical conditions must also be satisfied [1]. Each crystal forming the interface must have a low index zone axis (but not necessarily the same zone in both crystals) parallel to the beam and parallel to a vector in the interface plane. These conditions are illustrated in Figure 1. To achieve atomic resolution throughout the specimen and at the interface the sample is oriented using the biaxial tilt-stage so that these directions are accurately aligned with both the optic axis of the microscope and the electron beam.

Suitable specimens were prepared by chemical or electropolishing techniques or by dimpling and ion beam thinning and examined in the NCEM JEOL-1000 Atomic Resolution Microscope at 800 kV and other high resolution microscopes.

RESULTS

Hexagonal Silicon (Heterophase Interfaces in a Semiconductor)

The observation of thin ribbons of a diamond-hexagonal (dh) form of silicon in hot-indented specimens was first reported by Eremenko and Nikitenko [2,3]. The ribbons were observed to emanate from the highly deformed regions near the indent and propagate on {511} planes of the matrix. This interesting phenomenon was subsequently studied in detail using a variety of techniques, including high resolution imaging, by Pirouz et al. [4,5] and a comprehensive analysis will shortly appear in print [6]. It is shown that formation of the dh metastable phase can be treated as a martensitic transformation having its origin at the interfaces of deformation twin interactions. A schematic illustration of the formation of hexagonal phase in an fcc lattice in <110> projection is shown in Figure 2. Figure 2 illustrates the condition when a secondary twin T_2 nucleates in a primary twin band T_1 and subsequently propagates into the matrix M. This is the mechanism that gives rise to the long ribbons on {511} habit planes. An example of an hexagonal ribbon imaged at high resolution is given in Figure 3.

Grain Boundaries in ICB Aluminum Bicrystals (Homophase Interfaces in a Metal)

Unique continuous bicrystal films are formed when aluminum is deposited on a {100} silicon substrate by the Ionized Cluster Beam technique (see Ref. 7 for details). Two different {110} orientation variants of the aluminum form which are rotated relative to each other by 90^{o}. The resulting microstructure consists of islands of one variant in a sea of the other bounded by 90^{o} <110> tilt boundaries all of which close on themselves (i.e. the film contains no grain boundary triple points) [8,9]. If an annealing treatment at 400^{o} C is followed by removal of the silicon substrate a specimen ideal for high resolution TEM observation of the boundaries is obtained. This is because during the annealing the grain boundaries in the aluminum minimize their area by rotating normal to the foil surface. The result is a foil containing the ideal structure depicted in Figure 1. An example of an image of a $\Sigma 99$ symmetrical boundary.obtained under these conditions is given in Fig. 4. In the center of the image an asymmetrical section is seen to connect two symmetrical {557} boundary segments which exhibit clear atomic relaxations into structural units [10].

Needle Precipitates in Al-Ge (Heterophase Metal/Semiconductor Interfaces.

When a dilute Al-Ge alloy is given a suitable quench/age treatment, long needle-shaped Ge precipitates form lying parallel to <100> directions in the aluminum [11]. Consequently, if thin foils of this alloy are prepared and viewed along a <100> zone axis, observation of the end-on Ge needles allows a detailed examination to be made of the interface between the germanium and aluminum. It is found [12,13] that a <110> direction in the Ge is accurately parallel to <100> Al, thus again satisfying the conditions in Figure 1. The precise alignment is attributable to the $\sqrt{2}$ relationship between the lattice parameters of Al and Ge whereby [100] Al almost exactly matches [110] Ge. An example illustrating the image detail of the interface structure as well as the internal precipitate structure is given in Figure 5.

CONCLUSIONS

High resolution imaging is a powerful technique for observing the atomic structure of interfaces. The specimen geometry must be optimized to obtain maximum information on the interface structure.

ACKNOWLEDGEMENTS

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FIGURE CAPTIONS

Fig. 1 Schematic illustration of the geometric conditions that must be satisfied to image interface structures at high resolution.

Fig. 2 Schematic diagram showing, in a <110> projection, the formation of a hexagonal ribbon H when a secondary twin T_2 nucleates inside a primary twin T_1 and propagates into the matrix M.

Fig. 3 Ribbon of diamond-hexagonal material with {511} habit plane in hot-indented silicon (micrograph courtesy P. Pirouz)

Fig. 4 Faceted segments of a Σ99 89.4° <110> tilt boundary in aluminum

Fig. 5 Interface structure between aluminum matrix and a germanium needle formed by a precipitation reaction. Note the alignment of <100> Al with <110> Ge. (Micrograph by J. Douin)

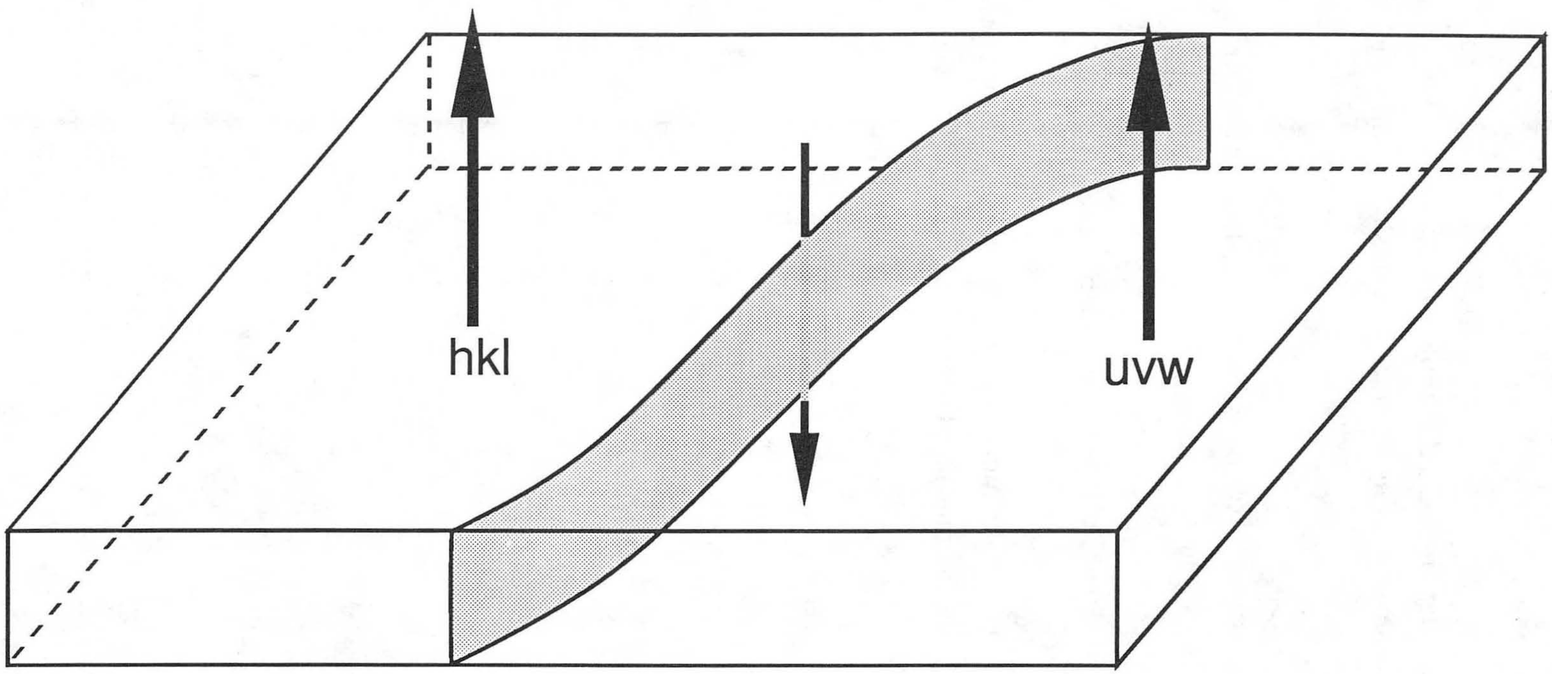

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FIGURE 1

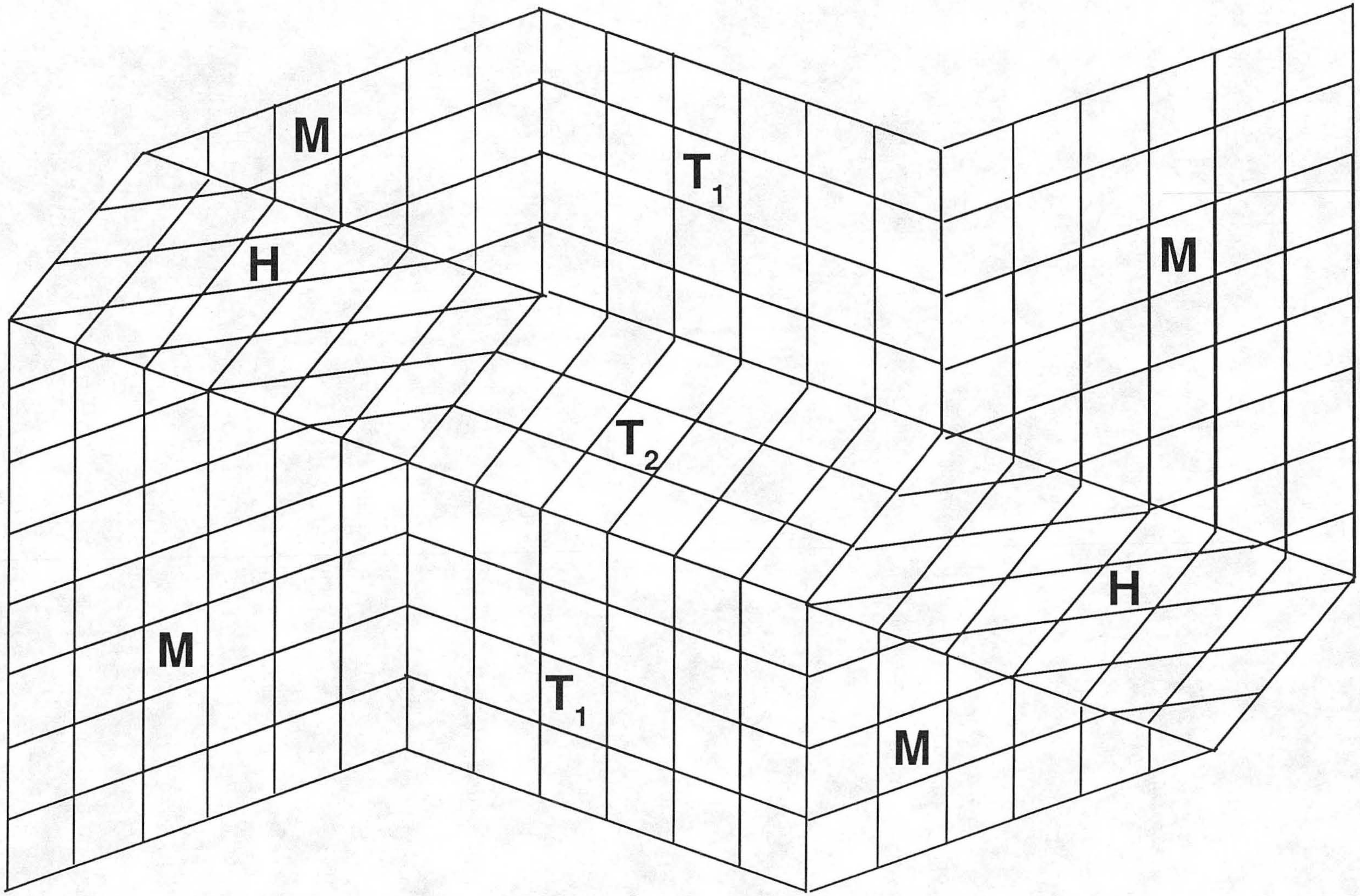

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FIGURE 2

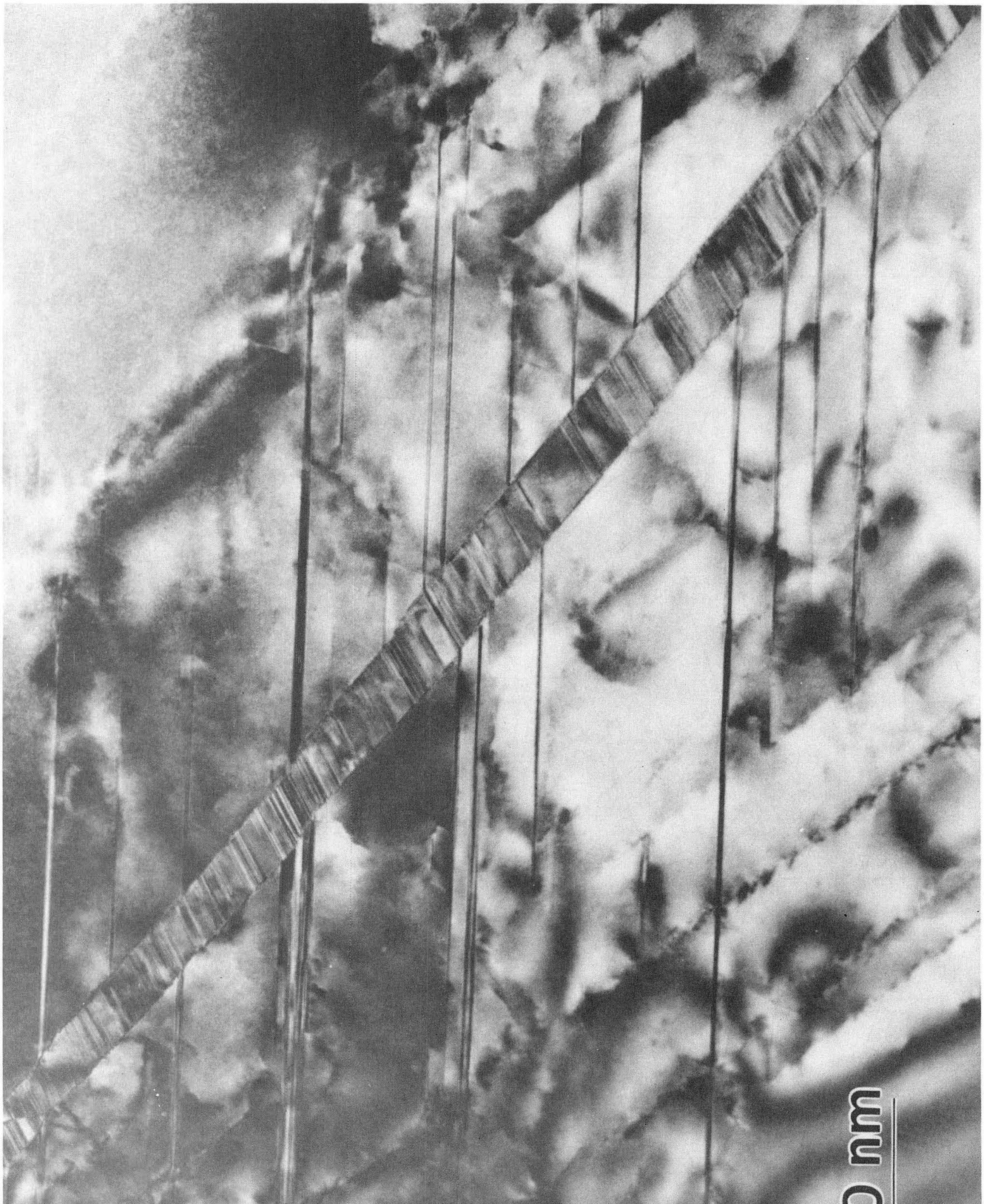

FIGURE 3

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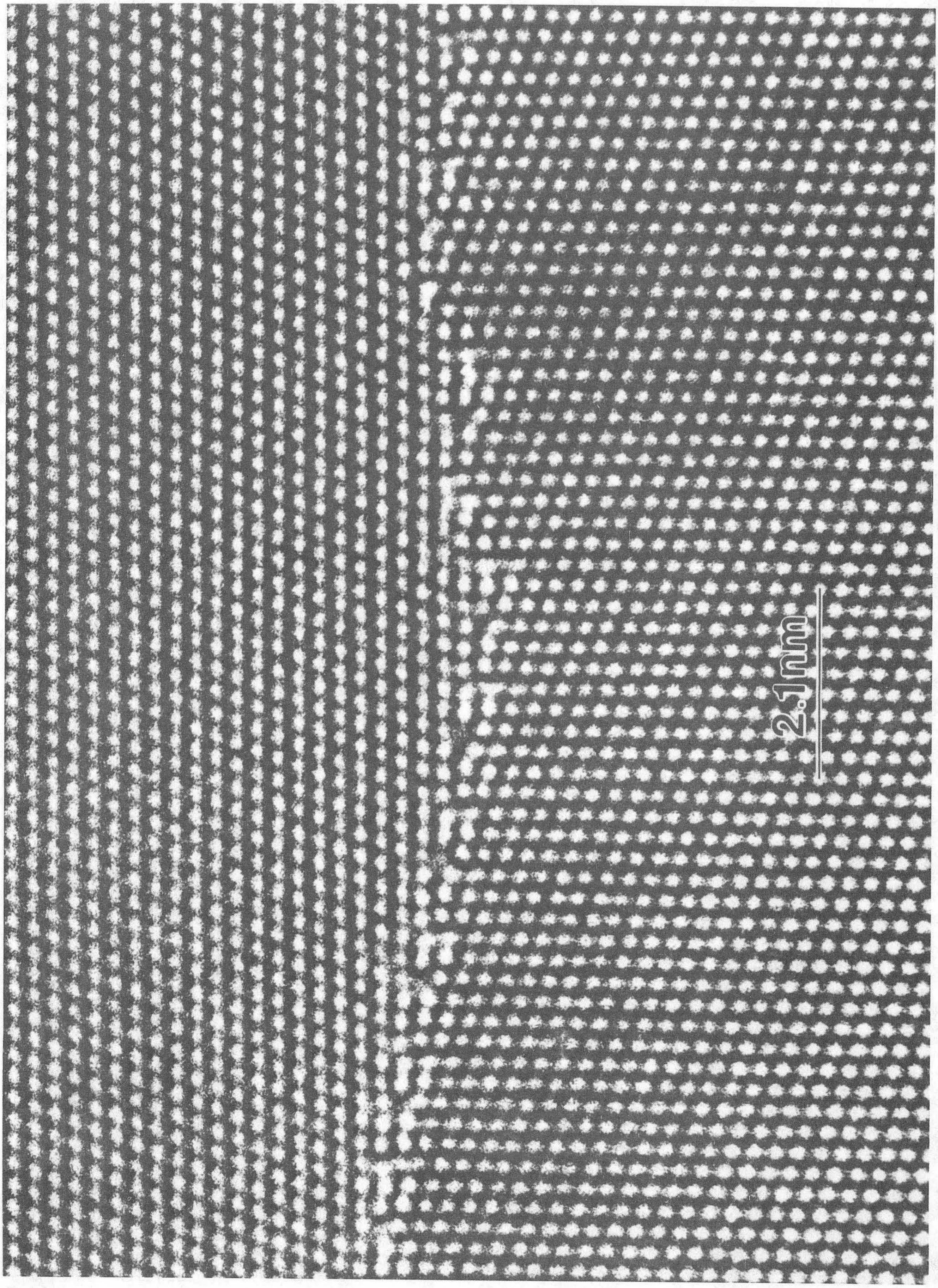

FIGURE 4

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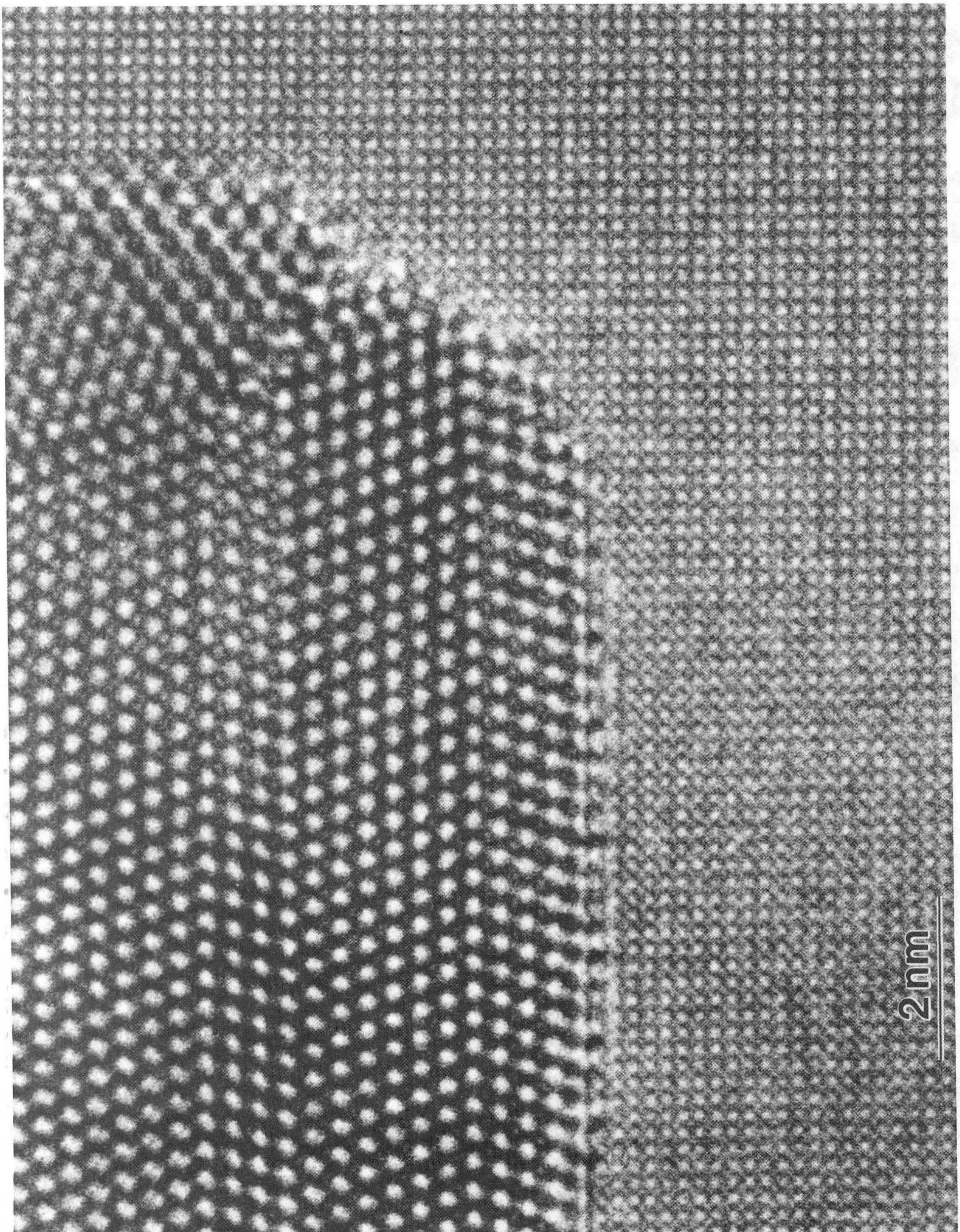

FIGURE 5

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