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Notes presented at the Workshop on Characterization of Crystalline Interfaces by Advanced Electron Microscopy, Berkeley, CA, August 20–24, 1990

Characterization of Crystalline Interfaces by Advanced Electron Microscopy

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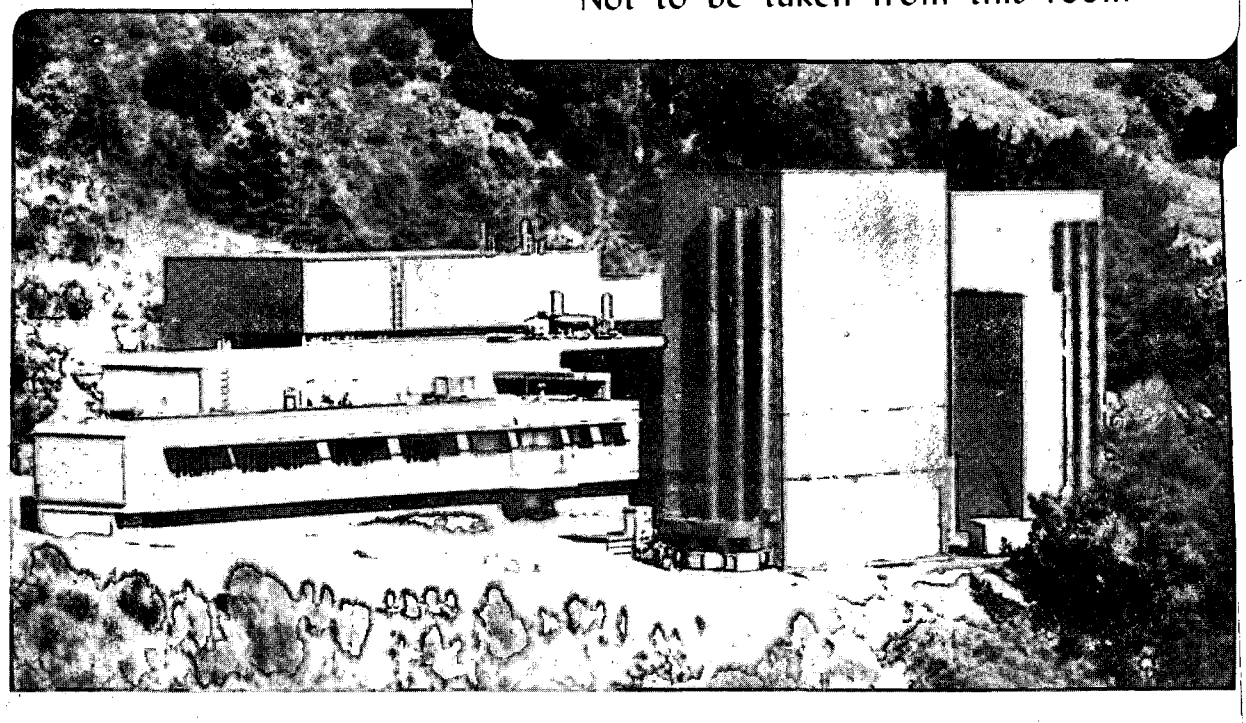

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Characterization of Crystalline Interfaces by Advanced Electron Microscopy

Notes from an international workshop held at the National Center for Electron Microscopy, Lawrence Berkeley Laboratory, University of California, Berkeley, August 20 - 24 1990

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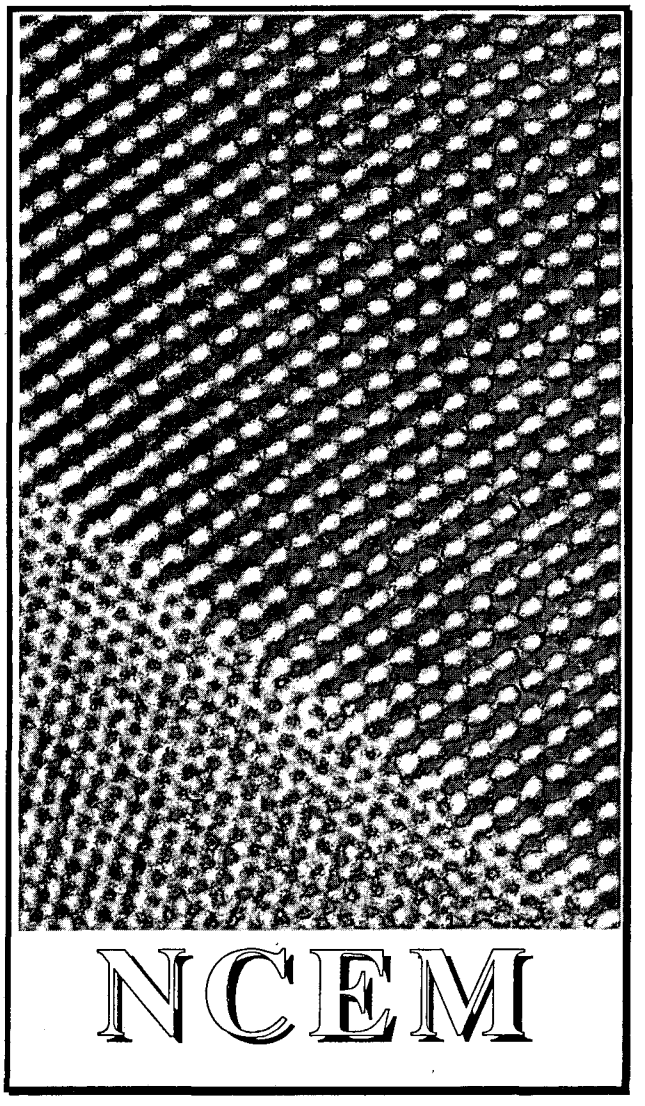

ARM image of interface between Ge and Al in cube-twin orientation

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PREFACE

This report has been collated from the notes of the session reporters. After some discussion about the format, it was decided to leave the individual reports basically unchanged and do only a minimum of editing. This has resulted in a more colorful document that maintains some of the diversity and excitement, as well as some of the confusion and uncertainties, of the workshop.

The notes are necessarily an abbreviated and incomplete rendition of what actually occurred. Some of the reporters were very conscientious about the accuracy of their notes and consulted each of the panel members before sending me the final version. Others were more trusting of their memory. Regardless of the individual approach, all of them took their task seriously, and it is the sum of their efforts that is represented in this report.

As an exchange of ideas the workshop was very successful indeed. A remarkable feature was the even participation by the entire group throughout the week. Participants learned of new facts or viewpoints on interface characterization, as well as contributed to the information of others. As a result the field of interface structure was defined more broadly than any one subgroup would have done. Yet, from the entire range of diverse topics a set of key problems emerged. Some of these led to such spirited discussion that task forces had to be assembled from the most vocal advocates. Topics and membership of these task force groups are listed with the Program and their conclusions are included in the summary reports.

One of the aims in assembling the program was to include areas that have traditionally been the domain of separate groups and this has resulted in a new and lively exchange. If any one area was under-represented in this meeting it was the field of analytical electron microscopy. This was not by design but because several invited participants were unable to attend.

After four excellent overview lectures to set the scene, the entire week was spent in discussions. The program was divided into three parts: one dealing with generic interface phenomena, one with materials-related aspects of interfaces and one concentrating on TEM technique. Naturally, there was some overlap between the different parts, but this only served to illuminate some problems from different angles. Although a good part of the discussion focused on high resolution imaging, it became clear that this is by no means the only electron microscopy technique capable of significant contributions to the field. Diffraction contrast techniques become increasingly important, especially in the area of irrational interfaces where HREM is severely handicapped by lack of crystal alignment. For the quantitative characterization of some interface features such as roughness, displacement and strain, convergent beam techniques are very powerful and probably underutilized.

There were many conclusions from this workshop but none were as clearly apparent as the call for a closer interaction between theory and experiment and a more quantitative approach to the interpretation of electron microscopy data. If electron microscopy is to continue to make a significant contribution to interface science, it is not sufficient to obtain improved spatial resolution. The race to break the One-Ångstrom barrier is meaningful only if we simultaneously address the problems of image interpretation, specimen noise, chemical resolution, modeling of interfaces and model/experiment comparisons. Computers will play an increasingly important role in quantitative image analysis as well as in the operation of the instrument itself. It is also important to combine electron microscopy with the results from other techniques wherever possible since the ultimate goal of interface characterization by TEM is the correlation between materials properties and the atomic structure, chemistry and bonding at interfaces.

U. Dahmen

Berkeley, California
6 March 1991

INTERFACES IN MATERIALS I

(T. Sands)

Chair: D. Cherns
Panel: R. Hull, S.Iijima, Z. Liliental-Weber, D. Maher, P. Pirouz

Interfaces in Electronic Materials

The discussion in this session concentrated on semiconductor/semiconductor, metal/semiconductor, and oxide/semiconductor interfaces in semiconductor heterostructures and devices. Issues that can be addressed by electron microscopy include strain in epilayers, interface roughness/sharpness, interface atomic structure, compositional homogeneity in semiconductor alloys, electrically active interfacial defects (can they be studied by TEM, at least indirectly?), impurity segregation. All of these issues are of critical importance to existing and future information technologies. The following is a selection of discussion highlights.

The Industry Perspective: D. Maher initiated the discussion with the Si industry perspective. He pointed out that by the time a semiconductor sample reaches the microscopist in an industrial laboratory, it has probably been characterized by a wide range of techniques, including Rutherford backscattering (RBS), Auger electron spectroscopy (AES), secondary ion mass spectrometry (SIMS), photoluminescence (PL), ellipsometry, and Raman spectroscopy. Electron microscopy is therefore one technique in a battery of techniques that together can provide a very complete understanding of the structure and composition on a nm scale in a semiconductor device or heterostructure. Certainly, the ability of a microscopist in this setting to request blanket wafers or wafers patterned with TEM test structures is a considerable advantage. T. Sands emphasized this point further by showing that marker layer substrates combined with cross-sectional TEM and backside SIMS analysis can provide nm scale compositional and structural information essential to the design of shallow ohmic contacts to III-V semiconductors. Specially designed marker layer substrates consisting of (Al,Ga)As/AlAs superlattices have allowed the accurate analysis of metal/GaAs consumption and regrowth reactions that involve as little as 2 or 3 nm of the substrate. Because the reactions are laterally uniform, the complementary large area analysis by backside SIMS or AES provides compositional information on a scale that would require the use of nanometer diameter electron probes if the sample were laterally inhomogeneous.

Atomic structure of nearly ideal metal/semiconductor interfaces: The phase stability, the high degree of crystalline perfection, and the atomic smoothness of interfaces such as $NiSi_2$/Si, $CoSi_2$/Si, ErAs/GaAs and NiAl/AlAs are providing new opportunities to study the structure and properties of nearly ideal metal/semiconductor interfaces. In the past few years, the structures of the epitaxial silicide/Si interfaces have been elucidated by TEM techniques, both plan-view and cross-sectional. Just recently TEM techniques have revealed two structural variants of the Al/GaAs interface. Other techniques such as ion channeling have provided information on the structure of the ErAs/GaAs interface. For all of these interfaces, measurements of Schottky barrier heights show that the barrier height depends strongly on the interface orientation and, in the case of the

silicides with the fluorite structure grown on {111}Si, on the crystallographic variant of the metal film. Furthermore, plan-view and cross-sectional TEM techniques have revealed **interface reconstructions** in at least three of these interfaces ($CoSi_2$/Si, $NiSi_2$/Si, and NiAl/GaAs). As the structural perfection of these interfaces is improved (i.e., larger interfacial terraces), the observation of interface reconstructions at metal/semiconductor interfaces should become widespread, opening up a new area of "surface" science. Given that conventional surface science techniques are severely limited in the analysis of buried interfaces, it is apparent that TEM will play an important role in exploring the science of nearly ideal interfaces. The question is, how do we use TEM techniques to provide information about these buried interfaces?

Several participants in the discussion (including S. Iijima, U. Dahmen, and R. Hull) advocated the use of plan-view techniques including the α fringe technique and the analysis of contrast from interfacial ledges for determining rigid body shifts. Plan-view imaging and selected area diffraction can also be used to study domain boundaries in reconstructed interfaces. D. Cherns discussed the use of CBED to determine interface symmetry as an aid to selecting interface models. Cherns showed that a combination of several techniques including CBED, Bloch waves (which are sensitive to atomic position shifts of 0.01nm), interfacial dislocation analysis, and HREM have yielded consistent results for the two observed structures of the Al/GaAs interface. Information from cross-sectional images alone, as pointed out by R. Hull and others, may be insufficient to uniquely determine the structure of an interface. The HREM technique is subject to beam tilt, specimen tilt, and specimen thickness nonuniformity problems which can completely muddle the determination of a rigid body shift. As with nearly every problem in materials science, the problem of interface structure is best attacked by employing a battery of techniques. The information obtained by such investigations is essential input for the theorists attempting to calculate the electronic structure of these nearly ideal interfaces.

Although a structural determination combined with the calculation of electronic structure may completely describe some interfaces, it is likely that the properties of many interfaces in electronic materials, especially those interfaces that are most perfect, will be dominated by a relatively small concentration of point defects at or near the interface. Certainly this is true for the SiO_2/Si interface in which one interfacial site in 10^4 or 10^5 may be defective. Clearly, TEM techniques are not suited for such discrimination. Many interfaces, however, may contain point defects or point defect clusters with areal densities that are potentially detectable by TEM techniques. Z. Liliental-Weber showed that minute As clusters may be detected by analytical microscopy in cross-sectional metal/GaAs samples. Since such analyses push the limits of analytical techniques (and since the results may be essential to understanding and controlling the properties of many technologically important interfaces), this is an area for future work.

Strain measurements in semiconductor heterostructures: Convergent beam techniques including conventional convergent beam diffraction (CBED) and convergent beam imaging (CBIM) can measure strain in epilayers down to 10^{-4}. Given the technological importance of strained III-V quantum wells and (Se,Ge)/Si heterostructures for bipolar and high-mobility transistors, this capability of convergent beam techniques may find wide application, especially since the technique is compatible with the plan-view sample geometry. The advantage over x-ray techniques is in the spatial resolution.

In summary, TEM has been and will continue to be a cornerstone technique for the characterization of interfaces in electronic materials. The high degree of perfection of these interfaces provides an opportunity to describe in detail the structure-processing-property relationships of an interface with a single atomic structure, uniform over relatively large areas, a feat that is not yet possible with most other classes of materials.

INTERFACES IN MATERIALS II

(K. Krishnan)

Chair: T. Mitchell
Panel: J. Howe, W. Mader, R. Mishra,

In this session, interfaces in metals, ceramics and magnetic materials were discussed. Case studies presented included structural ceramic-composite materials, grain boundary structures, MBE grown ceramic multilayers, ceramic packaging materials, NiO bicrystals, metal-ceramic interfaces, High T_c superconductors, magnetic materials etc. Materials issues such as the relationship between structure and properties as well as questions of technique/instrumentation pertinent to the solving of these problems were discussed.

The following conclusions can be drawn from the discussions in this session:

1. The study of interfaces in such materials is generally complicated. They are predominantly a *function of processing/synthesis* and the interfaces of interest can be further affected by the process of preparing electron transparent specimens.

2. The systems are often non-cubic and the interfaces are generally irrational with a wide range of orientation relationships. *Obtaining samples representative of the true interface is difficult but important.*

3. In many ceramic systems questions of interphase formation and stability are as important as the structure of interfaces. Grain boundary phases and their modification by post-sintering heat treatments are very influential in achieving good properties. In some cases the local chemistry is accommodated by structural changes (polytypoid formation) giving rise to interesting interfaces. *In these studies there is an acute need for high resolution analytical electron microscopy capable of obtaining compositional and bonding information at the nanometer scale of resolution.*

4. The issue of obtaining chemical information from HREM was further raised and discussed. The possibility of using cation displacements to infer anion occupancy and/or to resolve and identify the chemical species of a particular atomic column in HREM was debated. Improved resolution (~1.0Å) was suggested as one possible route. However, it was cautioned that in most such studies at the forefront of the limits of resolution achievable on current or projected (near future) instruments, *interpretation of HREM images would be noise limited.* The noise arising mainly from the specimen (and not from the recording process) could either be overcome by the preparation of super-clean specimens (alternatively, this could also be achieved by in-situ cleaning of the sample) or reduced by signal averaging methods that take advantage of the translational symmetry of the sample. It was agreed that *there is no substitute to preparing clean, uniformly thin, undamaged electron transparent samples representative of the interface to be studied.*

5. The problem of representativeness of the thin foils being studied could be overcome in some cases (such as boundaries with slight misorientation) by controlling growth conditions such that all of them are the same. Alternatively, one could work with model systems and

controlled growth of interfaces to understand general principles of the construction of such structures. In the case of metal/ceramic interfaces this includes epitaxial growth (MBE), precipitation, diffusion bonding and internal oxidation. Samples prepared by the last method and studied by HREM have revealed that the terminating plane at the Pd/MgO is an oxide, i.e oxygen termination (what else!).

6. A study of the structure of a glass/crystalline interface reporting the presence of a partially crystallized intermediate region served as a focus for the discussion of experimental conditions required for obtaining interpretable HREM images. *It was agreed that the alignment of crystal and beam was critical.* Further, it was suggested that one should ensure that (a) there is no damage (ion milling etc.) of the specimen prior to the experiment ; (b) there is minimal radiation damage during observation; (c) the sample should have very little thickness variation; and most importantly; (d) inherent symmetries of the sample in the image should be confirmed while setting up the experiment.

7. Misfit dislocations, not periodically spaced, observed in Au bicrystals led to a discussion on the definition of quasiperiodicity. It was mentioned, that from the theoretical point of view, continuous changes across such interfaces can be accommodated only by insertion of quasiperiodic units if it is assumed that there are no jumps as a function of misorientation. However, it is well known that quasiperiodicity implies the presence of two well defined spacings occurring in a well defined sequence. Given this definition, *such boundaries can at best be called aperiodic unless it is possible to measure over a sufficient length scale to ascertain that the above can indeed be satisfied.*

8. In magnetic materials interfaces play an important role in determining properties. Examples presented included complex multilayer structures used in thin film recording media and *grain boundary engineering issues* relevant to the development of hard magnets. The properties of the latter are almost entirely determined by the structure of the grain boundary phases -- coercivity is a strong function of impurity segregation and domain wall pinning as well as nucleation of reverse domains are influenced by the phases formed at the boundaries. Even though structural characterization is important, it was *emphasized that chemical and magnetic characterization of the inter-granular phase was critical. There is a severe dearth of facilities available for the characterization of magnetic microstructures at the nanometer scale of resolution. An FEG microscope with a DPC stem detector and analytical capabilities would go a long way in solving such questions.*

9. Finally it was emphasized that *all such problems should be addressed by a wide range of methods and measurements*. Key microstructural factors should be identified -- for example, in high T_c superconductors it was mentioned that T_c increases with the sharpness of the twin boundary, J_c is determined by the grain boundary structure etc., and *efforts should be made at not only characterizing them both structurally and chemically but correlating such measurements with appropriate properties.*

INTERFACE PHENOMENA I

(R. Gronsky)

Chair: J.M. Gibson
Panel: J. L. Hutchison, Y. Ishida, F. Ponce

The session opened with some questions concerning the preceding overview presentations by M. Gibson on *TEM of Interfaces* and A. King on *Crystallography of Interfaces*. This led into a debate on the nature and visibility of dislocations in grain boundaries and the usefulness of the CSL concept.

(Gronsky): Why in the Σ41 boundary is there no strain contrast?

(A. King): The dislocations are just not resolvable..except perhaps by weak beam. The problem is that DSC dislocations are identical to perturbations in the lattice dislocation array. The result depends very much upon the technique used.

(Merkle): At some point I will show another example of an Σ41 boundary at which the strain fields are visible.

(Olson): Doesn't the core width matter? For a given spacing, if the cores become too diffuse, won't they be invisible? I'm trying to bring this back to a physical model -- at what point does this become a problem?

(A. King): The issue of lattice dislocations vs. DSC dislocations has not been resolved. In the range of 12-25° misorientation you can treat the boundary as either small or large angle ... it is the properties that are important.

(Bonnet): We need a good elastic theory. When there is a family of one kind of dislocation, there is no long range strain field (Read & Shockley) in isotropic media. In anisotropic media, there are long range strain fields! (I have calculated them).

(Vitek): When the dislocations follow the Frank formula, then they cannot have long range strain fields (by definition!). The dislocations are simply the structure of the boundary! The field of the boundary is described by the field of the dislocations -- these are simply the tools to describe the structure.

Lively discussion ensued until terminated by the chairman and postponed for solution later in the week.

(Mader): A comment on the overview lecture (by M. Gibson): your work on semiconductor materials does not translate easily to metals and ceramics.

(Gibson): Yes, semiconductor materials are made with unusually large flat interfaces but efforts to make similarly well-defined boundaries in other materials are underway, for example W. King's project on diffusion bonding, based on the Stuttgart experience.

(W. King): All of our experiments are driven by theory. We try to produce the precise interfaces for which theoretical predictions have been made. For example we can now diffusion-bond Nb, flat to 30 Å, with only ~0.1-0.2° variation in misorientation.

(Mader): The problem is not only to produce interfaces, but to make TEM specimens as well. The largest problems come from sample preparation: amorphous layers due to surface damage or contamination. Don't these affect the image?

(Gibson): Yes, especially in a plan view dark field image the resolution is limited by noise. But for a periodic object and with spatial resolution limited to 30 or 40 Å the signal to noise ratio can be very high.

(Mader): In the thermodynamic sense, grain boundaries are not as stable as interphase boundaries. Misfit dislocations are an inherent part of interphase boundaries, even stand-off dislocation which are not located directly in the interface.

(Pirouz): In interphase boundaries, where the bond type changes, for example at metal-ceramic interfaces, the idea of coherency, semi-coherency and incoherency may have to be revised. Geometrical constructions like the CSL may be insufficient. Often we don't see misfit dislocations.

(Hull/Gibson): Regarding the change in bonding at interfaces, the value of the structure factor F_g at low scattering angles is very much dependent on ionicity and could even be used for analysis of the bonding (Spence wrote a paper about this).

(Hetherington): The F_g differences show up in smaller scattering angles, so these may be seen in Fresnel effects, but are difficult to see otherwise. One way to remove the uncertainties is to compare similar reflections at low and high scattering angles such as {200} and {420} in GaAs which are both due to the difference in scattering factor between Ga and As. This comparison might give information on ionicity.

(Howe): We have been concentrating on the problems. Why not be more optimistic? In surveying the literature on all types of interfaces, I'm impressed by the success of simple geometrical models. Simple rules like minimizing surface and strain energy go a long way to understanding most interfaces.

(W. King): For bcc metals, you can throw all of those intuitive models out the window.

(Hutchison): Since the phases of the electron waves vary with thickness,the two sides of an interface may behave differently. This is a serious problem for the determination of rigid body shifts when the image on one side is black dot and the other side is white dot, for example in $NiSi_2$ on Si (slides) for some thickness ranges. It is very important to know the precise thickness of the foil.

(van Tendeloo) This will become even more problematic if there is preferential thinning on one side of the interface.

(Cherns): Because there are problems with reliability of lattice images we are now looking at plan view specimens where the different view gives us new, complementary information. (Slides) The question of roughness can be addressed by looking at samples in plan view by CBED. This allows you to sweep out the reflection and carefully examine its shape for changes in structure factor or strain. A direct image reveals only a single point of the rocking curve. Imaging shows that layers may be buried in regions of various thickness. This method of looking at specimens in plan view is very sensitive to buried interfaces, and it is the buried interfaces that are the most important. The resultant contrast is the same as for stacking faults. Applications of this technique are: interface roughness; strains down to 10^{-4}, rigid body displacements.

(Ishida): Experimentally, how do you exclude effects of surface roughness?

(Cherns): The amplitude/phase diagram shows that this has only a small effect.

(Hull): Doesn't this overlook the most important piece of data: the power spectrum of the roughness because this is what can be correlated with macroscopic properties such as luminescence.

(Sinclair): From a materials science point of view, what is important is whether the interface influences the macroscopic properties of the material. (Slides) The barrier height of Ti Schottky barriers change very little with the thickness of an amorphous Ti-Si layer, but drops to near zero when the amorphous layer crystallizes. The breakdown field of a thermally-grown SiO_2 oxide on Si doubles after annealing removes the roughness of the as-grown interface. In both cases there is a clear correlation between effects observed in cross section and macroscopic properties.

INTERFACE PHENOMENA II

(A. Hütten)

Chair: G. van Tendeloo
Panel: K. Krishnan, S. Ranganathan, L. Thomas

This session addressed further generic interface phenomena such as segregation, precipitation, ordering and the concepts of rough vs faceted and sharp vs diffuse interfaces.

In response to comments by some experimentalists it was emphasized by several theoreticians that the coincidence-site lattice (CSL) description is a purely crystallographic device and as such cannot predict interfaces. The CSL is a convenient tool to describe and classify which interfaces can exist but it cannot predict which ones will be favored. Observed interface structures can be rationalized in the framework of the CSL but not predicted a priori. For example, investigations of the energy of <110> tilt boundaries in gold as a function of their inclination showed there is no direct correlation between the grain boundary energy and the density of coincidence sites.

As an example of precipitation at coherent interfaces, HREM images of the S phase in the Al-Li-Cu-Mg system were presented. This phase precipitates at the interface between the Al matrix and Al_3Li particles in a well-defined orientation relationship: (100) S-phase parallel to (012) Al matrix. These precipitates formed far from equilibrium and their heterogeneous nucleation was thought to be aided by the segregation of Cu and Mg in the interface of the growing Al_3Li phase. It was pointed out that *kinetic* segregation, i.e. rejection of solute from an interface can enhance heterogeneous nucleation while *equilibrium* segregation will tend to inhibit it. The S-phase precipitates appeared microfaceted and the question the interface state (rough or faceted) arose.

HVEM results of cyclic heating experiments of Ge precipitates in an Al matrix were shown. At low temperature the particle was faceted with octahedral shape whereas at high temperature, it was spherical. When the temperature was cycled, it was observed that the particle transformed from the faceted to the spherical shape at constant volume. It was concluded that the observed shapes were equilibrium shapes. It was proposed that the macroscopic shape change of the particle

corresponded to a microscopic change in interface structure. The octahedral shape reflecting a faceted and the spherical shape a rough interface structure.

As an example for diffuse interfaces, HREM images of the antiphase boundaries in Cu_3Pd at room temperature and at T = 500°C just below the order/disorder temperature of T = 507°C were shown. Whereas at room temperature the antiphase boundaries were sharp interfaces, they appeared to be broad, or diffuse, at 500°C.

These examples led to intense discussion of the differences among *faceted*, *sharp*, *rough* and *diffuse* interfaces. Several suggestions were made for a useful distinction between these types of interfaces, but simple characteristics that would allow the distinction to be made from TEM images were difficult to find.

One problem was the fact that the concept of a faceting/roughening transition at a critical temperature was originally introduced for solid surfaces or solid/vapor interfaces. At the critical temperature the step energy disappears and the surface becomes atomically rough, i.e it fluctuates about its mean position by a random arrangement of steps. However, at any time the interface is sharp, i.e. any one atom is clearly either in the solid or the vapor. For solid/solid interfaces it is possible to have a faceted yet diffuse boundary in which some parameter (order, structure, composition) changes gradually rather than abruptly. Such a boundary will have a finite width and would be called diffuse. The measurement of a parameter such as order, structure, composition from TEM images with sufficient resolution to determine the degree of diffuseness or roughness is not unambiguous.

Diffuseness is the thickness, or extent of the boundary normal to itself, whereas roughness is a fluctuation of the interface location. Chemical diffuseness is easy to visualize as a concentration gradient or gradual change in composition. Diffuseness with regard to an order parameter can be understood as a gradual change in order parameter, but there is an inherent limitation due to the fact that order at a given lattice site can be defined only relative to its neighbors. For example, if order is defined in terms of third nearest neighbors, then even the sharpest, most abrupt interface will appear at least as wide as the third nearest neighbor distance. Structural diffuseness is most difficult to visualize. Like order, structure also is defined in terms of coordination by neighboring atoms and as such is subject to the same inherent resolution limit as the order parameter. But in addition to this limitation structural diffuseness could only be realized by either a time-averaged rough boundary whose roughness fluctuates with time, or as a geometry in which small islands of one phase project into the other. Either situation avoids the problem by describing an average structure.
Both a faceted interface that is atomically flat or a rough interface with random steps can either be sharp (zero width) or diffuse (finite width).

It was suggested to use plan view diffraction techniques to determine not only the extent but the power spectrum of interface roughness. High resolution micrographs can reveal roughness or faceting but must be interpreted with caution if there is the possibility of steps along the beam direction.

HREM QUANTIFICATION I

(C.J.D. Hetherington)

Chair: R. Hull
Panel: L. Marks, M. Mills, J. Thibault

This session addressed experimental issues such as imaging artifacts/ misalignments/non-linearities and specimen relaxations, and their effect on quantification, such as rigid shift measurements, (the underlying materials science issues to be discussed elsewhere).

An overview of the problem

In the 3-dimensional object, there are N atoms each described by coordinates (x_i, y_i, z_i) and atom type Z_i, a total of 4N parameters. But, in general, we have images from only one or two projections from which to calculate these parameters. So mathematically, the problem cannot be solved rigorously.

What can be learned? Information about, e.g., a grain boundary, can be classified at three levels:

1. overall geometry: step height/Burgers vectors, rigid shift, orientation
2. atom positions (x_i,y_i): using maximum intensities in image or pattern/shape comparison with simulated image
3. chemical composition: Z_i

Comparison of images, simulated and experimental

Typically, an experimental image is compared with simulated images of the various models by simple eye inspection. A more quantitative procedure might involve locating the positions of the intensity maxima or minima, mapping the displacements between positions in the experiment and in each simulation, then comparing the displacement maps and calculating standard deviations.

Since the intensity of a (white) atom image is generally rather "flat-topped", a better location of the atom image might be the centroid of the contour of another intensity level--e.g., 90% of maximum--rather than the maximum itself.

It is preferable to compare experiment and simulation for a through-focal series and for several thicknesses. A match for a minimum of 3 thicknesses or 3 defocus values was suggested before confirmation of a model structure could be established. (3 meaning that 2 measurements establish the "straight line" and the third serves as a check.)

Interpretation of image detail at close to the point resolution of a microscope, such as the detail found in interfaces, is a dangerous area to work in because of the potential for spurious images. A flat transfer function (i.e. higher resolution microscope) is desirable for this reason.

The computation itself should be treated as an experiment. Perturbation of simulations, such as varying the slice thickness in a multislice calculation, should be performed to ensure convergence.

The ultimate goal is to refine the atom positions by matching simulated images to each experimental image, improving the match for each successive simulation. A non-linear least squares fitting procedure has been applied to a "perfect crystal" image to establish the thickness and defocus. (An "image map" is first used to estimate these values.) Atomic position and occupancy remain to be treated. Many of the calculations, such as images in a through focal series, are ideally suited to parallel processing.

The R-factor

The R factor is (basically) the normalised difference between predicted and experimental intensities and ranges in value between 0 and 1.0. 0.6 would be the value to expect if one image was of a random structure (i.e. totally wrong).

Instead of the R-factor by itself, the ratio of R-factor to errors (χ), is the appropriate quantity to use. With 20% error, an R-factor of 20% is a good fit; with 1% error, an R-factor of 20% is a bad fit.

The sensitivity of R to a parameter (i.e. the slope of R either side of the minima) should be noted.

Error bars should be measured by independent, non-theoretical methods.

Errors are to be found in the simulation as well as in the experiment (e.g. Debye-Waller factors and absorption parameters are, to a certain extent, unknown parameters and themselves contribute to the error bars).

An R-factor of 0.3 was found for an image of Th-superconductor (translationally averaged to reduce amorphous noise) yet inspection by eye shows the match is clearly wrong in some places and in good agreement in other places.

If R is 0.3 for the perfect and known structure, how do we cope with unknown defect structures such as interfaces?

Noise in the image

Noise in the image from surface layers (e.g. oxides, contaminants) can be treated as experimental error. Several experiments (i.e. images from different specimens or different areas of one specimen) can reduce this error, as can averaging within one micrograph.

It is a good idea to take note of the standard deviation between the images that make up the final averaged image.

Alternative treatments include: removing the oxide layer (!), adding oxide layers to the multislice calculation, or adding noise to the simulated images.

Surface relaxation of thin foils

The thin foil relaxations of strained systems can be calculated or modelled and then included in the image simulation; in any case, they should not be ignored. Thin foil relaxations at boundaries in unstrained systems (i.e. no long range stresses) can occur as a result of the interfacial tension in the boundaries but the relaxations would be of a smaller magnitude.

Thin foil relaxations can have long range effects, especially at hetero-interfaces in strained systems. If in the "bulk", with layer normal along z, we have $\sigma_{xx} = \sigma_{yy}$ (suppressing the lattice

parameter difference), $\sigma_{zz} = 0$ and σ_{xy} (etc,.) = 0. Then in a thin foil, $\varepsilon_{xx} \neq \varepsilon_{yy}$ and $\sigma_{xy} \neq 0$; this can affect the observed rigid body displacements and lattice parameters. These relaxations vary with the distance from the boundary; the width of the effect is of the order of, or greater than, the foil thickness and can be up to thousands of Ångstroms. 10^{-3} strains correspond to tenths of an Å which is of the order of the distances that can distinguish alternative models.

The "embedded atom method" has been used to calculate the effect of the free surfaces in a thin foil for atomic models of a dislocation and a boundary. The relaxed structures were then input into a multislice calculation of the image. Encouragingly, it was found that the thin foil relaxations did not have a noticeable effect for a boundary in aluminum with a foil thickness of 60Å.

Misalignments

The whole atom column is affected by crystal tilt, not just the surface atoms as is the case in some thin foil relaxation effects.

In certain circumstances, the crystal tilt can be measured or estimated sufficiently accurately for satisfactory inclusion in image simulations.

Beam misalignment is generally less than the beam divergence semi-angle; but even then, a misalignment of a few tenths of a milliradian can severely affect the phase change at those scattering angles where the slope of the CTF is large. This includes the scattering that corresponds to the low resolution (10Å or more) and high resolution (2Å or less) detail that is present in an interface, but absent from the bulk structures either side of an interface.

The contrast changes that are frequently observed across lattice images of twin boundaries are one common effect of misalignments.

A rigid shift in the image of one fifth of the fringe spacing can be brought about by a crystal tilt of 5 mrad or a beam tilt of 1 mrad. Early measurements of rigid shifts may have been incorrectly interpreted in terms of wrong structures rather than experimental misalignments. The technique of generating moiré patterns between a lattice image and an artificial, perfect array of dots can reveal rigid shifts and other long range strains.

Miscellaneous

In many cases it is impossible to verify the chemistry by HREM, but in some particular circumstances it is possible.

As an example of the difficulties in learning about the chemistry, a single experimental image could be matched to images calculated from any one of five different models (in which silver and copper were substituted for gold) if the focus and thickness were allowed to be varied slightly.

Radiation damage can limit the experiment at a fundamental level (it's like trying to shoot a moving target); it has been noted that damage can occur more readily at a defect or interface than in the bulk material.

Distortions are present in EM projector lens (3% in 200CX, <1% in 4000EX), and the photographic process.

HREM QUANTIFICATION II

(J. Howe)

Chair: R. Sinclair
Panel: R. Gronsky, W. Krakow, T. Mitchell

The purpose of this panel as outlined by the Chairman, R. Sinclair, was to discuss: i) analysis of the structural and chemical diffuseness of interfaces by AEM and HREM, ii) analysis of interface topography/roughness, and iii) the projection problem in HREM. Presentations were given in each area by participants in the workshop, and summaries of the most important points from these presentations and the accompanying discussion are summarized below. A brief set of conclusions is then provided.

i) Chemical and Structural Diffuseness

Presentations in this area were provided by R. Sinclair, K. Krishnan, J. Bentley, L. Thomas and J. Hutchison. Initial discussion concentrated on chemical analysis by EDS and EELS. Several examples were given where interface chemistry was determined with a spatial resolution of 2nm by EDS including segregation of 0.1 wt%P to grain boundaries and Sb to dislocations in steel. The question arose as to whether 1nm resolution is feasible with a FEG. For EDS, concern was expressed that problems such as probe broadening, radiation damage, drift, contamination, sample thickness, x-ray fluorescence and interface tilt may still limit the resolution to about 2nm. However, consensus was reached that 1nm resolution is possible with PEELS, although there still may be difficulties in various materials due to the problems mentioned above. Optimization of microscope operating conditions and techniques such as extrapolation back to zero thickness and zero dose may help alleviate some of these problems. Z-contrast STEM was shown to be a nice complement to EDS although averaging of signal through the specimen thickness is a limiting factor. An example where atom-probe analysis was able to detect a 1.2nm layer of B at a grain boundary in Ni_3Al demonstrated that this is a useful technique for chemical analysis of interfaces.

In HREM it was shown that chemical discrimination at interfaces is not optimized in thin samples at Scherzer defocus. Particular thicknesses and objective lens defocus values can be used to distinguish between chemical species across semiconductor interfaces and this should apply to other materials. Techniques for determining the occupancy of atoms in a single column need more development.

ii) Interface Topography

A brief presentation in this area was provided by R. Sinclair. Examples were shown where defects such as ledges and dislocations at interfaces were determined at 0.18nm resolution. The question then arose as to whether this resolution is sufficient. It was pointed out that local distortions cannot be resolved to better than the instrument resolution, and therefore, there is a need for better resolution, particularly in metals and ionic crystals. Better software is also needed to extract all the information from images taken at even 0.18nm resolution, particularly where surface noise is a problem. FIM profiling through boundaries is capable of revealing detailed interfacial topography and this technique could be pursued further.

iii) Projection Problem in HREM

Presentations in this area were provided by W. Krakow and J. Howe. This remains a difficult problem although some progress is being made in both chemical and structural discrimination along a column of atoms by extensive computer simulation. Imaging of a boundary along several different zone axes and good experimental design are particularly important for revealing the three-dimensional atomic structure of a boundary. It appears that HREM is not able to reveal the position of an atomic column to better than about 0.01nm accuracy in many materials and that HREM is very sensitive to the average Z of a column at one extinction distance thickness, almost to the point of single-atom discrimination in some alloys.

Conclusions

i) 1nm chemical resolution is possible with a FEG and PEELS although there is always a trade-off between signal, radiation damage, contamination, etc.. EDS is more limited in resolution than PEELS mainly due to beam spreading.

ii) APFIM provides single-atom discrimination normal to an interface and is complementary to other analytical techniques.

iii) The use of relatively thick samples and proper defocus conditions are necessary for chemical sensitivity in HREM. Chemical discrimination at 0.5nm resolution is readily possible.

iv) Better than 0.18nm resolution is necessary for imaging defects in a greater variety of materials.

v) Deconvolution of information along the third-dimension in TEM samples is becoming more critical and some progress is being made in this area.

INTERFACE MODELING

(M. Mills)

Chair: V. Vitek
Panel: R. Bonnet, S. Dregia, R. Kilaas, P. Pirouz

The discussions covered four broad topics: (a) complexity of real interfaces; (b) the geometrical construction of interface boundaries; (c) modeling of boundaries using elasticity theory; and (d) atomistic modeling of interfaces.

A. Complexity of Real Interfaces (*Pirouz*:)

In general, interfaces in materials can be extremely complicated. This complexity can arise for at least two reasons. First, if one or both of the perfect crystals bounding the interface are themselves complex, with large unit cells, then this complexity may be reflected in the interface. An example given was the precipitation of an alumina phase in a Cu matrix. The attempts to determine the interface structure using HREM require that reasonable models of the interface be constructed. In this case, the large unit cell of the alumina phase makes the construction of these

models extremely difficult. The ability to rapidly construct an arbitrary interface between different materials would be very advantageous. A method for constructing such interface models is discussed by R. Kilaas in the next section.

Even if the interface is formed between materials with relatively simple crystal structures, the boundary structure itself may be intrinsically complex. An example of this second type of interface complexity is the Σ3 (211) grain boundary in 3C-SiC. The observed structure in this case has been compared to four different models which have previously been proposed for the same boundary in Si. The models included a structure with a plane of mirror symmetry at the interface, as well as three others which have rigid body shifts parallel and perpendicular to the tilt axis of the boundary. However, comparisons between simulated images based on these models and the observed structures have failed to produce satisfactory agreement. The reason for the lack of agreement seems to lie in the fact that there is no long range periodicity in the observed boundary which may be the result of anti-site bonding.

Comments and Questions:

(*Dahmen*): Studies suggest that a great deal of insight can be gained by starting with a geometrical model and making certain reasonable assumptions such as: (a) optimizing the nearest neighbor distances or (b) minimizing the number of dangling bonds. However, this approach clearly depends on the type of bonding, and it is difficult to predict what factors will be most important in determining the structure.

(*Vitek*): The simple atomistic approaches such as those using pair potentials automatically account for the optimization of nearest neighbor distances. The "tight binding" approach can allow for a more rigorous consideration of the effects of bonding at defects as well. However, rather than expecting to obtain precise models, even the tight binding calculations should be used simply as a guideline in the interpretation of structure images.

(*Hetherington*): It should also be pointed out that whenever possible the interface should be viewed in orthogonal directions. This technique provides direct, 3-dimensional information concerning rigid body shifts at the interface which can be used to distinguish between various models, such as those discussed above for the Σ3 (211) grain boundary in 3C-SiC.

B. Geometrical Crystallographic Construction of Interfaces (*Kilaas*):

The ability to construct geometrical models of a general interface is a common starting point for those interested in both the calculation of relaxed structures or the simulation of HREM images of interfaces. A program is now available through the NCEM which runs on a VAX workstation (and soon on a Mac as well) with which general interfaces between similar or dissimilar crystals can be generated. The program is fully interactive and icon-driven, making it easy to learn and to use, allowing for the rapid construction of interface models. The steps to using the program are as follows:

a) Define the crystal structures and lattice site occupancies for the two crystals.
b) Rotate the crystals into the desired orientation relationship.
c) Display the interface structure assuming a planar interface.
d) Using a "mouse", interactively define alternate, arbitrary (planar or non-planar) boundary paths and change atom types and positions if desired.
e) Perform rigid body shifts both perpendicular and parallel to the boundary plane.
f) Define periodic lengths and store final configurations as a supercell file for use in HREM simulation or atomistic relaxation programs.

Comments and Questions:

(*Vitek*): The possibility of incorporating a scheme for relaxing the constructed interface was raised. However, it is not clear what method to use for the relaxation. At this point, the program would be more generally useful if subsequent optimizing of the structure were left up to each user.

(*Howe*): It might be useful to include a projected potential map of the generated structure to get a crude idea of the weak phase object image.

(*Shiflet*): The ability to rotate the structure and obtain a 3-dimensional view of the potential mapping might provide ideas about how to move the atoms in order to optimize nearest neighbor distances, etc.

(*Olson*): In addition to the ability to define an arbitrary path for the interface in the chosen projection, it would also be helpful to be able to define an inclined interface plane relative to the viewing direction.

C. Elastic Relaxations of Faceted Interfaces (*Bonnet*)

Very close to defects such as dislocations, grain boundaries and interphase boundaries, elasticity theory is not expected to be accurate due to singularities in elastic solutions. However, comparisons between elasticity theory and the observed atom displacements around stair-rod, matrix dislocations in both Ni and Al have demonstrated good agreement up to quite near the "core" region.

The extension of elasticity theory to the case of interfaces has now been made by use of Somigliana dislocation constructions. With this approach, the crystal is cut into faceted sections and the proper (observed) angle between the crystals is created. The two crystals are then placed together and the atom positions are relaxed about the Somigliana dislocations which have been inserted to accommodate the angle between the two crystals. The comparison of HREM images of interfaces with elasticity theory has several advantages:

a) The ambiguity of having two different elastic fields at a heterophase interface can be treated.
b) One can use closure circuits to analyze defects at interfaces.
c) Intrinsic stresses around interfacial steps can be detected.
d) The atomic structure of the interface is generated and can be used as input into image simulation programs.

An elasticity analysis has been applied to several complex interfaces. The section of the Ni_3Nb/Ni_3Al interface presented has facets separated by small ledges which are associated with interfacial dislocations of four different types. By comparing the observed image with elasticity solutions assuming different defect types and positions, it was possible to deduce the character of the interfacial dislocations. Several other examples were discussed including $\Sigma 3$ and $\Sigma 41$ grain boundaries in Ni_3Al and NiO, respectively, as well as other heterophase boundaries such as interfaces in the Ni_3Al/Ni_3Nb and Si/SiO_2 systems.

Comments and Questions:

(*King*): It would be interesting to compare the results for isotropic and anisotropic solutions. Is it possible to get as good a match with isotropic elasticity?

(*Vitek*): This approach is particularly useful for complex interface structures, where atomistic calculations might be intractable. However, the structural units which result from the atomistic calculations can be used as input data for fitting the elasticity solutions to the observed structures.

(*Bonnet*): It should be pointed out that this approach is strictly applicable only for cases in which the misfit across the interface is negligible.

D. Atomistic Modeling of Interfaces (*Vitek*):

This topic has already been partially treated in an earlier contributed paper by V. Vitek. The treatment of the relaxations depends on the description of the atomic interactions. Three treatments are discussed below in order of increasing level of description:

Pair potentials treat the total energy as a pairwise sum over all bonds in the system. No explicit description is given for the electron density, which provides for the cohesive energy of the system. Consequently, pair potentials are usually performed under constant volume conditions.

N-Body potentials (EAM) treat the total energy of the system as the sum of electrostatic interactions, V_{ij}, and an "embedding function", F_i, which is a function of the local electron density contributed by the surrounding atoms. The functional dependencies of V_{ij} on R_{ij} (where R_{ij} are the interatomic separations) and of F_i on r_i (where r_i is the electron density) are empirically defined by fitting the functions to known physical properties such as the lattice parameter and vacancy formation energies. In practice, only relatively subtle differences have been found between grain boundary structures calculated using pair and N-body potentials. However, the N-body approach should be advantageous for the treatment of (a) alloys; (b) HCP metals (where the potentials can be fit to the correct c/a ratio); and (c) surfaces. In the generation of the potentials, it is vital that one ensures that the correct crystal structure is truly stable for perturbations of stress and temperature.

Semi-empirical band structure (tight-binding) calculations solve an effective Schrödinger equation assuming that the total wave function of the system can be represented as a sum of individual atomic-like wave functions. As in the N-body calculations, the repulsive part of the total energy is normally derived empirically. The advantage of this approach is that the electronic contribution to the total energy, including angular effects to the bond energies, is described more accurately than in the N-body calculations. A detailed accounting of the electronic structure is necessary to understand certain reconstructions at surfaces, and is likely to be particularly important for interfaces in covalent materials where N-body approaches are not strictly applicable. Tight-binding calculations can also be used to treat interfaces between dissimilar materials. Although it is computationally more intensive than the simple interatomic potentials, tight-binding calculations can nevertheless be applied to fairly large ensembles of atoms. Consequently, tight-binding calculations can been used to calculate relatively complex (larger period) defect structures, and appears to represent a useful compromise between *ab-initio* approaches and the simplest interatomic force descriptions.

E. Interphase Boundaries Modeling Using the EAM (*Dregia*):

The thermodynamics of planar interfaces between dissimilar materials has been studied using elasticity theory and the Embedded Atom Method (EAM). For the case of a deformable layer on a rigid substrate, the interaction between interfacial misfit dislocations is a general Fourier series with both misfit and elastic energies. For this case it is possible to obtain a closed form, analytical solution with which to compare against the EAM calculations. The interfaces studied are composed of periodic displacement fields with the periodicity of the O-lattice. One-half of the total relaxation energy goes into accommodating the misfit. Special cases which have been considered are for both 1-dimensional and rotational misfit.

The EAM has been tested against the analytic solution for the case of a monolayer of Ni on a rigid Ag substrate for the [111] non-centrosymmetric orientation. Good agreement is found between the analytical and EAM models. The EAM has also been compared with experiment for the case of the Ni/Ag and Cu/Ag interfaces in this orientation. These systems are particularly suitable for the study of interfacial relaxations since there is very limited solid solubility between Ni (or Cu) in Ag. For various twist angles about the [111] rotation axis, it is found that the misfit dislocation grid is trigonal rather than hexagonal. Finally, dihedral angle measurements have enabled the experimental determination of excess interfacial stresses for the Cu/Ag system. From these measurements, close agreement is obtained with the calculated values of normal interfacial stress.

ADVANCES IN IMAGE SIMULATION AND ANALYSIS

(M. A. O'Keefe)

Chair: P. Pirouz
Panel: S. Iijima, J.-M. Penisson, K. Merkle, G. Anstis

The session addressed the current state of image simulation, image processing, and image analysis, especially as applied to interface problems, and looked at ways of improving these techniques in order to derive information about interface structures. Although many of the presentations described specific applications, discussion focused on methods and procedures for using image simulation reliably in order to interpret experimental images and thus derive structural information.

Structure Modeling

Current simulation programs can accommodate quite large structure models (of the order of 100Å by 100Å in extent), and are thus very suitable for simulations of interface structures; e.g. the "unit cell" of the Σ99 (557) 89.4° [110] grain boundary model produced by Vitek measured 28.4Å (the repeat distance along the boundary) by 163Å (a distance more than sufficient to well-separate the periodic grain boundaries produced by the use of discrete Fourier transforms). In fact, whole specimens can be modeled; Mills showed an image simulation for a Σ9 (221) 60° [110] grain boundary in an aluminum thin-foil specimen of 60Å thickness, in which the whole foil had been modeled, including relaxation effects at the surface. A multislice calculation was then carried out, with each layer having the appropriate structure for its depth in the foil. It was suggested that comparisons of the simulated images with experimental ones could be used to provide insight into the atomic potentials used to construct the grain boundary model.

Merkle demonstrated how comparison of experimental images with simulations from a model of a Σ41 grain boundary in nickel oxide could be used to gauge vacancy concentrations. He showed that the best match occurred when 25% of vacancies were placed in the atomic columns of the oxide.

Anstis applied image simulation to intrinsic faults in silicon and found that the calculation suggested that the presence of impurities is required to render a grain-boundary visible under weak-beam conditions.

Procedures for Image Matching

It was agreed that to unambiguously quantify a specimen structure required a match between experiment and simulated images for a number of values of specimen thickness and objective lens defocus. Penisson pointed out that, in addition to the more-obvious parameters such as objective lens defocus, spherical aberration, beam convergence, spread of focus and specimen thickness, care must be exercised in choosing suitable values for Debye-Waller factors, absorption coefficients (for thicker crystals), sufficiently-fine sampling (i.e. large-enough g_{max}), and choice of output scale for halftone mapping.

Penisson showed simulations for a $\Sigma 41$ grain boundary in molybdenum in which atoms positions matched the positions of the centers of black spots at -450Å defocus, but were offset from the centers of white spots at -760Å defocus; he suggested that such problems be avoided by plotting the total phase of each contributing beam as a function of specimen thickness and lens defocus in order to select values of defocus and thickness near stationary points where the rate of phase change is minimal. In a related technique, O'Keefe suggested the use of image maps consisting of images of perfect structure for a wide range of thickness and defocus; such maps combine the effects of all the contributing beams and highlight regions where image character changes only slowly with defocus and thickness. These regions can then be investigated for suitability as conditions for HREM of defect structures; an example shown was an image map for aluminium which was used to select the conditions used to image a $\Sigma 99$ (557) 89.4° [110] grain boundary.

Specimen Thickness

Although defocus values of experimental images are generally known to within a few tens of Ångstrom units (an error of less than 10%), specimen thickness is difficult to determine with the same precision. This imprecision can lead to ambiguities in attempting to distinguish between different structural models An example was shown where a single experimental image could be matched to any one of five different models if the defocus and thickness values used in the simulation were varied slightly.

In addition to the uncertainty in the experimental value of specimen thickness, the variation of simulated images with thickness is sensitive to several parameters. As well as the obvious effect of any change in incident electron energy, changes in apparent specimen thickness may be produced by changes in Debye-Waller factors, by changes in the degree of ionicity used in computing the atomic scattering amplitudes, and by slight misorientations of the specimen or electron beam. Penisson showed an example in which a 30% increase in the value used for a Debye-Waller factor produced a 10% increase in extinction distance, i.e. in the apparent specimen thickness. O'Keefe showed how small increases in ionicity produced similar decreases in the extinction distance.

Discussion on measuring specimen thickness included suggestions to tilt crystals containing defects by a known amount, to coat specimens with small particles on both sides and tilt (*after* any HREM imaging), and to compare computed extinction lengths with wedge crystal images. It was noted that the last method is sensitive to changes in extinction length caused by any off-axis tilt of the specimen.

Specimen Tilt

Hetherington demonstrated that crystal tilts in images of well-characterized materials can be satisfactorily modeled by image simulation. He showed an experimental image of multiply-twinned and hexagonal silicon in which the relative tilts between the matrix, two twin variants, and the hexagonal phase are known. Image simulations of silicon crystal at the relevant tilts produced matches to the various regions of the micrograph, and enabled models of the interface boundaries to be derived and tested against the experimental image.

O'Keefe showed how small crystal tilts can be used to simplify the interpretation of images of slightly thicker specimens by extending the limit of the thin-crystal "weak-phase-object" -- by a factor of two in the case of a one degree tilt in [110] β-SiC. He suggested that such small tilts are often present experimentally, since they produce "better" images at greater thickness than exact zone-axis orientations. Mills produced an image of a screw dislocation in Ni3Al, in which a twist effect introduced sufficient mis-orientation to produce a "thin-crystal" image around the dislocation, even though the surrounding matrix was too thick to have thin-crystal character when imaged on-axis.

Absorption

Penisson remarked that absorption should be included in image simulations for thicker crystals, but that the values suggested by Radi are too large by a factor of two. Marks agreed that absorption should be included, but pointed out that no image match has yet been shown to be improved by including the effects of absorption in the image simulation. The role of phonon-induced inelastic scattering in contributing to the image is still a matter for disagreement in the literature.

Reliability of Image Simulation Programs

The question of the accuracy of image simulation results was brought up; i.e. are workers too trusting of the results obtained from image simulation programs. Three problems can occur. Firstly, the program used may incorporate some faulty algorithm; secondly, the program may accept a parameter value that lies outside the range of values that is appropriate to the approximations used in the program; thirdly, an incorrect value may be entered, either because an accurate estimate is not available (for a Debye-Waller factor e.g.), or as a simple error in entering data values.

The last problem can be minimized by careful checking of input data; some programs are sufficiently sophisticated to notify the user of parameter values that seem outrageous or unphysical (e.g. later versions of SHRLI will warn of atoms closer than some user-selected value, which the program sets to 1Å by default). Similarly, programs should be capable of warning the user when parameters approach the limits of the built-in approximations. Faulty programs can be checked against ones which are known to be accurate. For example, the SHRLI programs have produced excellent matches with experimental HREM images of many different structures in numerous publications over 15 years, both in bright-field and dark-field, with the crystal on-axis and tilted by up to 23 milliradian, over extensive through-focus series, and for thicknesses up to 300Å. Results from other programs have been compared with SHRLI results by various workers; Marks presented a list of such persons known to him, and other names were added by various workshop participants. While not all programs were represented (e.g. the Van Dyck multislice programs have undoubtedly been compared with others, but no-one present knew by whom), quite a comprehensive set emerged (figure 1). The consensus of the workshop was that all widely-used programs are essentially correct. Note that the NCEMSS programs have been thoroughly checked, and are available from the NCEM for running on any DEC™ VaxStation® using the VMS® operating system.

Image Processing

Iijima showed some results from "interfaces" in small particles of a partially-ordered $GaInP_2$ structure. Fourier filtering of recorded HREM images was used to enhance the diffuse scattering contribution to the images. Chemical information then became visible in the form of domains in the particle images showing those regions enriched in Ga or In, and those showing superstructure character.

Johnston showed how the directions and degree of linearity in images of bi-crystals could be characterized by processing with Hough transforms to construct a plot revealing the amount of grain boundary lying along any particular direction.

IRRATIONAL FEATURES

(W. Mader)

Chair: A. King
Panel: C. Forwood, B. Muddle, G. Shiflet

Radiation Damage at GaAs/Au Interfaces (*Z. Liliental-Weber*)

Interfaces between GaAs and Au, prepared by ion milling were very unstable under the electron beam. At 800keV hole formation was observed within two through-focus series. Microcrystallites were formed at the foil edge of GaAs which, however, did not form at 200keV (JEM 200CX). No such damage was observed at crushed specimens even in the high voltage electron microscope.

This contribution evoked a vivid discussion on radiation damage:

(*Marks*) Differentiate between ESR (Electron Stimulated Reaction, e.g. cracking of hydrocarbons) and radiation damage.

Damage:
- Desorption
- Knock-on displacements up to core and valence excitations
- Athermal diffusion, does not appear in diffusion data or phase diagrams

Examples:

Knock-on even at around 200kV in Au, < 100kV in oxides. Damage in NiO [*K. Merkle*] was exceedingly high so that dislocation cores were amorphized. Reactions with C and CO may be responsible for this. (Don't forget ion damage. Ions created in the cathode space can be accelerated and may reach the specimen on very odd paths [*W. Mader*].

Loop formation in Al alloys is very fast. Radiation damage heals out in metals (in Al at ~200°C). In fcc metals such as Au, Ag and Cu stacking fault tetrahedra are frequently created at the lower foil surface. The natural oxide scale sometimes keeps the atoms in the foil. In TiC, Ti is

lost because of higher volatility. The Ti is shuffled via the vacancy formation mechanism down to the lower foil surface.

In conclusion, radiation damage cannot be avoided and one has to live with it. Radiation damage has many facets and it should be discussed only for a special material. It is very important to know if damage has already influenced the image or the spectrum, which can be tested by comparison of images/spectra after short and long time electron exposure. There is very little quantitative information available (temperature, contamination, vacuum, ...).

Irrational Planes between Growth Lamellae (*G. Shiflet*)

A frequent observation in lamellar structures is that despite a well developed and low index crystallographic orientation relationship, the interfaces are parallel to irrational planes.

Examples:

Growth lamellae in Cu_4Ti are nicely faceted at higher temperatures but smooth out at lower temperatures.

Irrational interfaces in eutectoid pearlite structures. Defects at irrational interfaces could not be characterized by CTEM. Lattice images show steps at low indexed habit planes. Cementite laid down on steps in eutectoid Fe-C with V which calls for further HREM work: determination of habit planes, characterization of steps and associated defects using edge-on microscopy,...

(*G. Thomas*:) Faceting of precipitates may be strongly influenced by impurities.

Characterization of DSC Dislocations (*C. Forwood*)

DSC dislocations at interface between Cr precipitates and Cu matrix were imaged under well defined conditions and image simulations of the diffraction contrast using the Head-Humble method were performed for different models. The Frank-Bilby equation was applied leading to a perfect agreement between experiment and analysis. However, there exists still ambiguity between two choices of the model.

Stand-Off Position of Misfit Dislocations (*W. Mader*)

Lattice images of periodic misfit dislocations at interfaces between Nb and Al_2O_3 precipitates clearly show that the dislocations are located in the metal 3-4 lattice plane spacings from the interface (stand-off position). This observation can be qualitatively explained by the balance of image forces acting on the dislocation at interfaces between phases with different elastic constants (repelling force for this material combination) and adhesive forces causing a lattice strain which attracts the dislocation. However, the calculated value of the stand-off distance does not agree with the observed distance, and other explanations such as that the observed configuration is not in equilibrium may also be possible.

Irrational Twins (*S. Ranganathan*)

In Al-Mn-Si with bcc lattice, twins occur which can be produced by a 72° rotation of the lattices around $[\tau 10]$ where τ results from the "golden cut" (so-called golden twins). The CSL lattice, however, would have a density of coincidence sites of $\Sigma^{-1} = 0$, i.e. $\Sigma=\infty$. The total diffraction pattern forms an icosahedral motif. All 5 twin variants together form orientations with 5-fold, 3-fold, and 2-fold symmetry as observed in quasicrystals. The interfaces can be constructed by structural units stacked in a non-periodic (irrational) way. The twin boundaries are,

however, heavily curved and may be difficult to investigate using HREM. [Theory by Bendersky, Cahn and Gratias (1989)].

DYNAMIC PHENOMENA

(C. Allen)

Chair: D. Howitt
Panel: H. Ichinose, F. Schapink, K. H. Westmacott

Dynamic Phenomena

This session included presentations and discussion of a variety of interfacial phenomena observed in situ, involving interface structure and migration. There are in fact a rather large number of phenomena involving changes in structure and/or position of interfaces which can be studied, at least in an exploratory manner, when specialized specimen holders for heating, cooling, straining etc. are employed in CTEM and HVEM and in an increasing number of instances in HREM as well. The presentations in this session included a variety of observations of dynamic phenomena, mostly occurring at elevated temperatures, both at conventional and high spatial resolution.

In situ observations in weak beam HVEM of the precipitation and dissolution of hcp γ phase in the terminal Al-rich solid solution with changes in temperature reveal the essential features of double kink nucleation and intersection. In this instance, the observed 3–4 nm high kinks are evidently collections of atom high kinks. Kink nucleation and motion in relation to degree of supersaturation may be described as the one dimensional analog of the situation in ledge migration. At large supersaturations the interface advances essentially at random; at lower supersaturation, ledges are present which exhibit a fluid-like motion without obvious involvement of kinks and at sufficiently low supersaturation, nucleation and migration of kinks are responsible for ledge advance. The barriers to kink nucleation which exist in this last regime for precipitate growth are probably absent for precipitate dissolution.

Other observations in CTEM employing in situ specimen heating which were presented for discussion included the disappearance of dislocation contrast "just below" the melting temperature of Sn, which may be evidence for grain boundary melting, possibly due to solute segregation in the boundary. A related situation was described for $AuCu_3$ in which disordering at both symmetric and asymmetric twin boundaries was observed in CTEM to occur 15-20°C below the bulk disordering temperature of the alloy. While disordering in bulk $AuCu_3$ exhibits first order kinetics, it is possible that at these interfaces disordering is second order as it is at free surfaces. Again the question of local composition may be raised as well.

The faceting/defaceting transformations of Ge in Al with temperature cycling result in reversible shape transformation between octahedron and sphere although the initial Ge precipitate is tetrahedral. The faceting/roughening transformation occurs at constant volume and within about 5°C.

In HREM the crystallization of Si has been very instructive by demonstrating that high resolution imaging can be maintained over long periods of time at elevated temperatures. For whatever reason the ledge mechanism of migration of the crystal/glass interface was not observed even for very slowly migrating interfaces. In such studies interface migration rates of the order of 0.1–0.2 nm/s are very convenient, if one can exercise some choice in the matter. In polycrystalline Au details of grain boundary structural changes can also be followed at elevated temperatures, which are associated, for example, with relative crystal rotation or with nucleation of a new boundary.

In every instance in which dynamic observations of thin foils are employed to study a solid state phenomenon of interest for bulk material, several questions must be asked routinely. In addition to those regarding the technique of observation itself and artifacts deriving therefrom, many of which have been discussed in this Workshop in connection with high spatial resolution imaging, there are questions of possible thin foil effects, of electron beam effects of heating, charging or displacement damage during the observation and of control and accurate measurement of environmental parameters such as the temperature of a heating holder. High resolution imaging at elevated temperatures demonstrates that heating holders can be designed and constructed which do exhibit both high mechanical and thermal stability while retaining double tilting capability. Unfortunately the same cannot be said up to now for cooling holders. The accurate inference of specimen temperature is certainly a perpetual problem which is exacerbated for specimens having poor thermal conductivity or inferior physical contact with the specimen holder. But it is really the inadequate stability and control of specimen holders employed in dynamic experiments which limits the present capability for dynamic observations at high resolution to a rather small number of experiments in a few places.

Several clear guidelines can be given for judging both the qualitative and quantitative validity of dynamic TEM experiments in general for the case in which bulk phenomena are of interest. Cross-checks with known bulk behavior must be made wherever possible, such as the net activation energy for a particular phenomenon determined *in situ* and *ex situ*, and, for example, the known nucleation sites for reactions such as at grain or interphase boundaries. In foil specimens severity of thin foil effects may also be assessed by comparison of kinetic details of a phenomenon in thin and thick regions of specimen. Other thin foil effects which may be troublesome for dynamic experiments include interface grooving at the free surfaces and the effects of surface contamination, including native oxides. For some phenomena such as diffusion-induced grain boundary migration, the influence of the surface morphology can totally dominate the process in thin specimens.

Thin film effects are the essence of studies of thin films, of course, so that in such TEM experiments, the principal concerns are the various possible effects of interaction of the film with the microscope environment, including the electron beam and the atmosphere or lack thereof. In thin film or thin foil specimens electron beam effects can be particularly serious, but can be sorted out provided good electron dosimetry can be performed in the microscope so that the effects of interest may be observed under identical local electron fluxes but different total energy deposition. Below the threshold for displacement damage (Frenkel pair production and resultant accelerated diffusion, for example), effects of temperature due to beam heating may be assessed by varying spot size at constant maximum flux. In general, atom displacement cross-sections increase with electron accelerating voltage so that amount of damage increases while heating effects are diminished for comparable electron flux and a fixed spot size. Ionization-related damage, in insulating ceramics for example, decreases as electron energy increases, all other things being equal. It is therefore of some importance that reliable electron dosimetry at the viewing chamber level of the TEM be available much more routinely than is presently the case.

The actual temperature in the region of observation may be in significant error with respect to the nominal indicated temperature. Although tedious, when absolute local temperature must be

known, it may be possible to establish the actual temperature by sandwiching the specimen to be studied with material of well documented thermal behavior, such as the crystallization of self-ion-implanted Si, and preparing a cross-section composite specimen with the calibrating material in the same area of view as the material of the experiment. Such absolute accuracy is usually not necessary though for even fairly reliable activation energy determinations in which accurate indication of temperature changes are more important than absolute temperature itself.

The satisfactory recording of dynamic phenomena is a common problem especially when events of interest occur at high rates. With the emergence of CCD systems with improved dynamic range and pixel size, one possible way of extending recording capability is to compromise image resolution in favor of recording speed, which is not possible with conventional video cameras.

INTERFACE PROBLEMS AND CRITICAL EXPERIMENTS

(D. Howitt)

Chair: K. H. Westmacott
Panel: A. King, T. Mitchell, G. Olson, S. Ranganathan, V. Vitek

This session was divided into two sections: the first addressed three specific problem areas that had been discussed earlier in the workshop but had not been resolved. The second section was devoted to a discussion of fundamental and technological problems and the experiments that might be used to solve them.

The first topic to be discussed was introduced by D. Howitt who presented the conclusions of the group which considered the implications of **radiation damage**. The importance of keeping the effect to a minimum by reducing the specimen exposure was well recognized as was the importance of working at accelerating voltages below those which will produce displacement damage. The discussion did not extend to radiation sensitive materials such as polymers or ionic solids where the perfect lattice visibly decomposes in the beam. D. Cherns raised the point that subthreshold effects such as those that he had observed in gold could be significant and C. Hetherington showed some micrographs of beam damage during a high resolution study of boundaries in silicon at 800keV. C. Allen commented that an exposure of 10^{19} electrons cm^{-2} would produce one displacement per atom in the sample so that conceivably half of the atomic columns could consist of vacant sites after the first micrograph was recorded. R. Kilaas pointed out that unless the damage is arranged periodically, the modification would not be resolved and so even in rather extreme cases, it may be possible to determine the structure of an interface. The broad conclusion was reached that duplicate images, which enable the extent of contrast modifications from beam effects to be deduced, are extremely valuable and should be recorded routinely. There are obvious benefits to cooling the samples, especially to liquid helium temperatures when interstitial diffusion can be halted, but at very high resolution, instability problems may overwhelm the advantages. No consensus as to the benefit of heating or cooling the specimens to enhance recombination was reached but it was pointed out by K. Westmacott that the former could probably produce the reorientation of an interface in less time than a direct radiation effect.

No formal report was presented on the second topic of **quasi-periodic boundaries** but two comments were made. V. Vitek pointed out that, since these boundaries can be considered as a straightforward extension of an irrational situation, it is probably impossible to distinguish between a long period boundary and an irrational boundary in the electron microscope. A. King made a general observation that in the quantitative evaluation of boundary structures the closure of Burgers circuits and the separation of ledges are the important features that need to be determined.

G. Olson gave a synopsis of the conclusions of the third group who considered the problems of **interface roughness** but only after first proclaiming that no one should worry about it. He pointed out that for external surfaces the roughness is simply the extent of step content and that the facets are flat. However, the situation for internal surfaces is less clear because the interfaces are not necessarily sharp. Using an extension of the core width as a measure of the loss of sharpness, he demonstrated how there is a limit to the core width at which a step can be defined. It was also concluded that it is not sensible to use the microscope as a fine determinant of rational (periodic) or irrational (non-periodic) interfaces, although it is certainly often possible to determine the spacing and strain field of the defects associated with an interface. The importance of this type of quantification was emphasized.

R. Hull presented the first of the summaries addressing **future challenges** and stressed the need to correlate structural properties from HREM with other physical properties. This is vital if HREM is to make substantial contributions to physics and materials science and very little of this has been done. As an example, in the semiconductor industry where the structure of interfaces is of paramount importance, he knew of only two examples where interface structural measurements had been successfully correlated with optical or electronic properties: the work of Krivanek and Liliental-Weber on mobilities at the SiO_2/Si interface and the work described by T. Sands on phase formation at metal/GaAs interfaces. Quantitative techniques for determining, or even defining, interface roughness in terms of its frequency spectrum are very much in their infancy. He quoted an example of some work that he had done attempting to study the effect of interfacial roughness in InP/InGaAs/InP quantum wells upon quantum confinement and hence radiative recombination energies. His final point was to emphasize the tremendous advantage of HREM in structure determination: the ability to measure phase differences between diffracted beams. In x-ray diffraction tremendous computation and effort have to be made even to get the sign of a structure factor, and so HREM is clearly at an advantage here. He pointed out that a classic technique used in x-ray diffraction structure determination, that of essentially establishing the signs of structure factors relative to a known (or guessed) heavy atom position, could be of enormous advantage in electron diffraction. Here heavy atom positions can often be unambiguously located using HREM and subsequent structure refinement using classic x-ray techniques such as Fourier differences maps, could be very much enhanced. (M. Gibson has applied some of these techniques to the Si/SiO_2 interface via electron diffraction.)

A. King, in discussing the topic of **grain boundaries**, specifically reinforced the concerns raised earlier about the control of experimental parameters such as thickness, beam tilt and sample orientation. The studies based on coincidence lattices were thought to be encouraging but the difficulties in the analysis of less perfect interfaces, such as in the silicon nitride/silicon carbide system discussed by T. Mitchell earlier in the week, have to be overcome. Low energy boundaries, which do not sustain a coincidence, present an extensive problem and the methodology and interpretation of multiple images orientated to the different sides of the boundary should be investigated. The study of this class of off-coincidence boundary and the transformations associated with them was seen as a major challenge. There was also the problem of how to distinguish the effects of segregation in a high resolution image, and although the solute will invariably be invisible, the effect upon the interface properties is certain to be reflected. It was also concluded that techniques other than HREM, e.g., FIM, are needed to distinguish an electronically clean interface.

T. Mitchell took an alternative tack. Confident that HREM was the answer, he set about how to determine the question. It may be straightforward to get a pretty picture but it is far more difficult to extract information. His point was that HREM is invariably the critical experiment and is highly specific. The interpretations always benefit from the information about the sample that has been accumulated beforehand. The importance of establishing the relationship between the structure and the physical properties has been exemplified in some of the work in thin film superconductors and semiconductor devices and should be more generally applied. The possibility of extending these methods via a more extensive application of dynamic HREM experiments, 3D reconstruction and quantitative microscopy was suggested and the caveat that specimen preparation still remains a problem closed the discussion.

S. Ranganathan outlined the **crystallographic aspects of interfaces** referencing the dichromatic patterns (α-fringe imaging), CSL and near-CSL analysis, hidden symmetry and quasiperiodic structures. He addressed defects in quasicrystals, pointing out the need for visibility criteria for dislocations in two and three dimensional structures as well as drawing attention to recent determinations of stacking faults in decagonal quasicrystals and some of the types of boundaries that have been observed.

The report presented by V. Vitek drew most of the discussion probably because it was the last presentation before the conference organizer closed the meeting. He pointed out that the description of metals is considerably simpler than ceramic or ceramic/metal systems. Calculations using empirical many-body potentials on a large scale are possible in metals but underdeveloped in these other systems. Mitchell commented that ionic materials are very well developed (HADES). Although full scale quantum mechanical calculations to determine cohesive energies can be done, they are a massive task and far more intensive computationally than the problem really requires. The need for closer interactions with the microscopist in the development of the interface models was stressed and it was pointed out how little progress had been made even in the study of metal alloys and intermetallics. The next direction for the modelling community was thought to be metal alloys since they can be fabricated within the framework of the current calculations. The refined potentials and success of this work would pave the way for the ceramic-based systems that need to be addressed in the future.

G. Thomas pointed out that in magnetic materials you could probably not get around the need for a complete quantum mechanical treatment and Y. Ishida reiterated that specimen artifacts from beam damage and specimen preparation had not been fully resolved.

V. Vitek added an additional comment that continuum methods of modelling boundaries should also be pursued (i.e., dislocation networks) and K. Merkle said that his group had successfully used such calculations to treat asymmetrical boundaries.

U. Dahmen made the final presentation demonstrating how it is possible to create multiple boundary structures by taking advantage of the different types of epitaxial growth that can occur on an orientated substrate. The advantages of this technique and the need for the engineering of boundary structures was the topic that brought the session to a close.

TEM TECHNIQUES

(S. Dregia)

Chair: R. Gronsky
Panel: J. Bentley, D. Cherns, C. Forwood, J. M. Gibson, W. Mader, M. A. O'Keefe

Overview (*R. Gronsky*)

Amplitude contrast techniques are useful for selective imaging to determine **g·R**, **g·b** and **g·b x u.** Weak beam dark field and high order bright field (HOBF) improve resolution by restrictive sampling; these techniques are especially useful for examining high defect densities, interface roughness and faceting. It was noted that HOBF should be used more frequently in the newer, high voltage microscopes (flat Ewald sphere). Amplitude contrast techniques are currently limited by specimen thickness, specimen life, control of specimen orientation, and signal/background ratio.

Implementation of phase contrast techniques requires image simulation and processing based on quantifying important specimen/instrument parameters: thickness, orientation, aberration coefficients, etc. The techniques also require control of the specimen environment and surface contamination, radiation dose, and mechanical and electronic drift. The most important limitation of these techniques is the control of signal/background ratio.

Interface studies in the TEM can also benefit from diffraction intensity measurements of the fine structure (rel rods) associated with the termination of structure.

Microanalysis is limited by probe size and broadening, especially at interfaces. Improved signals require use of higher voltages, but this must be balanced with risking radiation damage and decreased specimen life.

Convergent Beam Techniques (*D. Cherns*)

Plan View vs Cross Section

	Plan-view (CBED, diffraction contrast)	Cross-section (HREM, Fresnel contrast)
Suitable area	$1mm^2$	$10^{-10}mm^2$ (100Å x 100Å)
Surface relaxation	No	Yes
Sample preparation	Easy	Difficult
Atomic structure of the interface	Indirect (special cases)	Direct
Interface steps	Monatomic in favorable projection problem	Monatomic, 1-D,
cases, 2D		
Interface sharpness	Possibly	Yes
Defect structure	Good for large spacing	Good for small spacing
Strain measurement	Sensitive ($\sim10^{-4}$)	Sensitive ($\sim10^{-4}$)
reliable	less reliable	

Convergent Beam Diffraction

1. Rocking curve method (CBED, Large angle CBED, CBIM)

   This technique can be used to measure strains as small as 10^{-4}, determine the sign and magnitude of **b** for interface dislocations, detect monatomic steps, and in some cases, the technique can be used to determine rigid-body displacements at interfaces.

2. Bloch wave detail in HOLZ rings

   This is useful for studying interface structure in plan-view, e.g., Al/(001)GaAs. In cross-section, the technique yields very accurate microanalysis; e.g., in $Al_xGa_{1-x}As$, x can be determined to within ±0.05 with 20Å spatial resolution.

3. Zone axis critical voltage

   This is also very accurate for microanalysis; it has been applied to analyze various ternary semiconductors; e.g. $Cd_xHg_{1-x}Te$ to determine x to within ±0.03 with 20Å spatial resolution.

LACBED vs. CBIM

	LACBED	CBIM
Angle	6°	1°
Image resolution	50Å	3Å
Image contrast	good (small aperture)	poor (large aperture)

Future Developments

The application of FEG will improve the resolution of LACBED to 10-20Å, yet it will remain a low-dose technique because of the large area involved in the analysis. Current instruments will see further applications, particularly in the study of diffuse interfaces, rigid-body displacements, strain profiles, and small grain misorientations (~1°).

Microanalytical Techniques (*J. Bentley*)

Microanalytical techniques for studying chemical composition at/near interfaces include Atom Probe FIM, SIMS, EDS, EELS, PEELS, HREM, Z-contrast. The first two are complementary to the five TEM techniques. More FEGs will be used in future analytical TEMs, giving probe currents of 0.1-1 nA. The spatial resolution in EDS/(P)EELS is ~1nm; in EELS/HREM, it is limited by chromatic aberrations of the coupling lenses. The Z-contrast technique gives indirect information on composition. It is a powerful technique which can be applied in any TEM (resolution ~1nm) or in a STEM with a resolution of ~0.3nm.

Microanalysis is also applied in studying structure of crystalline and amorphous materials on a fine scale. Examples include CBED in crystalline materials (see above) and determination of radial distribution functions of amorphous materials (e.g. intergranular phases in ceramics) from diffracted intensities using parallel recording CCD.

High Resolution Techniques (*L. Marks*)

The concerns, uncertainties, and sources of error in quantifying/simulating HREM images are listed below with references.

Worries
- Precision of the envelope terms (O'Keefe, Bonevich and Marks, Ichizuka)
- Sampling/Cell Size (Wood et al., Bursill, Marks)

Uncertainties
- Scattering factors-covalent bonding (Cherns)
- Ionicity (Spence)
- Debye-Waller terms (Hull, Cherns, Penisson)
- Continuum in Howie-Whelan (Anstis)

Errors
- Cumulative numerical errors in multislice calculations increase with increasing thickness--use double precision (Lynch)
- HOLZ--solved in principle (Self and O'Keefe)
- Convergence through the specimen (thickness >100Å)(O'Keefe, Wood, Marks, Van Dyck)

  Unsolved in practice
  - Phonon inelastic scattering (Wang, Cowley)
  - Plasmons (Ahn and Krivanek, Wood, Saxton and Stobbs)

The application of statistical methods, e.g. R-factor analysis, was recommended in favor of visual comparisons of calculated and simulated images.

R-factor Analysis--a first word

$$R = \frac{\sqrt{\sum_i (e_i - c_i)^2}}{\sum_i (e_i - s)}$$

s = parameters
e_i = normalized expt.
c_i = calculated
Report scaling, offset terms. (See LEED and x-ray literature).

Use Monte-Carlo techniques to determine the confidence levels. Calculate images with random variations in parameters such as defocus, tilt, astigmatism, atom positions, etc. Calculate the R-factor and determine the confidence level which is equivalent to the error bar of the measurements. See books on numerical techniques; e.g., "Numerical Recipes in Fortran (Pascal, etc.)."

High Resolution Techniques (*M. A. O'Keefe*)

It was noted that visual inspection is sensitive but qualitative and less objective than statistical evaluation. Standard "specimens" have been constructed (Cowley et al.) for testing simulation programs. There are no serious disagreements among various image simulation programs. NCEM programs are available and can be run on VAX stations.

Summary on "Rigid Body Shift" (*C. Hetherington)*

1. Definition

Displacement vector R = (R_1, R_2, R_3) with components R_1, R_2 in the plane of the interface, i.e. describing translations parallel to the interface, and R_3 perpendicular to the interface defining the crystal separation or the excess volume.

The components R_1 and R_2 can be defined for special grain boundaries such as symmetrical tilt boundaries and twist boundaries. They cannot be defined at incommensurate boundaries, i.e. at boundaries between crystals with incommensurate spacings parallel to the interface. Such boundaries may be grain boundaries [e.g. (110) plane of crystal I parallel to (100) plane of crystal II] or, in principle, all phase boundaries since the lattice spacings between two phases are never commensurate. R_1 and/or R_2 can be determined in the case of strained thin layers which match epitaxially perfect. In the case of semicoherent interfaces, positions of atoms at the interface in crystal I relative to the atoms in crystal II may be well-defined in regions between the dislocations (e.g. atomic rows of crystal I fit into the valleys of crystal II). This, however, is not the definition of a rigid body shift and should be named differently such as local matching of atoms at the interface. R_3 can always be measured.

The problem arose of choosing the origin from whence the translation vector is measured. This problem is no problem because the choice of the reference atoms is arbitrary. They have just to be named. All other choices of reference atoms can be related via lattice vectors or vectors between atoms in the perfect lattices.

2. Measurement

2.1. Fringe Methods. (i) the α-fringe method [see R. C. Pond and V. Vitek, Proc. Roy. Soc. (London) **B357,** 453 (1977)], is very precise for small shifts of ~0.01Å and images obtained near the Bragg condition are used for the analysis. (ii) The displacement fringes obtained at off-Bragg conditions (weak-beam) can be used for the measurement of larger shifts near 0.1Å (see K. Miyazawa and Y. Ishida, Ultramicroscopy **22**, 231 (1987).

The displacement fringe methods are very precise and are based on the use of common reflections for imaging. Therefore, they are restricted to the displacement analysis of coincidence site lattice boundaries. Displacements at more general grain boundaries or phase boundaries cannot be analyzed in this manner.

2.2. Lattice Imaging. Two components of the rigid body shift, one parallel (R_1) and one perpendicular (R_3) to the interface, can be measured at interfaces which are imaged edge-on using HREM. The precision is not as high as for the fringe methods and is on the order of 0.1 - 0.2Å. The method must include the comparison of experimentally observed images with calculated images, and the precision depends on the quality of this comparison. A quantitative comparison using, e.g. cross correlation functions will lead to a higher precision than a visual comparison such as the overlay of prints.

Additionally, the measurement depends on most of the parameters and quantities critical for lattice imaging: focus, crystal thickness, beam tilt, crystal tilt, microscope resolution (e.g., if dumbbells in Si are resolved or not) and eventually thin foil relaxation. A help in obtaining the sensitivity of the contrast to these parameters is the simulation of images with such misalignment included. The simulations should be performed on the actual specimen.

FUTURE DEVELOPMENTS IN INSTRUMENTATION

(E. Kvam)

Chair: G. Thomas
Panel: C. Allen, R. Gronsky, K. Krishnan, W. Mader

As chairman of the session, G. Thomas began his introduction by setting the historical perspective of the application of TEM to grain boundaries. Conventional EM imaging of grain boundary dislocations had been done in the 1950's and then developed in the 1970's to early (tilted-fringe) HREM imaging of grain boundaries. The first true HREM imaging of a grain boundary was the work of Krivanek, Isoda, and Kobayashi (1977), in which the 5- and 7-fold rings at $\Sigma = 9$ tilt boundary in Ge were clearly resolved. The latest studies have progressed further, a good example being the recent work of Dahmen, et al., on the $\Sigma = 99$ grain boundary in Al. Thomas pointed out that current resolution is about 1.7 Å, and posed the question, "Should we go further?"

Continuing on this theme, he discussed the possibilities and requirements for reaching a 1Å interpretation limit. The first, of course, was to begin with very thin materials, but a large number of improvements were necessary in instrumentation. One primary requirement would be variable accelerating voltage to minimize radiation damage problems. To emphasize this need, Thomas pointed out the substantial time already spent during the workshop discussing the problems and causes of radiation damage, particularly in ceramics, which constitute a large portion of the materials for which better resolution is highly desirable. Possible improvements of the three standard parameters of the microscope are currently limited: λ cannot be reduced except by increasing E_V, and further exacerbating the problem of radiation damage; C_C and C_S improvements are limited because the available lenses are at magnetic saturation. (M. Kersker later noted that an improvement in magnetic materials was needed for better lenses, so that this was an opportunity for some of the participants to help themselves.)

Something to focus on was the information which can be extracted from a modern TEM viz., structure, composition, taking note of the possible effect of the microscope environment. To this end, some attention needs to be paid to accessories, such as stages for in-situ work, and for analytical acquisition; on the latter point, EELS is probably the only practical approach, and is itself somewhat limited. Controlled environments are also desirable for the objective chamber, i.e. other than UHV, so as to prevent compositional changes.

As an example of the imaging information currently available, the work of Epicier on mullite was shown. By using the ARM's stable, high-tilt stage, the [012], [001], and [103] poles from a single grain could all be imaged at 1.7Å point resolution in HREM. To illustrate the possibilities from further improvements in resolution and in order to demonstrate requirements for anionic resolution, six simulations of one of these poles were then shown. In each of the six figures, with C_S-limited resolution ranging from 3.8 to 1.2 Å, a distinct change in image was clear with 1.2Å needed to detect oxygen columns. Another example of problems typically faced by materials scientists is that of glassy interfaces, but these are compounded by the projection problem and the need for extremely thin materials: to analyze the structure of a glassy phase, the thickness must be only 2-3 molecular layers thick, beyond which information has become too convoluted due to overlap.

G. Thomas concluded by noting the cost problems of any advance in EM resolution (see figure 2), and emphasizing the need for national and (alluding particularly to the Brite-Euram-project : see van Tendeloo, below) international cooperation. One such possibility was multi-agency support. The newly proposed AARM at Berkeley could cost $10 to 20 million; however, could the U.S. afford to "miss the boat" as they had when RCA, the last domestic manufacturer, had left the business in the 1960's?

Support of AARM requires expert computing facilities. Also experimental methods for improving specimens, measuring thickness etc. still need much attention.

Magnetic and Analytical Instrumentation (*K. Krishnan*)

K. Krishnan gave a lucid overview of the needs in analytical instrumentation, beginning with a short summary of how LBL has proposed to address the needs in interface studies, and examples of specific applications. These included (1) composition and microanalysis of compound semiconductor contacts (e.g., metal or Ge on GaAs), of ceramics (grain boundary phases), and of magnetic materials (Cu segregation to enhance Fe/Nd alloys); (2) bonding and electronic structure, in semiconductors (electronic changes at interfaces and defects) and metal/ceramic contacts (electronics of interface bonding); (3) structure (interface strains and displacements); and (4) magnetic structures (e.g., domain structure at or below the 10 nm scale).

On the last of these points, some progress is already being made with the development of the differential phase contrast imaging microscope technique at IBM Almaden Research Center (see figure 3). The instrument is a modified version of the Vacuum Generators HB501 STEM, in which the standard detector has been replaced by a quadrant detector. The essence of its magnetic domain imaging capability is the same effect as that of Lorentz microscopy, i.e., that electrons will be deflected by a magnetic field. Unlike Lorentz microscopy, however, the deflection is directly manifested in a form which can be detected in standard imaging modes, due to the configuration for STEM imaging in the HB501, and the unique design of the quadrant detector with its associated software.

When the beam passes through the specimen (which is in a zero-field region), it is deflected by the local magnetic field. As the beam is scanned, crossing a domain, the field and deflection are reversed (provided the beam center impinges on the detector center), the current at the detector is inverted, and this effect is easily visible; in fact, it has been shown that the core structure of domain walls can be deconvoluted by proper handling of the information from the four detector sectors, and complete field maps can be drawn (automatically) at about 0.1 to 0.2 micron resolution.

K. Krishnan then went on to discuss the needs for, and capabilities of, specific improvements currently being made for analytical instrumentation. Some of these areas, with example applications, are laid out in figure 4. Some specific needs which were identified were a high intensity source (1 nAmp/nm^2), preferably cold (0.2 eV energy spread); a fine-probe system (0.5 nm diameter beam); very stable stages (0.1 nm/min specimen drift); good vacuum (<10^{-9}torr); large collection angles in EELS to yield high energy resolution from highly spatially confined beams (8 to 10 milliradians at 100 keV); low-field designs for examination of magnetic materials (leakage flux of only 0.4 mTesla); the need to reduce or eliminate effects of radiation damage.

Argonne *in-situ* HVEM (*C. Allen*)

C. Allen then presented the design for the proposed (nominal 2 MeV) tandem facility at Argonne National Laboratory. The proposed facility would be similar in some ways to the current HVEM, which incorporates two ion accelerators and allows simultaneous imaging and ion bombardment of a specimen. Further modifications include the possibility of linking a synchrotron

directly to the EM, and of incorporating sizable "experimental modules" within a very large gap pole piece.

A simple cut-away of such a module (still under design) is shown in figure 5. Since the dimensions would be available to users, a user could either bring devices for insertion to a module, or design an experiment-specific module to put into the pole piece. The large size of the module would allow a great variety of *in-situ* experiments to be performed, in conjunction with the use of a substantial variety of stages. Argonne has dubbed the concept "Microlab." The large working area would allow evaporations or high-pressure environmental cell use, as two examples.

It is unlikely that the ultimate design would actually incorporate a 2 MeV accelerator, partially because no manufacturer seems interested in building such a machine, and partly because essentially no applications would actually use such a voltage for imaging: nearly all current use is done below 1MeV, the highest available voltage being used to induce radiation effects, with actual imaging work done at a lower voltage.

HR/HVEM in Japan *(Y. Ishida)*

Y. Ishida gave a brief overview of the state of new HREM and HVEM instruments in Japan, summarized below. (He pointed out that most of these projects had been initiated by retiring faculty members; G. Thomas inquired as to whether there was a message in this.) He went on to state that international cooperative projects were possible, and encouraged. Interested parties were told to contact University professors or relevant industrial Principal Researchers, who could forward proposals to the Science and Technology Agency (STA), whereby a large number of researchers and funding may be made available. Best time would be in April.

New HVEM Instruments in Japan

Kyoto University	1MeV	Organic Materials	JEOL
Tohoku University	1MeV	High Resolution	JEOL
NRIM (Tsukuba)	1MeV	High Resolution	Hitachi
Tokyo University	1.25 MeV	High Resolution	JEOL
		Imaging Plate	
		PEELS	
		High T. Stage (Cryo. Stage)	
Osaka University	3MeV		Hitachi

Stuttgart ARM *(W. Mader)*

W. Mader gave the current status of the ARM to be installed soon (12/91) in Stuttgart. The JEM 1250 ARM (1.25 MeV) will utilize a separate generator and accelerator for control of HT instability problems. It will work at variable voltage, and allow $\pm 40°$ specimen tilt in two directions. A specimen prebake chamber will be attached. The machine will actually be dual-purpose, with separate lens sections for top- and side-entry stages.

For the top-entry lens, C_S should be 1.6 mm at 1250 keV, giving 0.10 nm theoretical (0.12 nm guaranteed) resolution, or at 400 keV a C_S of 1.0 mm for 0.17 (0.18) nm resolution. For the side-entry lens system, C_S at 1250 keV should be 2.75 mm, giving 0.12 (0.15) nm resolution, with C_S and resolution at 600 keV of 3.3 mm, and 0.23 (0.25) nm. Some other salient features of the system will be in-situ specimen heating capability (to 600°C for top-entry, to 800 for side-entry), and parallel EELS collection capability at resolution of 3 eV or better. The side entry lens would also incorporate, for example, an ion port; the concept, like the Argonne proposal, is to have an *in-situ* laboratory as well as premier resolution.

W. Mader pointed out that one of the most pressing practical problems was that of defining the experimental resolution, i.e., how to test this? This point was not solved in the ensuing discussion.

We also emphasized how difficult it is to measure foil thickness and this is very important for image simulation and interpretation.

Berkeley ARM 1000 (*C.Hetherington*)

C. Hetherington gave a perspective on the current capabilities and limitations of the ARM 1000 at Lawrence Berkeley. Although this microscope does have a small edge in resolution over the JEOL 4000, its real advantage remains the capability to tilt $\pm 40°$ in both orthogonal directions. As an example, he showed high resolution micrographs of prismatic growth islands taken at right angles to each other, both along and perpendicular to the long direction of a single island.

The present limitation for resolution in the ARM is chromatic aberration, which may be taken as a "soft" limit, since the contrast transfer function slowly approaches zero (as opposed to the "hard" C_S limit, in which the c.t.f. crosses zero). Thus, the microscope has produced crossed fringes at 1.4 Å (in Au), because the {220} are so strong, thus difficult to completely damp out.

The limitations which are being dealt with now are primarily those of mechanical stability, such as acoustic transfer. One of the methods for handling this has been to turn off the turbo pumps after the specimen has been inserted into the lens.

Brite-Euram (*G. van Tendeloo*)

G. van Tendeloo presented the Brite-Euram project. The project is a European collaboration between three Universities (Antwerp, Delft, and Tübingen) and two industrial companies (Tietz and Philips). Over four years, the consortium will build four machines capable of 1Å resolution. Although full details were not given, some of the expected capabilities of the EMs were obvious from the assigned tasks of each group. These included development of the 300 keV EM with field-emission gun (Philips), holographic imaging (Tübingen), recording-processing-CCD capabilities (Tietz), and three-dimensional reconstruction from through-focal series, to get crystal potentials from separate phase and amplitude information (Antwerp). It was pointed out that this project is an excellent example of international funding cooperation to provide a major facility.

Proposed Berkeley AARM (*R. Gronsky*)

R. Gronsky presented the case for the Advanced Atomic Resolution Microscope (AARM) proposed by Lawrence Berkeley Laboratory. First discussed were the present limitations on high resolution.

The Berkeley ARM is limited to 1.6 Å point-to-point resolution by the chromatic aberration envelope function; this is caused by instabilities in the high voltage power supply. Less serious mechanical (acoustic) and electrical instabilities provide another barrier; this may be about 1.4 Å. In any of the current high-voltage HREMs (see figure 6), high energy electrons induce serious radiation damage for most specimens, inhibiting or preventing the accumulation of multiple images from the same area. Large-angle tilting of the specimen (which is essential for three-dimensional reconstruction from projected atomic potentials) comes at the expense of resolution, through the need for a larger lens bore.

Hybrid alternatives to high voltage for high resolution exist, incorporating image-plane, off-axis electron holography. These have been initially developed by Lichte (Tübingen) and Tonomura (Hitachi). In this way, one can record both the amplitude and phase of the image wave independently. If the holographic sampling periodicity is less than the resolution limit, it is possible to correct for aberrations and so to achieve the desired resolution. The use of field emission guns has allowed the necessary sampling periodicity, by providing fringes of sub-Angstrom spacing.

Present limitations of the technique are principally noise (both electronic and sample-induced), limited specimen region (because of the need to have a reference beam in the viewing area), and the very high radiation dose the FEG would impose on the specimen. The advantages, however, probably outweigh the concerns; the capability to capture both amplitude and phase information, and to correct C_S is very significant. Further, the system is easily convertible to conventional operation.

The proposed AARM would achieve high spatial resolution by a number of hardware and software attributes. These were enumerated: TEM, rather than STEM, imaging would be employed to take advantage of the greater research base and proven performance record; intermediate voltage operation (200-500 keV) would be employed for improved HT stability and reduced radiation damage; a field emission gun would be used for high spatial and temporal coherence of the electron source; electron holographic methods would be used, requiring developmental research in both hardware (off-axis, biprism methods) and software (on-axis, through-focus and off-axis, reconstruction methodologies); utilization of fast RISC-based processing of digitally recorded and rendered output; a number of other, more standard, attributes would be employed, including large-angle goniometry, ultra-high vacuum, acoustic shielding, vibration isolation, and design for mechanical stability.

In summary, the AARM would provide atomic resolution imaging capability beyond existing ARM limitations (HV instability and radiation), incorporate new design concepts, provide technological challenges, and advance the instrumentation forefront.

Finally, R. Gronsky suggested than an alternative name for the project would be the Advanced Atomic Analytical Apparatus (AAAA), thus providing the highest visibility in alphabetic budget listings.

IBM, Yorktown Heights (*W. Krakow*)

W. Krakow gave a perspective on instrumentation improvements in his laboratory at IBM, Yorktown Heights. A great deal of advantage has been gained by good image processing capability, as illustrated earlier in the workshop, and this will continue to be used with the delivery of a new machine. The new EM, a Hitachi UHV machine, is in the intermediate voltage range as well, and should also incorporate some in-situ capability.

Conclusion (*R. Hull*)

R. Hull concluded the session with a discussion of the importance of various factors in producing better high-resolution work. He started with the question, "What is the most limiting factor: point-to-point resolution?" No positive responses were made. He continued by discussing those experimental factors which could be addressed in parallel with improved resolution, and which could provide more data than instrumentation improvements alone.

These included crystal misalignment, which can be addressed by use of a very fine probe; the projection problem, which can be circumvented by large-angle tilting; radiation damage, which can at least be minimized by reducing accelerating voltage; and signal detection increase by use of

CCD's, etc. On-line corrections and aids can also be provided, e.g. noise reduction, automated stigmation and beam tilt corrections, and simulations for image comparison.

A number of things can be done independent of the specific EM, or pending the arrival of a new microscope. Noise reduction can be handled by carefully preparing and cleaning samples, and by image processing, including averaging over periodic translations. Data collection hardware can be improved by incorporation or further optimization of CCD or channel plate detector systems. On-line correction algorithms can be optimized. The effects of amorphous or disordered surface layers, and the effects of crystal tilt, can be better simulated. Vibrations from pumps, gauges, or the environment, could be damped or otherwise dealt with.

A final point was the use of a UHV dedicated machine. As highlighted by W. Krakow, this could reduce the substantial problem of noise, and minimize irradiation effects. However, the turn-around time for such a machine (2 days) makes it impractical in many situations.

ACKNOWLEDGEMENTS

This workshop owes its success to the active participation of all invitees and guests. The excellence of the overview lectures was of particular importance. I would like to thank the thirteen session reporters for their diligence in preparing the notes. The role of the session chairmen and the panelists in maintaining a lively discussion throughout the week is greatly appreciated.

I am grateful to he staff of the NCEM for helping with many aspects of the organization, in particular Ken Westmacott for many useful discussions that were essential in shaping the final program, John Turner for his intelligent and skilled handling of the sound and projection equipment, and most of all Madeline (M^2) Moore for operating the strings behind the curtain, for making everyone feel welcome and for her efficiency in dealing with all aspects of this workshop, some of which I did not even know existed.

Assistance with organizational issues was provided by M. Field.

Sponsorship of the Bay cruise and conference dinner by JEOL and refreshments by ISI contributed much to the social success of the meeting and are gratefully acknowledged.

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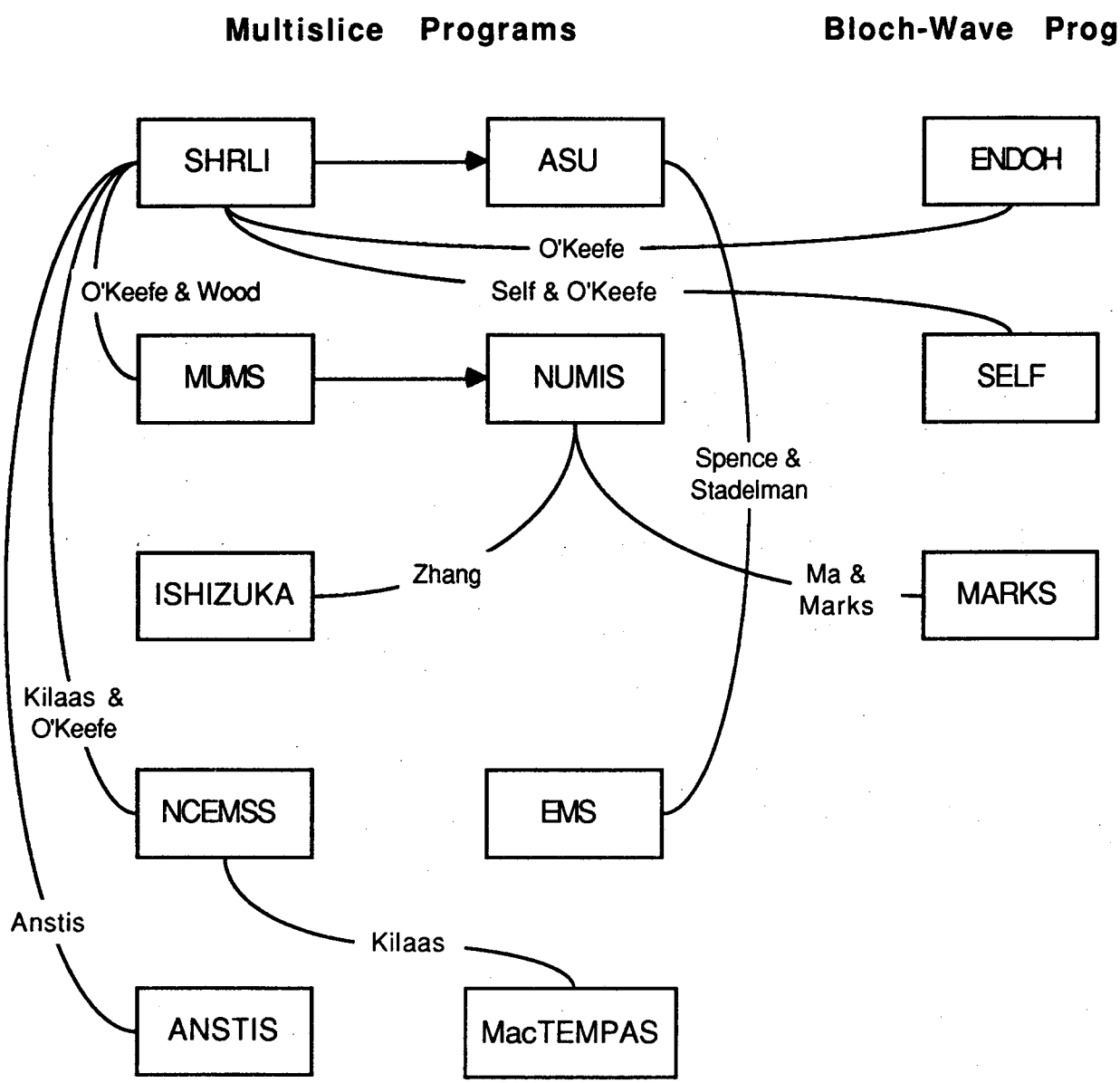

Inter-comparison of some image simulation programs

Figure 1. Image simulation programs and known comparisons, together with names of persons rumored to have carried out comparisons, both formal and informal. The ASU programs were derived from SHRLI, and NUMIS from MUMS.

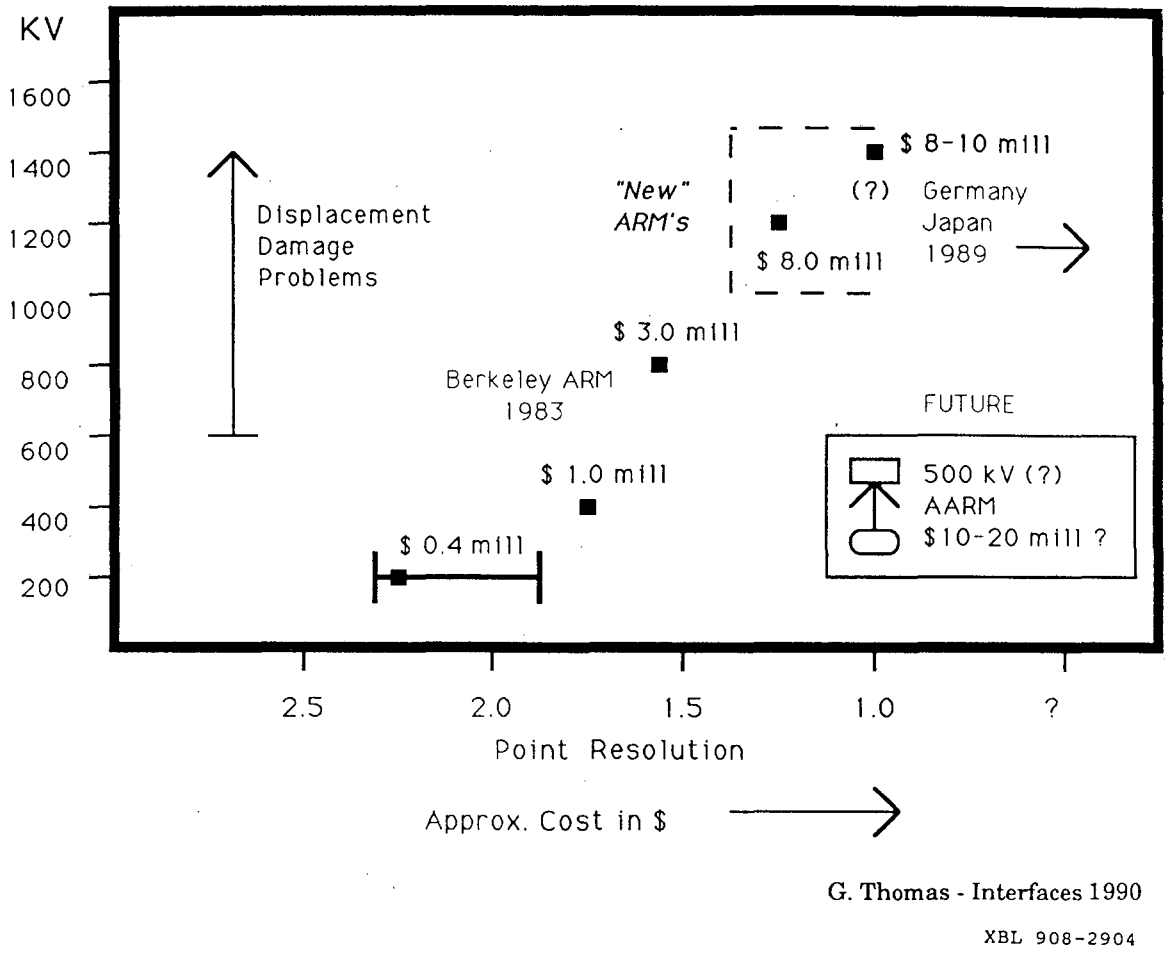

Figure 2. Approximate cost of new high resolution instruments.

Differential Phase Contrast Imaging

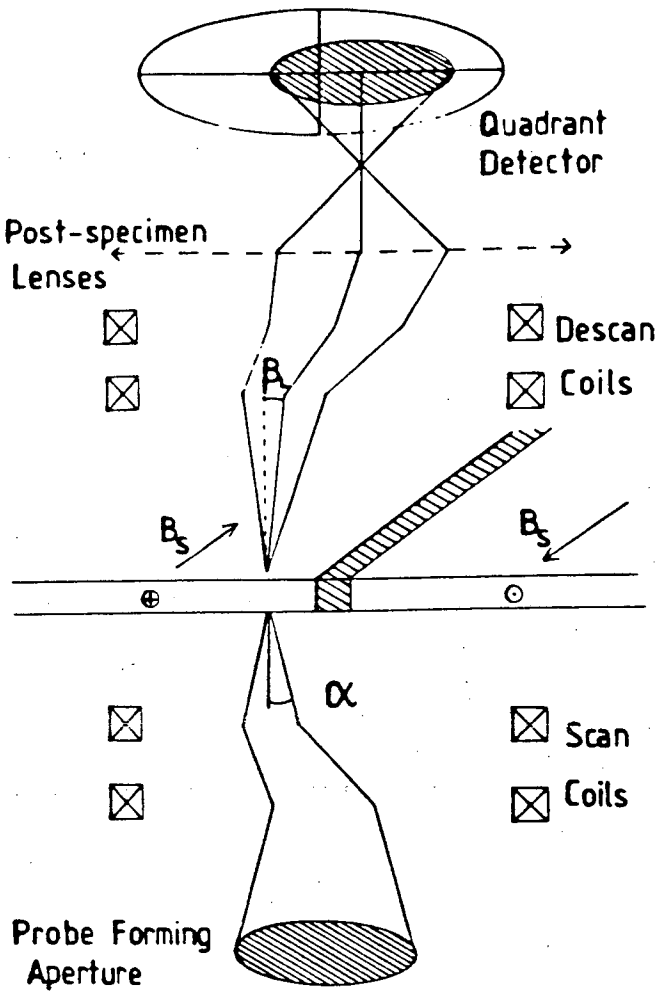

Fig. 1: Schematic representation of how magnetic contrast arises in the DPC imaging mode.

Requirements (ideal)

1. 10^{-4} -10^{-5} nAmps sensitivity

2. 1- 2 nm spatial resolution

3. Minimum Field in the specimen environment

Figure 3. Schematic ray diagram showing image formation in Differential Phase Contrast Imaging mode

Analytical Electron Microscopy and Related Spectroscopies

New Instrumentation

CRITERION	Current	New FEG UHV STEM	Applications	
			Technique	Applications / Advantages
Spatial Resolution (Usable Electron Probe)	100 A	<5 A	EDXS Microanalysis EELS Diffraction	Characterization of Interfaces, Grain Boundaries, Small Particles etc.
Gun Brightness	10^7 Amps /cm^2/sr	10^9 Amps /cm^2/sr	EDXS Microanalysis EXELFS	Minimum Detectable Mass Nearest Neighbour Distances Improved Signal intensity
Energy Resolution	1 - 2 V	< 0.5 V	ELNES	Electronic Structure, Bonding and Valence Information
Specimen Drift	3-5 nm/min	0.1 nm/min	EDXS Microanalysis EELS Diffraction	Spatial Resolution X-ray Maps Energy Filtered Imaging
Minimum Detectable Mass	1.0 wt%	0.1wt%	EDXS / EELS	Microanalysis
Vacuum	10^{-7} Torr	10^{-9} Torr	All Techniques	Contamination Clean Specimen Environment

Kannan M. Krishnan 7/89

Figure 4. Performance criteria for new analytical electron microscopes.

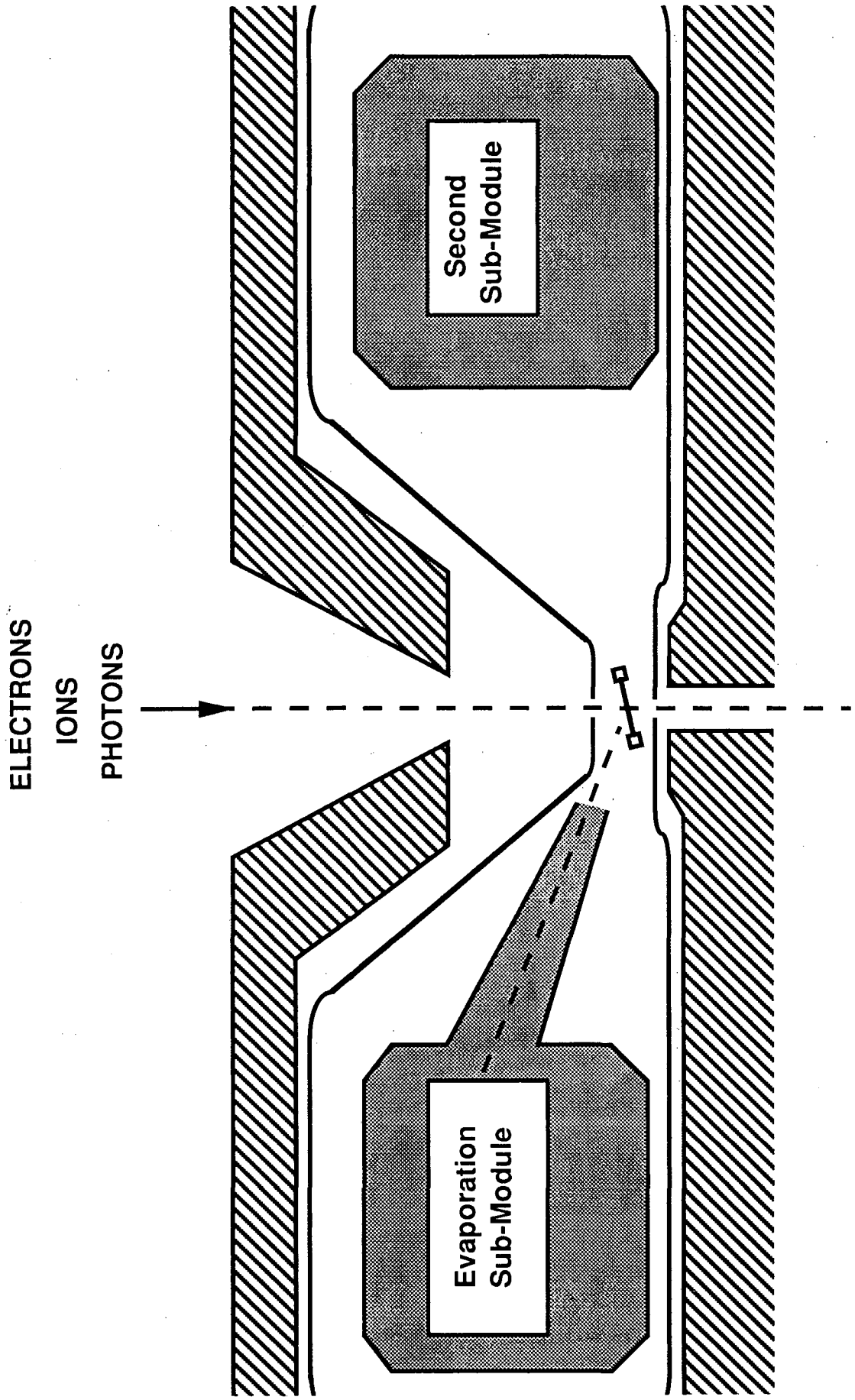

Figure 5. Schematic of modular design in Microlab concept.

High Voltage Alternatives

LBL (1982)	
Up bore	14.0 mm
Lo bore	4.0 mm
Gap	10.0 mm
Tilt	40.0 °
kV	1000.0 kV
Cs	2.3 mm
Cc	3.6 mm
Fo	5.6 mm
Th.Res	1.3 Å
G.Res	1.8 Å

Kyoto (1989)	
Up bore	8.8 mm
Lo bore	3.6 mm
Gap	8.0 mm
Tilt	40.0 °
kV	1000.0 kV
Cs	1.7 mm
Cc	3.6 mm
Fo	6.0 mm
Th.Res	1.2 Å
G.Res	1.4 Å

Stuttgart (1990)	
Up bore	8.8 mm
Lo bore	3.2 mm
Gap	7.8 mm
Tilt	40.0 °
kV	1250.0 kV
Cs	1.6 mm
Cc	4.5 mm
Fo	7.0 mm
Th.Res	1.0 Å
G.Res	1.3 Å

Also: 1250kV Hitachi instrument at Nagoya University...1.4Å resolution.

Figure 6. Characteristic feature comparison of high-voltage high-resolution microscopes.

APPENDIX I: PROGRAM

Monday, Aug. 20

1:30 Welcome

1:40 Overview

2:10 Lecture: *TEM of Interfaces*
Gibson

3:10 Lecture: *Crystallography of Interfaces*
King

4:10 Break

4:30 Discussion: *Interface Phenomena I*
Gibson, Gronsky
Hutchison, Ishida, Ponce

- bonding
- reconstruction
- roughening

5:45 Social

Tuesday, Aug. 21

8:00 Coffee

8:30 Lecture: *Interface Structure and Kinetics*
Olson

9:30 Lecture: *Atomistic Modeling*
Vitek

10:30 Break

10:50 Discussion: *Interface Phenomena II*
vanTendeloo, Hütten
Krishnan, Ranganathan, Thomas L

- segregation
- precipitation
- ordering
- dissociation

12:30 Lunch

1:45 Discussion: *HREM Quantification I*
Hull, Hetherington
Marks, Mills, Thibault

- rigid body shifts
- model/experiment comparison
- strain measurement
- specimen noise

3:30 Break

3:50 Discussion: *HREM Quantification II*
Sinclair, Howe
Gronsky, Krakow, Mitchell

- interface topography
- chemical diffuseness
- interface roughness
- projection problem

5:30 Social

Wednesday, Aug. 22

8:00 Coffee

8:30 Discussion: *Dynamic Phenomena*
Howitt, Allen
Ichinose, Schapink, Westmacott

- migration
- shape transformation
- in-situ TEM
- in-situ HREM

10:20 Break

10:40 Discussion: *Irrational Features*
King Mader
Forwood, Muddle, Ranganathan, Shiflet

- steps/ledges
- curved interfaces
- enclosed crystals
- dislocations

12:30 Picnic Lunch

Thursday, Aug. 23

8:00 Coffee

8:30 Discussion: *Interfaces in Materials I*
Cherns, Sands
Hull, Iijima, Liliental, Maher, Pirouz

- electronic materials
- thin films
- heteroepitaxy
- interfacial defects

10:20 Break

10:40 Discussion: *Interfaces in Materials II*
Mitchell, Krishnan
Howe, Mader, Mishra

- metals
- magnetic materials
- ceramics
- quasicrystals

12:30 Lunch

1:45 Discussion: *Interface Modeling*
Vitek, Mills
Bonnet, Dregia, Kilaas, Pirouz

- interface construction
- relaxation guidelines
- defect behavior

3:35 Break

3:55 Discussion: *Advances in Image Simulation and Analysis*
Pirouz, O'Keefe
Iijima, Penisson, Merkle, Anstis

- model/experiment comparison
- standards of simulation
- artifacts
- TFS maps/reconstruction

7:00 Banquet

Friday, Aug. 24

8:00 Coffee

8:30 Summary: *Interface Problems and Critical Experiments*

Westmacott, Howitt
King, Mitchell, Olson,
Ranganathan, Vitek

9:30 Summary: *TEM Techniques*

Gronsky, Dregia
Bentley, Cherns, Forwood, Gibson
Mader, O'Keefe

10:30 Break

10:50 Summary: *Future Developments in Instrumentation*

Thomas G, Kvam
Allen, Gronsky, Krishnan, Mader

- *Argonne HVEM*
- *Stuttgart HREM*
- *Magnetic Imaging Microscope*
- *AARM*

12:30 Concluding Remarks

Special topic "task forces"

Quantitative image/model comparison in HREM *(Gibson, Hetherington, Hull, W. King, Marks, O'Keefe)*,

Rigid body shift measurement *(Cherns, Hetherington, Hull, Mader, Vitek)*

Radiation damage *(Allen, Cherns, Howitt, Ichinose, L. Thomas, Westmacott)*

Interface roughness *(Dahmen, Gibson, A. King, Kvam, Olson)*

Temperature calibration for in-situ experiments *(Hull, Van Tendeloo, L. Thomas, Westmacott)*

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