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LATTICE IMAGE AND OPTICAL DIFFRACTION STUDIES OF ALLOYS

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INTRODUCTION

In order to understand fundamental aspects of materials, their examination should be performed at the highest resolution possible. For instance, phase transformations occur by the rearrangement of atoms. Lattice defects such as vacancies and interstitials, dislocations, grain boundaries, and the first stages of formation of new phases, occur spatially over only a few atomic distances. The research philosophy of this current program is to provide such basic knowledge by studying atomic arrangements in alloys directly. The lattice imaging technique of transmission electron microscopy (TEM) is capable of providing such information, which can then be complemented by diffraction data at an extremely localised level using optical diffraction methods. The present article is limited to a discussion of the progress our group has made in this area.

EXPERIMENTAL PROCEDURE

Figure 1 represents schematically our approach to the experiments. The parameters which affect the image formation process in the TEM are carefully considered. From the point of view of the specimen the most important of these are thickness and orientation. In the microscope they are the objective lens defocus and the electron beams selected by the objective aperture, as well as day-to-day considerations such as cleanliness and column alignment. Multi-beam dynamical electron diffraction calculations are performed, both to assess the optimum experimental conditions and to provide a basis for image interpretation. With experience the appropriate conditions may often be recognised during experimental observation, although generally we record through-focus series for comparison with computed image profiles. The results of such comparisons for ordered alloys have been described elsewhere (1,2), indicating how information may be derived at the atomic level about such materials.

We regard the lattice image as the most important source of data concerning the atomic lattice. However, this may be complemented in reciprocal space by taking laser optical diffraction patterns from the images in a conventional optical bench. To date, we have used these patterns in two ways (3). Firstly, as a check that the lattice image is a true representation of the lattice structure of the specimen. This is achieved by comparing the optical diffraction pattern from the optimal lattice image with the original electron diffraction pattern from the specimen. If the lattice image faithfully records the lattice structure of the material then the two should be essentially identical, for those beams used in the image formation process, and this has been found to be the case for ordered and spinodal alloys(3). Secondly, the optical method may also be used as a micro-diffraction technique in its own right. The lattice image is taken at high magnifications (usually 500,000 times in the present study) and thus readily available apertures used to select areas on the image for diffraction are extremely small at the specimen plane (e.g. 1 cm is 200Å and 1mm is 20Å, for 500,000 times magnification). The intermediate aperture size for conventional TEM selected area electron diffraction is limited by spherical aberration of the objective lens to about 2μ diameter at 100 kV (4). The aperture for optical diffraction is not restricted in this way. A practical minimum for alloy studies has been found to be 10Å diameter, limited by the Fraunhofer pattern of the aperture itself (3).

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The specimens for our work are preferably prepared by vapour deposition to the desired foil thickness and orientation. When accurate composition is required or when this method is not feasible, conventionally electropolished thin foils are used. Such work requires careful choice of thickness for the lattice images, for which thickness contours may be used as a guide. With experience the optimum images may be obtained as a matter of routine.

APPLICATIONS OF LATTICE IMAGING

The alloys from which images have been taken in our group during the present study are listed below in Table 1.

TABLE 1

1. Ordered Alloys
Cu_3Au, CuAu, Ni_4Mo, Au_4Cr, Au_3Cr, Fe_3Al, Mg_3Cd

2. Spinodal Alloys
Cu-Mn-Al, Cu-Ni-Cr, Au-Ni

3. Grain Boundary Reactions
Al-Zn

In every system studied new information has been obtained which has not been available from alternative techniques. Furthermore, this information is now at an atomic level so that our goal of studying fundamental phenomena is being achieved. An example from each of these categories serves to illustrate the stage we have reached.

1. Ordering in Mg_3Cd

During observation of ordering of Mg_3Cd with the high flux electron beam used for lattice imaging, an unusual reaction occurs, whereby the DO_{19} ordered hexagonal phase transforms to the orthorhombic B19 phase (5). The domain size (~100Å) and orientations of the latter are such that selected area electron diffraction patterns on the basal plane do not distinguish these phases. This applies also to optical diffraction patterns taken using an aperture corresponding to 400Å diameter at the specimen plane. However, by using an aperture smaller than the B19 domain size, viz. 40Å diameter, a diffraction distinction may be readily achieved: The DO_{19} phase still shows hexagonal symmetry whereas the pattern from a single B19 domain variants superpose so as to become identical with the DO_{19} pattern (6). This experiment serves to illustrate the powerful microdiffraction capability of the optical diffraction method.

Direct lattice imaging has revealed that unit cell high steps are present in domain interfaces and order-disorder boundaries in B19 Mg_3Cd. An example of such steps in a rotation domain boundary is shown in Fig. 2. This indicates that the atomic mechanism of ordering in this system occurs by a ledge mechanism. Such detail is not available in conventional micrographs. These observations are the first to show the presence of such small features at interfaces, clearly demonstrating the superiority of the lattice image for identifying alloy behaviour at the atomic level.

2. Spinodal Decomposition in Au-Ni

During spinodal decomposition solute segregation occurs by wavelike composition modulations in particular crystallographic directions, commonly <100> in alloys. As the interplanar spacing is a function of composition a corresponding d-spacing variation is also achieved. In the lattice image this is manifested by fringe spacing modulations, as recently demonstrated for Au-Ni (3).

An alternative method of processing the direct image data is to take a series of optical micro-diffractograms. If the aperture size is smaller than the fringe periodicity, a single diffracted spot will be produced whose position depends on the average fringe spacing enclosed in the aperture. As the aperture is moved about the image, the average spacing (related to the local composition) changes and this

will be reflected by the position of the diffracted spot. An example of a series of such micro-diffractograms is shown in Fig. 3. The aperture size corresponds to 20Å at the specimen plane (the modulation wavelength is 29Å in this case) and the aperture has been moved in 10Å steps in the <100> direction on the image. The variation of spot position is quite clear.

The micro-diffractograms in Fig. 3 are compared with an optical pattern taken using a 200Å aperture. In the latter the average diffraction spot is flanked by two satellite reflections the positions of which are related to the modulation periodicity. This larger-scale pattern is similar to the electron diffraction pattern from the specimen, confirming that the lattice image is truly representing the lattice structure of the alloy (3).

3. Grain Boundary Precipitation in Al-Zn

The experimental difficulties of lattice imaging are compounded when attempting to study grain boundary reactions. A simultaneous image is required not only in both crystals on either side of the boundary but also in any second phase particle present at the boundary. Although greater experimental expertise is necessary, we have found that such studies are indeed feasible.

Figure 4 shows the lattice image near a grain boundary in Al-Zn. In this particular example only one matrix grain and the precipitate are imaged. The coherent matrix-precipitate boundary has remained stationary whilst the precipitate has migrated into the grain with which it has no orientation relationship, showing classical behaviour. As the precipitate and matrix have different lattice spacings, the coherent boundary contains a dislocation network which is revealed by the image. However, the most important discovery of this study concerns the structure at the moving incoherent interface. All current discontinuous precipitation theories have assumed that this is a normal high angle grain boundary whereas lattice imaging has revealed the presence of a transition region between the two phases (7). Conventional microscopy has not resolved this feature, again showing the superiority of lattice imaging for information at the atomic level.

SUMMARY AND CONCLUSIONS

This paper has reviewed some of the continuing progress in our group in applying lattice imaging to problems in alloys at the atomic level. This has been illustrated by results on unit cell high steps in interfaces in ordered Mg_3Cd, on direct and reciprocal space studies of spinodal decomposition in Au-Ni and on grain boundary precipitation in Al-Zn. These observations, at present, are unique in the metallurgical field.

Direct images have been complemented by optical diffraction patterns, with the possiblity of using selecting apertures down to 10Å in diameter, which is a considerable reduction over conventional electron diffraction. Such apertures are also smaller than areas currently selected by the beam size in scanning transmission electron microscopy (8). The microdiffraction capability is being used to solve ambiguous, conventional diffraction patterns, such as those obtained from short-range ordered alloys, and for more complex patterns, as for instance obtained from ordered stacking structures in SiC (9). The optical patterns from larger areas are also compared with the original electron diffraction patterns to ensure that the lattice image contains the pertinent information of the specimen lattice.

It can be appreciated that lattice imaging is assuming an important role in studying fundamental aspects of materials science.

ACKNOWLEDGEMENTS

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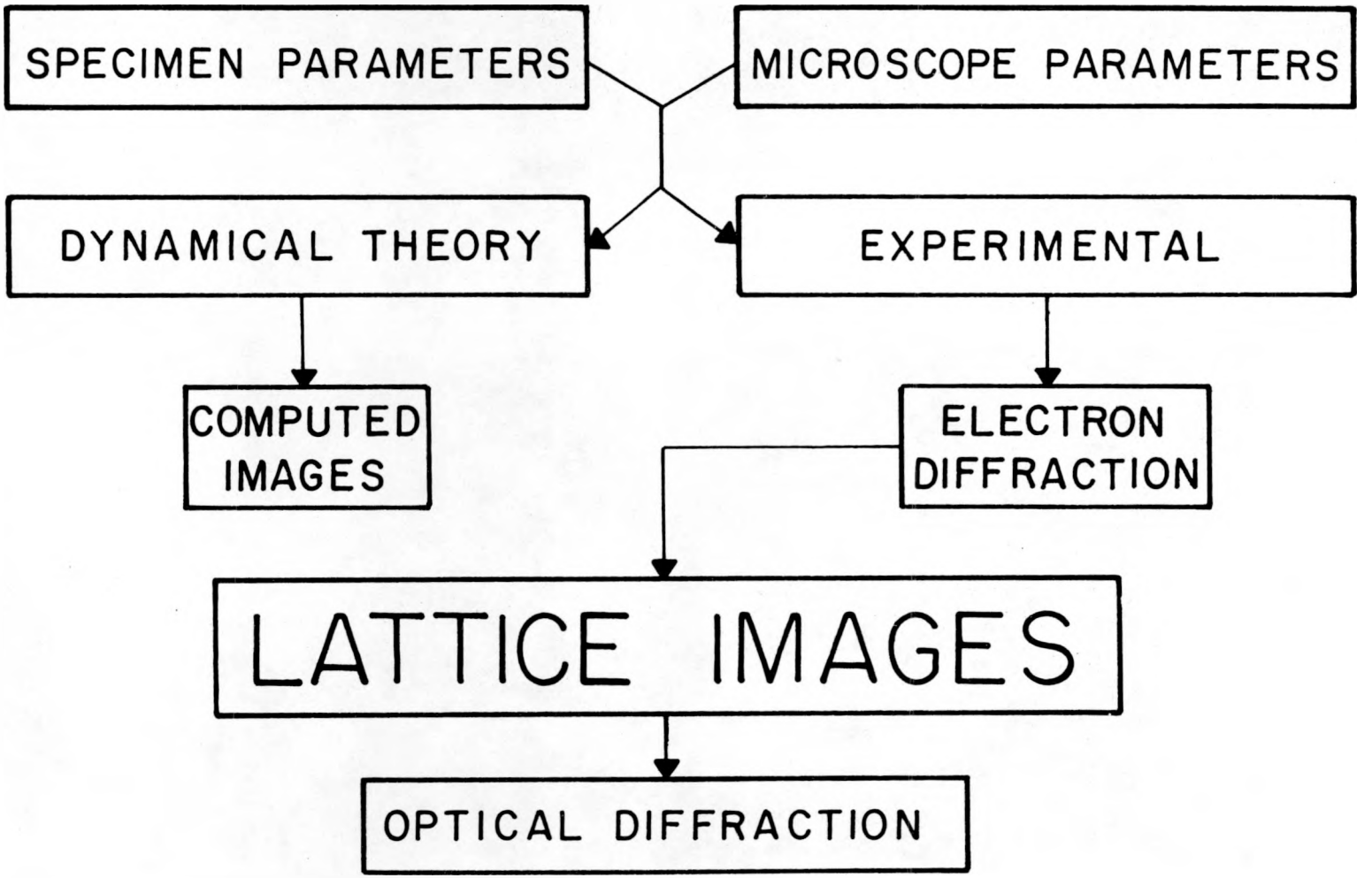
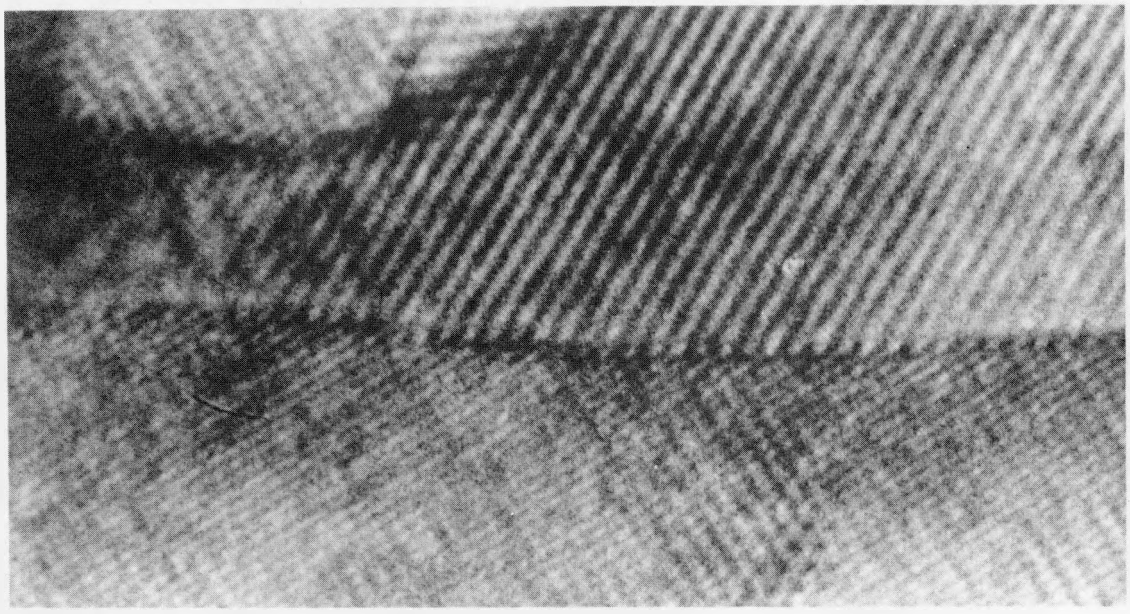


XBL 762-6470

Fig. 1

Schematic representation of lattice image experiments of the current program.



XBB 762-1115

Fig. 2

Lattice image of B19 ordered domains in Mg_3Cd illustrating the presence of unit cell high (5.3Å) steps in the interface

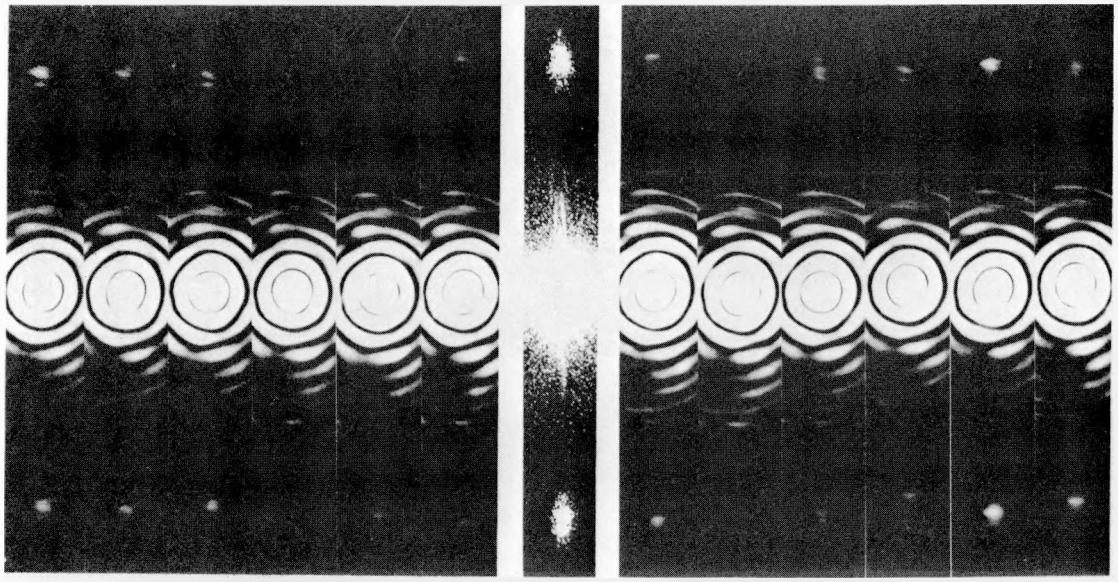


Fig. 3 XBB763-2668

Series of optical micro-diffractograms from a lattice image of spinodally decomposed Au-Ni, taken at 10Å intervals on the image.

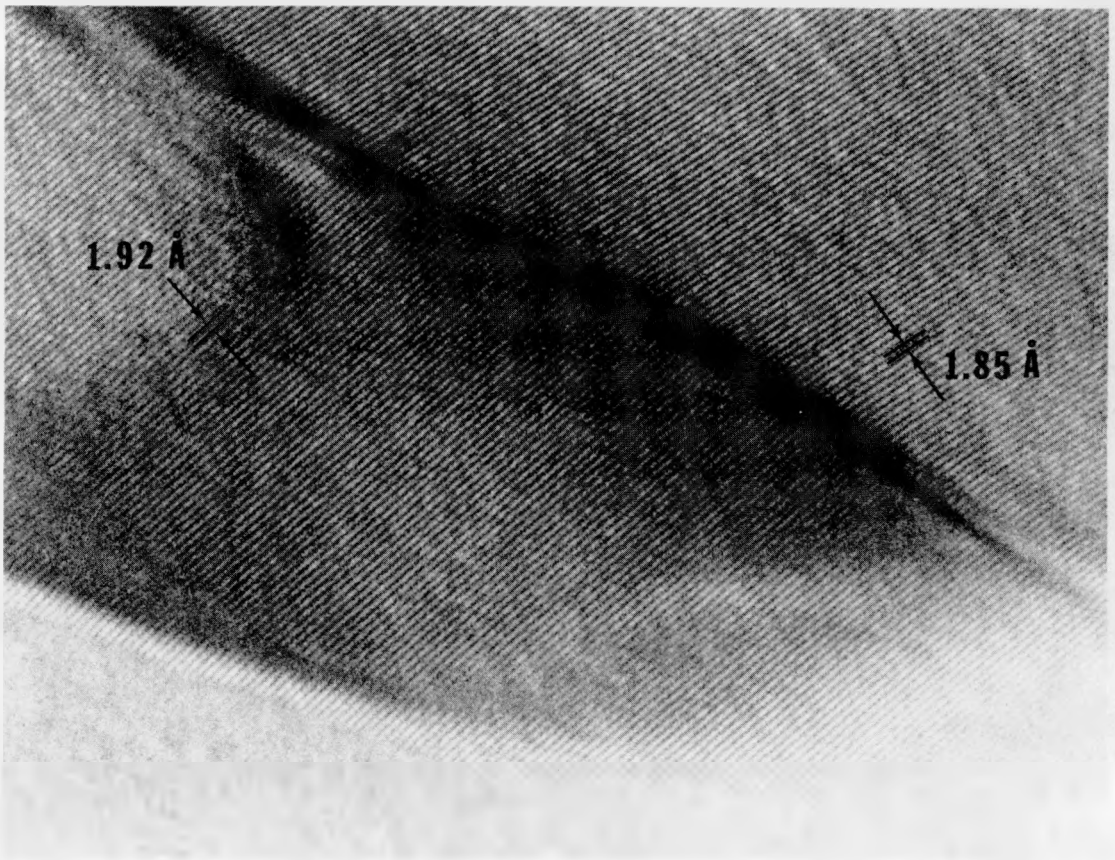


Fig. 4 XBB 761-602

Lattice image in a grain boundary region of Al-Zn, showing fringes in one matrix grain (M) and the grain boundary precipitate (P).

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