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R. Sinclair and G. Thomas

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DETERMINATION OF LOCAL COMPOSITION BY LATTICE IMAGING

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ABSTRACT

An assessment is made of the potential for direct lattice imaging to be used for the determination of local chemical composition in alloys by localized lattice parameter measurements. The principle involves correlation of electron microscopy lattice fringe image spacings with composition. It is found that in favorable circumstances (e.g., Fe-C, Al-Cu, Au-Ni) composition differences of the order of 3% can be detected with a resolution of 20 Å. In addition the results indicate that for many cases the method both has higher resolution and is more precise than currently available, alternative analytical techniques. Whilst the results are very encouraging, the role of electron optical and lattice constraint effects must be examined in more detail to allow general application of the method, especially for the case of multiphase systems involving large interfacial strains.

I. INTRODUCTION

In understanding alloy behavior at the atomic level the following basic information is needed:

- i) the local chemical composition;
- ii) variations in crystalline structure;
- iii) the role and configuration of lattice defects.

The direct lattice fringe imaging technique of transmission electron microscopy (TEM) is capable of providing such information at the atomic plane level in crystalline solids, as has been demonstrated, especially for the latter two, in studies of a variety of phase transformations [1-6]. The purpose of this article is to consider in detail the first of the above parameters. The advantages and limitations of the lattice imaging approach for composition determination are demonstrated and the potential of the technique in comparison with alternative methods is discussed.

It should be emphasized that the interest here is in the determination of localized rather than bulk composition. The possible applications are far-reaching (e.g. estimating localized compositional gradients near grain boundaries, interphase interfaces, etc.). Modern analytical techniques such as energy dispersive x-ray analysis and Auger electron spectroscopy suffer from relatively poor spatial resolution ($\sim$ several hundreds of $\text{\AA}$). Similarly diffraction data for sufficiently accurate lattice parameter measurements must also be collected from fairly large volumes (e.g. $\sim 10^{18}$ unit cells in x-ray and neutron diffraction, $\sim 10^{12}$ unit cells for selected area electron diffraction and

-3-

$\sim 10^3$ unit cells in scanning transmission electron microdiffraction methods).

This problem is one of the most intractable in materials characterization and one for which lattice imaging appears to be an exciting new development.

II. MEASUREMENTS OF INTERPLANAR SPACINGS

Lattice imaging is now established as a very powerful method for characterizing the physical structure of materials especially in cases where large localized strain effects (e.g. at dislocation lines) are not of principle concern. A main problem now is that of obtaining highly localized information about chemical content. The present basis for establishing composition using high resolution techniques lies in the interrelation of interplanar spacing (i.e., lattice parameter) with chemical composition (Vegard's law), which is considered in later sections. The certainty of the composition determination depends on the precision with which the lattice spacing can be measured, the latter being obtained either directly by processing measurements from the image or indirectly from their associated optical diffraction patterns [4].

Direct measurements can be made either on a print or by using a microdensitometer trace from the original electron microscope negative. The experimental uncertainty arises in defining the origin and end-point of the fringe sample (Fig. 1), which should be at an identical position for both fringes (e.g. the peak position is often the easiest to specify). The absolute error is the same for all measurements but clearly the relative error decreases with the number of fringes sampled. From our measurements on a variety of fringe images, the error in a single-fringe spacing determination is assessed to be within $\pm(5-10\%)$ depending on the quality of the image in the area of interest. Increasing the number of fringes over which the measurement is made proportionately decreases this error. Typical values are given in Table I. Highest precision is attained by using the greatest number of fringes but, by so doing, the localized scale of the information is sacrificed thus losing resolution. Depending on the circumstances a compromise must therefore be made between accuracy and resolution.

TABLE I.

<u>Precision of Fringe Spacing Determinations</u>					
<u>Number of fringes</u>	1	5	10	30	100
<u>Method</u>					
Direct Print	$\pm 5\%$	$\pm 1\%$	$\pm 0.5\%$	$\pm 0.2\%$	$\pm 0.05\%$
Microdensitometer Trace	$\pm 10\%$	$\pm 2\%$	$\pm 1\%$	$\pm 0.3\%$	$\pm 0.1\%$
Optical Diffraction	$\pm \infty$	$\pm 2\%$	$\pm 1.5\%$	$\pm 1\%$	$\pm 0.5\%$

The decision whether to use microdensitometer traces or the prints depends on the quality of the fringe profile. In our experience the eye is rather good at reproducing peak positions on a print and it is rare for an advantage to be gained by microdensitometry. This conclusion was also reached by Phillips [7]. However, it becomes useful in processing long sequences of measurements such as the spinodal analyses described in Section IV.2.

In the optical diffraction pattern from the lattice image, the position of the reflections is related to the reciprocal of the average fringe spacing contained in the selecting aperture. Using a laser optical system, this aperture can be as small as the equivalent of 10 Å diameter, limited by the Fraunhofer scattering of the aperture itself [4], so that very localized fringe spacing analyses can be carried out. In this case the error is due to difficulties in locating the maxima of the diffracted spots, the quality of the image tending to have less influence. For smaller apertures, the diffracted spots are broader thus increasing the uncertainty. A single fringe diffraction pattern, for instance, is

a continuous streak, for which the error is $\pm \infty$ (Table I). With small usable apertures an error similar to the direct print measurement is attained. However, as the aperture size increases perfectly sharp diffraction spots are often not obtained (e.g. Fig. 2) and it is difficult to increase the precision to be better than $\pm 0.5\%$.

Thus for $2\text{\AA}$ lattice fringes, in favorable circumstances a single spacing can be specified to $\pm 0.1\text{\AA}$. If averaged over $10\text{\AA}$, a 1% accuracy (i.e. $\pm 0.02\text{\AA}$) can be achieved and by increasing the distance to about $30\text{\AA}$, a respectable precision of $\pm 0.3\%$ can be obtained provided the image is of good quality. A typical working value is $\pm 0.5\%$ for $20\text{\AA}$ resolution. It may be realized that no other technique currently can give such high precision for highly localized interplanar spacing data.

Two further points of interest concern complementary utilization of the direct image or optical diffraction methods. Firstly, as the fringe spacing decreases, the fringe profile becomes more poorly defined and it is much more difficult to make accurate measurements on the image. Conversely, the spacing of the optical diffraction spots increases and now its relative error correspondingly decreases. Secondly, rather sharp diffraction spots can often be obtained from poor quality lattice images (e.g. Fig. 3). Thus for closely spaced or poorly defined fringes, the optical diffraction method may well provide superior fringe spacing data, whereas in large spacing materials the direct image is normally preferable. This point will be illustrated in Section IV.

III. COMPOSITION DETERMINATION

In order to establish a composition from fringe spacing measurements we must of course define the relationship between the two. The best situation would be the validity of the assumption that the bulk variation of lattice parameter with composition holds also for lattice fringes. For the purpose of demonstrating the capability of lattice imaging this will be accepted for the present. However, two important factors must also be taken into account: firstly, the effect of lattice constraints in changing localized d-spacings from those expected for a certain bulk composition; and secondly the correspondence between lattice fringes obtained in the electron microscope with the real lattice planes in the specimen (i.e. electron optical effects). These will be discussed in Section V.

Figure 4 shows schematic variations of lattice constant with composition ($\frac{da}{dc}$) for three categories of alloy systems with low, medium or high $\frac{da}{dc}$. For the low slope case (e.g. Al-Zn, spinodals such as Cu-Ni-Fe) it can be seen that high precision is necessary in order to obtain accurate compositions and that, in this situation, resolution must be sacrificed considerably. For instance, to detect a 10% change in composition, better than 0.1% accuracy is needed which requires a measurement over at least 50 fringes, typically 100 Å in alloy systems. Even this figure is still superior to the analytical techniques currently available. For 0.5% accuracy over 20 Å, large changes in composition can be detected and in some instances this is sufficient, as will be seen in Section IV.

For alloys with medium $\frac{da}{dc}$ (e.g. Au-Pt, Fe-V), the usefulness of the method becomes evident, with a 10% composition difference detectable with a 20 Å resolution. In systems with larger slopes (e.g. Al-Cu, Cu-Be, Au-Ni) lattice imaging is extremely powerful. Thus at 20 Å resolution, compositional changes can be detected down to ~ 3%. 1% differences may be established with 60 Å resolution, whereas a single fringe measurement can separate 30% compositional variations. These figures are the basis for allowing the composition profiles to be established at G.P. zones in Al-Cu by Phillips [7]. Strain effects are likely to be more important in these situations (see section V).

An extremely important class of alloys lies in this latter category, viz. the interstitial solid solutions, particularly C and N in steel. For example, nitrogen ferritic steel has a variation of 0.007 Å per atomic percent nitrogen [8] i.e., a slope ~ 23%. Thus it is feasible to extract extremely precise values of interstitial profiles by lattice imaging, with gross carbon and nitrogen segregations detectable at atomic plane level. We predict therefore that the technique will prove extremely useful in studies of steel for determining the distribution of atomic species, particularly the interstitials. This is of particular importance since these elements have low atomic numbers and are often difficult to analyse by standard spectroscopic analytical methods, e.g. X-rays. The prediction has recently been verified by Koo and Thomas [9] who imaged the (101) planes in ferrite and martensite simultaneously in duplex Fe-2% Si - 0.1% C steels. A difference in fringe spacing was observed which corresponded to the carbon concentration in martensite expected from the tie-line composition at the ($\alpha + \gamma$) aging temperature prior to quenching.

IV. EXAMPLES OF COMPOSITION DETERMINATION

IV. 1. Deviations from Stoichiometry at Stacking Faults.

It can be appreciated from the above considerations that for many purposes single fringe measurements are insufficiently precise for providing useful composition information. However, one area where it does have important implications concerns localized deviations from perfect crystalline periodicity at stacking faults, where phase stoichiometry may not be maintained. An example of such a lattice defect is a non-conservative antiphase boundary in ordered alloys, so-called because the alloy composition is not conserved at the defect. Such faults occur when the displacement vector does not lie in the plane of the fault [10,11].

Ordered Ni_4Mo has the D_{16}h structure which can be described [11] as a stacking sequence of four (420) fundamental fcc lattice planes of Ni followed by one (420) plane of Mo. A change of this stacking sequence locally changes the composition away from the 4 Ni: 1 Mo stoichiometry. For instance insertion of an extra Ni plane results in a local composition Ni_5Mo whereas removal of a Ni plane changes it to Ni_3Mo . The local lattice periodicity is also correspondingly altered from $5d_{420}$ (disordered fcc notation) to $6d_{420}$ for the former and $4d_{420}$ for the latter, and this would be reflected in the fringe spacing of the lattice image.

Figure 5 shows a lattice image of ordered Ni_4Mo taken using the transmitted beam and $\pm \frac{1}{5}$ (420) reflections, the first order superlattice reflection. As in the isostructural system Au_4Cr , such images reveal directly the superlattice periodicity of the structure [3]. In the arrowed

position, the lattice periodicity is locally altered to be (1.43 ± 0.14) times the average fringe spacing away from the fault. This indicates that the local stacking is 40% greater than normal, such that six nickel planes separate the molybdenum planes at this position. The local composition is thus Ni_6Mo , the stacking fault vector length being $2d_{420}$ perpendicular to the imaged $\frac{1}{5}$ (420) planes. This boundary is clearly non-conservative.

It can also be seen in Fig. 5 that the fault is curved. As the fault plane moves away from the imaged planes, the characteristic lattice image contrast of a translational antiphase boundary is seen [1,2,6] where the fringes of one domain are shifted with respect to those of its neighbor. The amount of displacement equals the antiphase boundary vector, resolved perpendicular to the imaged planes, and also has the magnitude $2d_{420}$ (i.e. 40% of the superlattice periodicity).

This micrograph also illustrates several advantages of the lattice image over the conventional superlattice dark field micrograph [Fig.6] of the same area. Firstly, the displacement at the APB can be seen directly whereas a series of contrast experiments are necessary to establish the fault vector by dark field microscopy. Secondly, the lattice fringes are continuous, to within an atomic diameter of the defect, showing it to have a width of atomic dimensions. The dark field picture shows the presence of a black line on a bright background, the width of which is much larger than that of the actual defect (being as wide as $20 \text{ \AA}$ in some positions). Thirdly, the lattice fringe visibility remains constant right up to the fault indicating that there is no decrease in degree of order in its vicinity - information which cannot be derived

from the dark field image. Similar advantages of lattice imaging over conventional microscopy for atomic detail studies have been emphasized in previous papers. [1-6].

IV. 2. Composition Fluctuations

Although the single fringe measurement is useful in situations such as the above where the deviation from the norm is large, it becomes less beneficial for composition fluctuations occurring in a solid solution. For instance Fig. 7a shows the variation of individual (200) fringe spacings in a lattice image of a spinodally decomposed Au-Ni alloy [4], as measured from a microdensitometer trace. It is clear that the spacing varies markedly from place to place but the definition of the composition profile is rather vague. In addition some of the spacings are considerably higher or lower than those allowed in this system from the composition extrema of almost pure gold or almost pure nickel.

If atomic plane resolution is sacrificed so that we average the measurements over five spacings (i.e. $\sim 9\text{\AA}$), the accuracy rapidly increases (as in Table I) and the now-smoothed profile shows dramatically the fringe spacing variations present in the image [Fig.7b]. Not only is the modulation wavelength ($\lambda = 29 \pm 8 \text{\AA}$) shown by this plot [4] but so also is the wave amplitude. Such an analysis provides information on the form of the composition modulation at an extremely localized level and directly from the specimen. Such information is not present in the conventional images and may only be obtained as an average from processing diffraction data.

The above results demonstrate how lattice imaging can be used for studying systems with large spacing variations (c.f. Fig.4) and it is

to be expected from Table I that its application would be limited in low $\frac{da}{dc}$ systems. This reservation is true to a certain extent, but it is surprising what can still be derived from such alloys. Figure 8 shows an analysis of the (200) d-spacings in a Cu-Ni-Cr alloy aged for a short time [12]. In this system x-ray diffraction data show the maximum lattice parameter difference (Δa) between the segregating fcc phases to be only 1% [12]. Nevertheless, the profile of Fig. 8 clearly shows that there are fringe spacing modulations present, with an approximate amplitude of 0.5%. Resolution was sacrificed to a larger extent to obtain this profile, with an average taken over 15 fringes ($27\text{\AA}$). It is not possible of course to distinguish the spacings of adjacent points on this plot but the overall profile is clearly demonstrated. More details are given in the following paper. Thus information can be obtained even in systems with small lattice parameter differences, as long as a sufficiently large number of fringes are averaged to reduce the experimental uncertainty.

IV. 3. Lattice Constants

It can be seen from Table I that if averaged over a large number of fringes then very precise average spacings may be established, so that the technique becomes a high precision method for lattice constant determination. However, for reliable absolute values the microscope magnification must be known exactly. Although this may be found from a standard specimen, sufficient variation occurs just from day to day and from specimen to specimen that a nominal microscope magnification setting is rarely reproduced exactly. In fact, variations occasionally over 5% have been found in our microscopes during the period of installation so

that it is not possible to trust the setting at any particular time. This means that accurate lattice parameters of a single phase specimen are extremely difficult to obtain, requiring very careful calibrations for each image. However, local deviations from the mean may be established as described above.

A situation where lattice constants may be readily obtained is in a two- or multi-, phase system in which one of the phases is well characterized (e.g. by X-rays) and can be treated as a standard. This is the case for ordered Mg_3Cd thin foils where the hexagonal DO_{19} and orthorhombic B19 phases coexist [6,13] (see Fig. 9). Using the DO_{19} phase as the reference, the B19 constants, found from images and optical diffractograms, are compared in Table II to those predicted from x-ray analysis (extrapolated to the composition 25% Cd from data in Pearson [14]). The results show excellent correlation and emphasize the points made in Section II: (i) that direct measurements on the image are more accurate than those on diffractograms and (ii) that microdiffraction is less precise than macrodiffraction (as seen from the uncertainty in results obtained from the 30 Å and 300 Å equivalent diameter apertures respectively).

TABLE II.

B19 Phase Lattice Constants		
i) Fully ordered alloy	<u>b</u> (Å)	<u>c</u> (Å)
X-ray (estimated)	3.24	5.30
Image (~ 300 Å)	3.24 ± 0.02	5.33 ± 0.03
Optical macrodiffraction (~300 Å)	3.24 ± 0.03	5.27 ± 0.05
Optical microdiffraction (~ 30 Å)	_____	5.21 ± 0.08
ii) Partially ordered alloy		
Image	_____	5.29 ± 0.02

Similar measurements can also be made for dispersed second phase particles such as ordered nuclei in a disordered matrix. This is the condition of partially ordered Mg_3Cd [6]. It can be seen in Table II that once again the lattice constants are those expected of the B19 phase.

It must be pointed out that these latter values were taken (i) from the center of a large domain the fringe profile of which indicates it is fully ordered and (ii) from disordered material some distance ($> 200 \text{ \AA}$) from any ordered regions. These precautions are necessary because of the influence of degree of order and coherency strain respectively on the observed fringe spacing. For instance the transition from perfectly ordered to perfectly disordered lattice periodicity (viz. $5.29 \text{ \AA}$ to $5.46 \text{ \AA}$) occurs over a region $\sim 70 \text{ \AA}$ in width. This is larger than the transition from perfect order (degree of long range order, $S = 1$), to perfect disorder ($S = 0$), which is $\sim 25 \text{ \AA}$ [6]. There must, of course, be the contribution to the lattice constant change from the degree of order profile, but the wider volume over which this change occurs indicates the extent of coherency strains in the vicinity of the interface. It is difficult to separate the two effects in this case to obtain the strain profile alone.

Corresponding analyses of domains either imperfectly ordered or too small to overcome the constraining effects of the matrix revealed that such regions possess fringe spacing values intermediate from those of the two perfect phases. For example, the thin ordered "ribbons" 2-3 unit cells wide (see Fig. 6 of reference [6]) could not be distinguished in spacing from the adjacent matrix (to a precision of $\sim 1.5\%$). Small

coherent domains have values between the two limits depending on their size and degree of order. It is clear then that lattice coherence is an important factor affecting the observed spacings near interphase interfaces. More detailed investigations of such effects are required both experimentally and theoretically. Thus we should emphasize that composition determination should only be made in regions where strain fields are minimal until this problem is resolved. Consequently we do not discuss heterogeneities such as G.P. zones [7] for which the strains are large.

V. DISCUSSION

The above considerations have shown that precise lattice fringe spacings can be determined in highly localized areas. A typical working figure is $\pm 0.5\%$ accuracy with a resolution of $20 \text{ \AA}$. In favorable situations (e.g. interstitial solid solutions, stacking faults) even individual fringe spacings can be used reliably for analysis. This potential capability is indeed an exciting development for characterizing defects and transformations close to the atomic level.

In terms of spatial resolution this new approach is superior to conventional TEM methods such as electron microscopy with microanalysis, EMMA, ($\sim 1000 \text{ \AA}$) [15] and electron velocity analysis ($\sim 100 \text{ \AA}$) [16], and to scanning transmission electron microscopy (STEM) with x-ray microanalysis ($\sim 500 \text{ \AA}$ as currently available) [17]. In the future, electron velocity analysis in a scanning TEM with a field emission source may be able to achieve similar resolutions. Field-ion microscopy (FIM) with an atom probe [18] of course is capable of individual atom analysis, although the difficulties associated with collecting sufficient data and the limited range of materials appropriate for FIM restrict its general applicability. Lattice fringe imaging, like TEM, has a broad range of application. Its own limitation, for compositional analysis, lies in the dependence of lattice spacing on composition and if there is insufficient variation then high resolution data may not be acquired.

The present article has relied on the assumption that a straightforward relationship exists between local composition and lattice spacing. However, this may not be exactly the case. Electron optical phenomena

are known to affect lattice fringe spacings [19], the most important parameters being specimen thickness and deviation (s) from the exact Bragg condition. These factors can be controlled by careful experimental work. For two beam lattice images, if the specimen is oriented exactly on the Bragg condition ($s = 0$), the observed fringe spacings have exactly the spacing of the diffracting lattice planes. As s increases from zero, the observed spacing can also increasingly deviate from the true spacing, depending on the specimen thickness and shape [20]. In our experiments we take care to be as close to the Bragg condition as possible by reference to the diffraction pattern from the area of interest and to the position of the appropriate bend contour. It also transpires that the lattice fringe visibility rapidly deteriorates as the orientation departs from $s = 0$ (e.g. ref. [2]), so in fact obtaining good quality images goes a long way in ensuring close proximity to the desired orientation. The situation where large, very localized changes occur in lattice configurations (and hence Bragg diffracting conditions) such as at dislocations is a more complex problem which is currently undergoing theoretical investigation [21].

The second point concerns lattice constraints, which are manifested in the last example (Section IV.3). It does not seem possible to separate coexisting composition and strain effects by a single lattice image experiment. In an order-disorder system the strain effect is obtainable since the degree of order profile (and hence its contribution to the lattice spacing variation) is tractable from the computed fringe profile versus degree of order. However, this is not a trivial problem. Some carefully designed experiments are necessary to resolve the situation, with accompanying theoretical analyses taking into account

the elastic properties of the material. Thus at present the method is best applicable for alloy systems where strains at heterogeneities (e.g. interphase interfaces) are small. Recent work by Grønsky [22, 23] on grain boundary interfaces further indicates that the lattice imaging method can give valid composition profiles. In such cases the strains are expected to be limited to the boundary plane itself.

However, we should emphasize that caution needs to be exercised in applying this method in general. For highly strained discontinuities in alloys it is not possible to separate the influence of the strain field on the composition profile and in such cases evaluation of absolute composition profiles are intractable until appropriate theoretical work is carried out.

VI. CONCLUSIONS

Lattice imaging provides a method of obtaining lattice parameter data at an extremely localized level. For instance in typical metallurgical studies an accuracy of $\pm 1\%$ may be achieved over $10 \text{ \AA}$, whilst averaging over $30 \text{ \AA}$ involves an uncertainty of $\pm 0.3\%$. In order to obtain higher precision the resolution of the method must be sacrificed. This situation implies that, in appropriate systems where there is sufficient variation of lattice spacing variation with composition ($\frac{da}{dc}$), information can be obtained on composition in highly localized areas. Thus in an alloy with $\frac{da}{dc} \sim 0.16$, composition differences of only $\sim 3\%$ can be detected with a resolution of $20 \text{ \AA}$. This should prove to be particularly important for interstitial solid solutions.

It must also be pointed out that these conclusions are subject to the limitations imposed by consideration of electron optical and lattice constraint effects. It is thought that by careful choice of imaging conditions the former is readily overcome for the present application, whereas the influence of the latter is yet to be established. The method should be limited for the present to cases such as those considered here.

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- Figure 1. A lattice image of gold. The average lattice spacing is determined more precisely if a larger number of fringes are used in the measurement. In this case the position of the lattice fringes can be specified to $0.1 \text{ \AA}$, and thus the experimental uncertainty is $\pm 5\%$ for one fringe, $\pm 0.5\%$ for ten fringes and $\pm 0.05\%$ for one hundred fringes.
- Figure 2. A (001) optical diffraction spot from a lattice image of Bi_2Se_3 , taken using a $370 \text{ \AA}$ equivalent diameter aperture. The fine structure in the spot limits the accuracy of defining its position and hence the determination of lattice spacing by this method. The dimension shown corresponds to an uncertainty of $\pm 0.15 \text{ \AA}$.
- Figure 3. Weak lattice image of a Cu-Ni-Cr alloy and its corresponding optical diffraction pattern taken with a $120 \text{ \AA}$ diameter equivalent aperture (courtesy of C-K Wu [12]).
- Figure 4. Variation of lattice parameter difference ($a - a_0$) with composition (c) for three typical alloy systems, (with high, medium or low da/dc). If the accuracy of the spacing determination is 1%, only gross composition differences can be detected in the latter case. With 0.5% accuracy very small compositional variations can be detected in the former cases, but still only large variations in the latter.
- Figure 5. A lattice image of superlattice planes in ordered Ni_4Mo in the vicinity of a translational antiphase boundary. The change of fringe spacing of the non-conservative portion of the boundary (arrowed) is clearly seen.

Figure 6. A conventional superlattice dark field image of the anti-phase boundary shown in Fig. 5. considerably more information concerning the defect is present in the lattice image (Fig. 5).

Figure 7. Variation of (200) d-spacing in spinodally decomposed Au-Ni (a) determined by measuring individual fringes; (b) determined by averaging over five fringes [4]. The improvement in clarity of the profile by the averaging procedure is clearly shown (a) courtesy of R. Gronsky [22], (b) courtesy of Acta Metallurgica).

Figure 8. Variation of (200)d-spacing in spinodally decomposed Cu-Ni-Cr showing that even in systems with small lattice parameter difference, highly localized spacing modulations can be detected.

Figure 9. Lattice image of Mg_3Cd showing the coexistence of the hexagonal DO_{19} and orthorhombic B19 phases.

XBB 777-7170

XBB 777-7171

Fig. 2

XBB 773-1685

Fig. 3

XBL 769-7538

Fig. 4

XBB 7410-7097

Fig. 5

XBB 7412-8987

Fig. 6

XBL 757-6760

Fig. 7(a)

XBL 757-6648

Fig. 7(b)

XBL772-5109

Fig. 8

XBB 770-11426

Fig. 9

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