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SOME APPLICATIONS OF TRANSMISSION ELECTRON MICROSCOPY*

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1. Introduction

The most important advantage of transmission electron microscopy in materials science is its ability to provide almost all the data needed to characterize completely the microstructure of materials,\(^{(1,2)}\) as indicated by the scheme shown in Fig. 1. Today it is possible to resolve substructural details down to \(~20\text{Å} \) (discrete particles) and atomic plane spacings \(~1\text{Å} \) can be imaged. The new technique of velocity analysis (e.g. Refs. 3-5) has considerable promise for obtaining information regarding the chemistry of the specimen down to the resolution limits of the instrument.

The applications of the technique are almost unlimited\(^{(1,2)}\) and it is obviously impossible to describe all of them here. However, some general methods of identification are discussed as applied to two or more phase systems resulting from phase transformations, excluding martensites.

Most commercially useful alloys contain two or more phases as a result of thermal or more complex thermal-mechanical processing. It is important to characterize the structure in terms of alloy composition and treatment as this approach is a necessary part of efforts to control alloy performance through control of structure. This chapter is not

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intended to be comprehensive, but is representative of some typical research work in materials science and engineering at Berkeley.

In the analysis of structure equal attention must be given to the diffraction pattern, corresponding bright and dark field images, and how these change upon tilting. As indicated in fig. 2 of the Chapter on Kikuchi diffraction, only two orientations are necessary for the analysis of microstructure, namely a) the symmetrical orientation, to provide accurate crystallographic information, e.g. orientation relations, identification of phases by utilizing the camera constant equation, and so on, and b) the two beam orientation so that contrast effects can be investigated in terms of various $\mathbf{g} \cdot \mathbf{R}$ conditions.

The interpretation of the image and the diffraction pattern is of course facilitated by careful selected area diffraction analysis, together with dark field investigations of the features present. It is important to emphasize that several photographs of a given area under various controlled conditions is far better than random photography of numerous areas. When the second phase particles are large enough or are sufficiently numerous to provide good diffraction patterns the analysis is generally straightforward except that care must be taken to isolate diffraction phenomena resulting from double diffraction as will be indicated in section 3.

Before discussing some typical examples of analysis of multiphase structures some points will be made with regard to the two basic techniques of identification viz selected area diffraction and the dark field technique.
2. Basic Techniques

a) Selected Area Diffraction

This technique\(^1,2\) provides a means for selecting various features of the image for examination by diffraction and dark-field imaging. In order to distinguish between several different regions of the specimen it is necessary to be able to select the smallest possible area e.g. in diffraction analysis of small and different particles.\(^1\) The minimum selected area \((\Delta A)\) contributing to the diffraction pattern is limited chiefly by the spherical aberration \(C_s\) of the objective lens as follows:

\[
(\Delta A) \approx C_s \theta^2_B = C_s (\lambda/d)^3
\]

where \(\theta_B\) is the Bragg angle of a set of reflecting planes spacing \(d\) for incident wavelength \(\lambda\).

Since \(C_s\) is roughly proportional to \(\lambda^{-1}\), then \(\Delta A \sim \lambda^2\) and decreases rapidly with increasing accelerating voltage (decreasing \(\lambda\)). Thus, there are advantages in high voltage microscopy for identifying small particles by selected area diffraction, e.g., at 100kV \(\Delta A \sim 2\mu\), while at 1 meV \(\Delta A \sim 0.02\mu\).

The procedures which must be observed to eliminate errors and misinterpretation in carrying out selected area diffraction (SAD) are discussed in detail in Ref. 2. It must be remembered that when correlating the SAD pattern to the image, all rotations (optical and magnetic) must be accounted for and pre-calibration is essential. For example, in microscopes of lens sequence objective, intermediate, projector there is an optical rotation of 180° between image and pattern. For alignment fix the image and rotate the SAD pattern (both plates emulsion...
(up) $180^\circ$ plus the magnetic rotation, clockwise.

b) **Dark Field**

High resolution images are obtained only when the diffracted beam to be utilized for the dark field image is tilted so that it passes down the optical axis of the microscope. This is necessary in order to minimize errors such as chromatic aberration and spherical aberration, but objective aperture images of off axis beams can be used for preliminary investigations since these can be obtained very quickly. The important point is that in order to obtain the dark-field image of maximum intensity by tilting the illumination and without tilting the specimen, it is necessary to tilt the optical system in a direction which translates the origin to the point occupied by the Bragg spot responsible for bright field contrast. (6) This operation reverses the sign of $\mathbf{g}$ as shown in Fig. 3. This procedure applies whether manual or electrical tilting systems are to be used. The advantages of the electrical system include ease and speed of operation and fast switching from bright to dark field and vice-versa.

The advantages of dark field imaging are many as some of the illustrations indicate. Also the images of defects near either the top or bottom surfaces can be selectively brought into contrast by dark field imaging at $s < 0$ and $s > 0$, respectively (7) e.g. Fig. 5(a).

As an example of the combined use of diffraction pattern and dark field imaging consider Fig. 3 which is of a commercial aluminium alloy designed for cryogenic applications. * The two beam bright field image at $s = 0$

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* I am grateful to Dr. D. Rowcliffe of Lockheed, Palo Alto, for providing this specimen.
of the 200 reflection (Fig. 3a) shows strain contrast from plate shaped particles. No strain contrast images are seen for \( \mathbf{g} \cdot \mathbf{R} = 0 \). For fig. 3 the plates on (100) are visible, and so \( \mathbf{R} \) is normal to (100). The orientation is [011] (Fig. 3b) and the inclined plates on (0110) and (001) are not in suitable orientation for strain contrast since they (and hence \( \mathbf{R} \)) are inclined at 45° to the foil plane normal. In order to obtain strain contrast \( \mathbf{g} \cdot \mathbf{R} \) must be a maximum i.e., \( \mathbf{R} \) should lie in the foil plane. Thus plates must be oriented to be nearly parallel to the incident beam. In fig. 1(c) a dark field image is obtained of the streak near the 200 spot. No strain contrast is observed, only "structure factor contrast." In order to utilize such dark field images small objective apertures are required so that only one diffraction phenomenon is to be imaged at a time. This is not always possible of course when reflections are close together.

3. Double Diffraction

The complexities of a diffraction pattern are most easily solved with the aid of dark field analysis of the various features present. In this way the maximum information can be obtained with minimum errors of identification. In two phase structures it is essential to recognize special effects due to double diffraction in order to correctly identify the phases which are present. This is also effected by dark field experiments.

In single phase BCC or FCC structures, double diffraction does not produce reflections of zero structure factor in the pattern since multiple reflections yield only allowed positions, e.g., in [110] BCC orientations

\[
002 + \pm 110 \approx 112 \text{ or } \overline{1}12
\]

or

\[
110 + \pm 112 \approx 222 \text{ or } 002 \text{ and so on.}
\]
Twinning in these systems can however produce double diffraction, \(^2\) and it also occurs in less symmetrical structures such as diamond cubic or HCP, e.g., 200 (DC) and (0001) HCP.

In situations where two (or more) phases can exist, then double diffraction becomes more probable and care must be taken to identify spots which are due to this phenomenon. A spot which is present as a result of double diffraction, when used to form a dark field image will simultaneously reverse contrast in the corresponding diffracting regions of the phases responsible.

As an example consider Fig. 4 taken from a Fe-Ni-Co maraging alloy which consists of a Widmanstätten pattern of austenite plates in martensite \(^8,9\) (for review see Ref. 10). The SAD pattern consists of a mixture of the basic BCC [0\(\bar{1}\)1] orientation (martensite) on which is superimposed several FCC orientations (austenite) in (111). For example, spot C is a 220 austenite reflection which in the dark field image (Fig. 3c) reverses contrast only at those austenite plates that have this particular orientation. Spot (d) is a double diffraction spot produced by interactions of BCC and FCC reflections as shown by its dark field image in Fig. 3(d); similarly for spot (b). Thus, the "quartets" of spots along (112) BCC in the pattern are mixtures of FCC reflections and double diffraction. They are not due to any intermetallic compounds. Similar arguments apply to any multiphase structure.

4. Detection and Identification of Small Coherent Particles

When second phase particles are large enough to give diffraction effects which are recognizable from those due to the matrix, their contrast can be reversed by dark field imaging and detection is not
difficult. However situations can arise when it is not easy to be
certain that the microstructure is indeed two-phase or not since the
diffraction pattern may remain single crystal orientation with no
extra spots visible and other diffraction effects due to second phases
(strain, shape factor) may be too diffuse to be easily detected. Thus
in characterizing the microstructure of alloys, it is important to
attempt to utilize all possible pieces of information which may be
present - observable or otherwise. This requires painstaking work in
bright and dark field, obtaining high resolution, well exposed diffraction
patterns, and ensuring that no spurious effects due to foil preparation
(e.g. rough surfaces) do not confuse the issue. As will be illustrated
in Section 4(b), even though individual particles may not be resolvable,
evidence for decomposition into two (or more) phases can be obtained
by considering various possible contrast effects. Thus it is convenient
to consider two general cases a) when individual particles can be
resolved and b) when they cannot. Case (b) seems to be characterized
by systems in which small, very coherent particles form in very large
densities (> $10^{17}$ cm$^{-3}$) so that any strain fields overlap and strain
contrast images reveal net matrix strains rather than individual particle
strain contrast. Examples include Cu-Be, short range ordering in
alloys such as in Fe-Al, Fe-Si, Cu-Al, Ni-Mo and possibly in interstitially ordered alloys such as Ta-C.

a) **Resolvable Particles**

The visibility of precipitate particles depends on one or more
different contrast mechanisms as discussed in detail in Ref. 2, e.g.,
a) diffraction contrast due to coherency strain fields, resulting from the
matrix deforming locally to accommodate the precipitate, e.g. Fig. 3a,
b) contrast due to the differences in structure factor between the phases present e.g. fig. 3(b), (c) interface contrast in the form of fringes due to misorientation, phase contrast (as with stacking faults or antiphase boundaries) or geometrical effects such as at wedges, c) Moiré contrast due to overlapping reflecting planes diffracting simultaneously in matrix and precipitate, e) contrast due to the matrix being in diffracting orientation when the particle is not (or vice versa), f) thickness differences due to preferential thinning of the one phase with respect to its neighbor when preparing foils, g) interface dislocation contrast when phases are partially coherent (e.g. figs. 14, 15).

When the precipitate particles are coherent, of similar lattice spacings, and are small with respect to the extinction distance (~100Å or less) only the first two contrast mechanisms are significant. Such cases of precipitation occur generally in alloys which undergo decomposition from supersaturated solid solutions such as age-hardening alloys (see e.g. Ref. 16 for review).

In the early stages of precipitation when large numbers of small particles can be formed several factors influence the microstructure and observed contrast, e.g., a) coherency strains, b) volume fraction, c) elastic anisotropy, represented by the ratio of the shear moduli \( G_{(110)} : G_{(100)} \).

In aluminum alloys where the anisotropy is \( \approx 1 \), the shapes of particles are determined by the atom size factor. Spherical (as in Al-Ag), needle-like (Al-Mg₂Si) and plate-like (Al-Cu) particles have all been identified. Needles and plates are most easily distinguished in the early aging stages when particles are very small by analysis of the SAD pattern since this is modified by the shape factor. Needles give rise to rel-sheets of intensity perpendicular to their axes while
plates give rise to reI-rods, normal to the plane of the plate.\(^{(1,2)}\)

Figure 5 shows examples of needles in Al-Mg\(_2\)Si and the diffraction pattern shows the curved streaks which are produced when the reflecting sphere intersects the sheets of intensity which are normal to \(\langle 100 \rangle\) for all needles which do not lie normal to the beam. By comparison to Fig. 5(b) the straight streaks in Fig. 6 are due to small plates (GP zones) on \(\{001\}\). Thus, the [110] orientation immediately distinguishes between plates and rods in these alloys. Shape factor effects can be understood geometrically by the superposition of all respective reciprocal lattices.\(^{(17)}\)

If the volume fraction of the particles is not too large so that individual small particles can be resolved by diffraction contrast, and the strain fields do not overlap to a considerable extent, qualitative information can be obtained as to the direction and magnitude of strain and the shape and dimensions of the particles by utilizing the strain contrast rules to dark-field images first developed by Ashby and Brown.\(^{(18)}\)

These rules can be summarized by reference to Fig. 7 which shows the type of strain contrast images expected for different (simple) defects under conditions where \(\mathbf{g} \cdot \mathbf{R} \neq 0\) (where \(\mathbf{R}\) is the displacement vector). These images show dark contrast to the same side as \(\mathbf{g}\) when the strain field is interstitial in nature and white when the strain field is of the vacancy type, provided that exactly \(s = 0\) condition applies to the area being considered.\(^{(18)}\) In principle, therefore, the sense of the image contrast with respect to the direction of \(\mathbf{g}\) can be used to determine the sense of the strain fields. However, due to the periodic intensity oscillations with depth in the foil as illustrated in Fig. 8, it is essential that the position of the particle in the foil be accurately known when
the size of the particle is smaller than the extinction distance before this rule can be applied. Details of this type of analysis are described in the chapter by Wilkins. If precipitation has in fact occurred in the bulk of the material and not on the surfaces then roughly half the particles have white contrast and half have dark contrast on the same side as the direction of \( \mathbf{g} \), (Fig. 9). Also, if the particle size is large (of same order as the extinction distance) there is no problem in defining the sense of displacement from the image e.g. the larger \( \theta' \) plates in Fig. 9, all have their white images on the same side while the GP zone images have mixed contrast.

b) Alloys Containing Very Large Volume Fractions of Small Particles

The illustrations of Figs. 3, 8, and 9 show that strain contrast images are determined by \( \mathbf{g} \cdot \mathbf{R} \) where \( \mathbf{g} \) is a matrix reflection and \( \mathbf{R} \) represents the displacement(s) around the precipitate. If the precipitate reflection is used to form a dark field image, e.g., the streak in Fig. 3, only structure factor contrast is observed. Such images thus may provide more accurate information regarding true particle size and shape than the strain contrast image.

Difficulties in resolving particles may be encountered when the volume fraction is very large and the particles are very small (< 100 Å) so that the strain fields overlap. The image contrast can then be determined by net strains rather than by individual particles and it is convenient to refer to this effect as net strain contrast. The simplest example is found in the Cu-Be precipitation hardening system(11) in which only (110) strain "striations" (also called tweed patterns) can be observed during the initial stages of aging (see Fig. 10a). Tanner(11) in a detailed investigation concluded that the strain patterns
were due to small plates on (100), but with strain fields in (110).
This result was inferred from a series of $\mathbf{g} \cdot \mathbf{R}$ experiments. However, in Cu-2% Be additional helpful information can be gained from the diffraction patterns, for example in Fig. 10(b) streaks are observed in (100) from rel-point to rel-point proving that small plates with (001) habit must be present. The interpretation of strain fields in (110) is confirmed by the short (110) diffuse streaks observed in the pattern. The reason for (001) (110) strains may be related to the anisotropy factor $> 1$. When the volume fraction is high, Fig. 7 is no longer an accurate guide to interpretation but the zones in Cu-Be if resolved should appear as in Fig. 7 II with $\mathbf{b} = [100]$ and $\mathbf{q} = [110]$.

Similar but not identical results occur in short range ordered alloys. For example, Fig. 11 shows a series of $\mathbf{g} \cdot \mathbf{R}$ experiments on Ni-20%Mo which indicate that $\mathbf{R}$ is principally along (110) but no individual particles can be resolved until aging is prolonged sufficiently so that superlattice spots can be seen. In this case the dark field image of a superlattice spot reverses contrast for particles of that particular orientation (Fig. 11g). However, unless diffuse or superlattice spots are present the net strain contrast image may be the only indication that precipitation has occurred. If the individual particles cannot be resolved and shape factor and/or strain diffuse streaks cannot be seen or distinguished, there is little more to be concluded other than some small particles, of undefined shape and

*The anisotropy factor is also important when coherency is lost; e.g. partially coherent dislocations are generated with minimum energy expenditure so that dislocations with $\mathbf{b} = (110)$ rather than (100) may exist. (cf. figs. 14, 15)
associated strain vector are present. Considerable effort is currently being devoted to this problem in order to better define physically the meaning of short range order. For example fig. 12 shows a SAD pattern of Fe-19% Al after aging one week at 300°C. This pattern may be interpreted to be single phase, single crystals, but ordered with super-lattice spots at \(h00\) and equivalent positions. However, a dark-field image of a "superlattice" spot, fig. 12(b), clearly reveals small particles. The microstructure may thus be characterized as two phase with small ordered particles in a disordered matrix, and the pattern in (a) can be regarded as consisting of two superposed ordered and disordered patterns of the same \([001]\) orientation and almost identical lattice parameter since no doubling of the spots is seen. Thus the two phases in (b) must be almost ideally coherent. This characterization seems to indicate that short range ordered alloys consist of small ordered particles in a disordered matrix. It should be emphasized that it is difficult to prove that two phases exist when the contrast is very weak and no obvious effects are to be seen in the SAD patterns. Other techniques such as field ion microscopy may then be combined to advantage.

5. **Spinodal Decomposition**

In spinodal decomposition, periodic fluctuations in composition occur along the elastically soft directions (generally \(001\) in metals). The consequence of this on the diffraction pattern is to produce "side-bands," i.e., satellite reflections alongside the main reflections in a direction parallel to the direction of modulation. The important feature of the sidebands is that their spacing depends only on the wavelength of the compositional fluctuation. For cubic phases the wavelength can be calculated from
\[
\lambda = \frac{h(a_h k' l') \tan \theta}{(h^2 + k^2 + l^2) \Delta \theta}
\]

where \(h'k'l'\) is direction of fluctuation and \(\Delta \theta\) the angular separation between spot and satellite. For high energy electrons and for fluctuations along [h00] this reduces to

\[
\lambda = \frac{ha}{(h^2 + k^2 + l^2)} \left( \frac{g}{\Delta g} \right)
\]

where \(a\) is the lattice parameter, and \(g : \Delta g\) is given by the ratio of the distances from 000 and satellite to the hkl spot. Similar equations apply to the O00, 00l directions of fluctuation. The wavelengths obtained from Fig. 13 increase from 60\(\text{Å}\) (b) to 160\(\text{Å}\) (e).\(^{(21)}\) This method of measuring \(\lambda\) is more convenient than direct measurements of the image for two main reasons viz., a) because the sideband spacing is independent of magnification and b) because mean values are immediately obtained for all distributions of \(\lambda\) contributing to the pattern (i.e., within the selected area).

When the phases are coherent with small strains involved the images do not show strong strain contrast. For Cu-Fe-Ni the strain is < 0.6\% [see Fig. 17(b)]. Interface fringes (\(8\)-fringes\(^{(22)}\)) typical of coherent boundaries can be observed when the interface plane is inclined to the incident beam. Many examples of \(8\)-fringes have now been given, e.g. at domain interfaces.\(^{(22-24)}\) The characteristic of these fringes is that their contrast is the opposite to that of stacking fault contrast in FCC systems. Delta fringes show asymmetry at the top and bottom of the foil in bright field, and are symmetrical in dark field. These fringe patterns occur due to the slight mismatch in lattice spacing at the interface giving rise to a \(\Delta g\) (and \(\Delta s\)) mismatch in the reciprocal lattice. In such cases splitting of the diffraction spots and Kikuchi lines may be resolved particularly at large \(\theta\) values.\(^{(25)}\) From measurements of the Kikuchi patterns the axial ratios of non-cubic structures
may be measured quite accurately. (23) It should be noted that satellites from modulated (side-band) structure can be distinguished from $\Delta g$ splitting since the spacing of the former depends only on spinodal wavelength whereas the latter increases with $|\vec{g}|$.

6. Partially Coherent Phases

When the misfit between two phases exceeds that which can be tolerated by elastic distortion (visible as strain contrast), coherency breaks down and interfacial dislocations can be generated. Mechanisms to account for loss of coherency have been investigated in several alloy systems by Weatherly and Nicholson. (26) In Al-4% Cu coherency between $\theta'$ plates and the matrix is lost by the generation of prismatic pure edge-dislocation loops inside the plates with the loop lying parallel to the habit plane (001). The contrast from these loops is directly analagous to the contrast from large edge-loops in quenched or irradiated crystals. For a plate viewed with its normal parallel to the incident beam $\vec{g} \cdot \vec{b} = 0$ but the resolved components of the displacement normal to $b$, i.e., $\vec{b} \times \vec{u}$ where $\vec{u}$ is a vector normal to $\vec{b}$ are only normal to $\vec{g}$ where $\vec{g}$ is tangent to the loop ($\vec{g} \cdot \vec{b} \times \vec{u} = 0$). This is the only condition when $\vec{g} \cdot \vec{b} = 0$, and $\vec{g} \cdot (\vec{b} \times \vec{u}) = 0$ is simultaneously satisfied. The image has maximum contrast when $\vec{g}$ and $(\vec{b} \times \vec{u})$ are parallel. Thus, the image consists of double arcs normal to $\vec{g}$ and the images are complementary on opposite sides since $\vec{g} \cdot (\vec{b} \times \vec{u})$ changes sign about a line through the center of the plate. An example is shown in Fig. 14. The doublets and triplets in the images are due to the effect of foil thickness on image contrast. Triple images occur for loops near the bottom surface of the foil.

For precipitates, such as $\theta'$ in Al-Cu, which form on cube planes,
the misfit dislocations are expected to have their Burgers vectors along (100) in the interface, i.e., be pure edge. However, this is not a universal rule and interesting differences occur in the Cu-Fe-Ni spinodal system when the wavelength > 1000Å and coherency is lost. (21)

Figure 15 (a,b) shows the interface structure. Only one set of interface dislocations has been generated and these have \( \mathbf{b} \) of the \( a/2 \langle 110 \rangle \) type. The mismatch \( \delta = \frac{|b|}{d} \), where \( d \) is the spacing, is \( \approx 0.6\% \). The corresponding diffraction pattern (Fig. 16) shows a splitting in the [010] but not in the [100] direction, (e.g., the 440 spot). The mismatch in Fig. 14(c) \( \approx 0.7\% \). This result shows that as coherency is lost the structure must change from cubic to tetragonal. At later stages when a second set of dislocations has been generated so that all strains are relieved at the interface, the structure becomes cubic again. (21)

The contrast experiments illustrated by Fig. 15 prove that the Burgers vectors of these interface dislocations are parallel to \( \langle 110 \rangle \) directions with the dislocations lines along \( \langle 100 \rangle \). Thus, roughly twice as many dislocations are required to relieve the mismatch than if dislocations of \( \mathbf{b} = a\langle 100 \rangle \) were formed. It is possible that nucleation of dislocations with \( a/2\langle 110 \rangle \) Burgers vectors is more favorable energetically than \( a[100] \) type in this system and in view of the high anisotropy ratio.

7. Note on Grain Boundaries

Phase transformations from supersaturated solutions occur neither by nucleation and growth, (N and G), homogeneously as in the spinodal, or martensitically. The latter case will not be discussed here. Homogeneous and heterogeneous decomposition can be distinguished morphologically
since in the latter case nucleation is favored at defects such as dislocations, boundaries, faults, point defects, etc. Noticeable differences occur at and near grain boundaries as illustrated in Fig. 17. Figure 17(a) shows a grain boundary microstructure in Al-Zn-Mg alloy showing typical N + G conditions with precipitation along the boundary, precipitate free zones adjacent to boundaries (PFZ) (27) and a decrease in number (but increase in size) of particles in the matrix adjacent to the PFZ. The homogeneous microstructure resulting from spinodal decomposition of Cu-Fe-Ni on the other hand (Fig. 17b) shows none of these features. Although it is easy to distinguish such heterogeneous and homogeneous transformations, it is not recommended as a general principle to use microstructure to define the type of transformation since this is determined by kinetic and thermodynamic principles, and usually the experimentalist never really "sees" the nucleation (or lack of) stage in the process, even when direct observations are possible.

8. Dynamic Studies of Phase Transformations

As indicated in Fig. 1 it is possible to obtain direct observations of dynamic events, e.g., deformation, precipitation, recovery, etc., with the use of appropriate specimen stages and cine techniques (see Ref. 28 for review). The high voltage microscope may be useful in this regard since it will be possible to work with thicker specimens and possibly have fewer problems due to surface effects. Some examples have already been given by Fujita, et al. (29)
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Fig. 1  Scheme illustrating the main functions of the electron microscope.

Fig. 2  Sketches showing that for maximum contrast the dark field image must be formed of the negative diffraction vector corresponding to the bright field image; thus the sign of $\mathbf{g}$ is reversed from bright to dark field. The specimen is not tilted.

Fig. 3  Al-6%Cu-0.15%Cd-0.05%Sn (+Zr, Ti) designation Al 2021 aged 160°C for 18 hrs; a) bright field image $g = 200$, b) dark field image of streak (circled), c) diffraction pattern.

Fig. 4  Fe-Ni-Co alloy air cooled from 2100°F and aged 3 hrs at 900°F. a) bright field image, b) dark field image of double diffraction spot b, c) dark field image of austenite spot c (220 type), d) dark field image of double diffraction spot (d) notice weak dark field contrast of precipitate and matrix, e) dark field image of austenite spot e (220 type).

The diffraction pattern shows the [011] symmetrical BCC with three superimposed FCC patterns in (111) orientations corresponding to the (011) Widmanstätten plates of austenite. Spots such as at b and d are due to double diffraction of austenite and martensite reflections.
Fig. 5 (a) foils of Al-Mg$_2$Si quenched and aged 5 hr. 220°C; dark-field at s < 0, only precipitates near top surface are in good contrast. The needles along [100] and [010] inclined at 45° exhibit extinction contrast and strain contrast. The strain contrast indicates interstitial strain fields. (b) SAD pattern dark-field of (a) notice parabolic streaks spread about [002] due to the [100] and [010] needles, and straight streaks along [220] due to the [001] needles. (c) Schematic representation of the intensity sheet distribution about rel-points. (d) Diffraction pattern (~[103]) of needles showing cancellation of intensity at nonallowed reflecting positions (cf-c).

Fig. 6 [110] orientation of Al-Cu aged to form GP zones on [100] the streaks along [001] show that plates and not needles are present (compare to 5b).

Fig. 7 Scheme illustrating strain contrast images expected from individual precipitates

I plate shaped particle displacement vector $\mathbf{b}$ normal to habit plane or rods viewed with axes normal to the beam.

II As I but displacement vector inclined to habit plane.

III Similar to a screw dislocation end-on.

IV Spherical particle or rod viewed end-on.

In I and II the image (line of dividing contrast LC) is independent of $\mathbf{g}$; in III LC is parallel to $\mathbf{g}$, in IV LC is normal to $\mathbf{g}$.

These predictions may be modified when the volume fraction of particles is very large so that strain fields overlap and interact, and for more complex shaped particles.
Fig. 8  Rods of precipitate formed in silicon doped with phosphorus strain contrast images; the rods lying normal to the beam indicate vacancy contrast (cf. Fig. 7-I) but the inclined rod shows oscillatory contrast due to the depth dependence of the intensity.

Fig. 9  Al-4% Cu dark field strain contrast image showing heterogeneously precipitated θ' (large plates) and GP zones. Compare to Figs. 3 and 7-I. Displacement R parallel to $\mathbf{g} = 200$. Foil orientation [001], plates on (010) are not visible since $\mathbf{g} \cdot \mathbf{R}$ is zero for these.

Fig. 10  Cu-2% Be aged to form GP zones (a) bright field image $\mathbf{g} = [220]$, (b) sketch of SAD pattern to show (110) strain diffuse streaks and (100) form factor streaks.

Fig. 11  Ni$_4$Mo quenched to ice brine from 1100°C aged 5 secs 750°C (a-d) foil near [110]. Courtesy P. R. Okamoto.

a) bright field two beam $\mathbf{g} = 002$

b) bright field two beam $\mathbf{g} = 1\bar{1}1$

c) bright field two beam $\mathbf{g} = \bar{1}11$

d) bright field two beam $\mathbf{g} = 220$

Solid and dashed lines show traces of (110) planes. Strain contrast in (110) is parallel to solid lines.

e) bright field $\mathbf{g} = 0\bar{2}0$ after tilting to near [001] zone.

f) dark field of superlattice reflection showing one of the six orientational variants possible for ordered Ni$_4$Mo particles.
Fig. 12  Fe-19% Al aged 1 week 300°C, a) diffraction pattern
b) dark field image of "superlattice spot," showing
two-phase nature of the alloy.

Fig. 13  51.5 Cu-33.5Ni-15 Fe alloy quenched and aged at 625°C
showing development of side-bands due to spinodal decompo-
sition along (100).  a) fast quenched (no decomposition),
b) 1 min, c) 5 min, d) 15 min, e) 60 min.  Notice decrease
in satellite spacing at 400 reflection as aging increases
($\lambda$ increases).

Fig. 14  Showing interfacial pure edge prismatic loops around
partially coherent $\theta'$ plates in Al-4% Cu.  Foil in [001].
Notice that $\vec{g} \cdot \vec{b}$ is zero for one set of edge-on plates
and $\vec{g} \cdot \vec{b}_{\text{alu}}$ and $\vec{g} \cdot \vec{b}$ is zero simultaneously for parts
of loops with $b = [001]$ normal to the foil plane.  Compare
to strain contrast images of $\theta'$ in Fig. 9.

Fig. 15  Interfacial dislocations in spinodal Cu-Fe-Ni alloy aged
200 hrs 700°C visible for $g = 1\bar{1}1$ in (a) but invisible
for $g = \bar{1}11$ in (b).  The dislocations lie in [100]; their
most probable Burgers vector is along [011].

Fig. 16  Diffraction pattern corresponding to area similar to
that in Fig. 15.  Zone is [001].  The resolvable doublet
at 440 is parallel to the interface normal.  Notice that
there is no splitting along [100].  The structure is
thus tetragonal.

Fig. 17  a) Heterogeneous precipitation in Al-Zn-Mg.
b) Homogeneous precipitation in Cu-Fe-Ni spinodal.
Fig. 1

XBL 695-581
Fig. 2

(a) Bright field
(b) Dark field

Transmitted beam
Diffracted beam
Ewald sphere
Diffracting planes in crystal
Electron gun

$\theta$

$2\theta$

$\vec{g}$

Aperture
Axis of microscope column

MUB-3766
Fig. 5
Fig. 7
Fig. 8
Fig. 9
Fig. 10
Fig. 11
Fig. 12
Fig. 13
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