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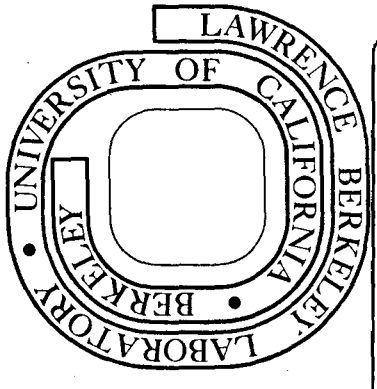

DISCLAIMER

ON STRUCTURAL INTERPRETATION OF SHORT RANGE ORDER†

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ABSTRACT

The various structural models of short range order in alloys are briefly reviewed. The success and limitations of structural models in interpreting diffuse scattering data are discussed. Particular emphasis is given to the systems for which the diffuse short range ordered maxima in the X-ray and electron diffraction patterns are non-coincident with the position of the superlattice reflections. The recent electron diffraction results on these systems suggest microdomains with different superstructures was present in the short range ordered state. The justification for such multiple microdomain configurations is discussed in the light of some recent theoretical developments.

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I. INTRODUCTION

There are two different models used to interpret the diffuse scattering observed in X-ray, electron or neutron diffraction patterns due to short-range ordering of atoms. One is the statistical thermodynamic model which tries to interpret the diffuse scattering directly in terms of the pair-wise interaction potentials between atoms. There are various approximate treatments[1-3] that allow a calculation of short-range order parameters and the most recent treatment being that of Clapp and Moss.[4-6] These statistical theories have been very successful in predicting the diffuse scattering data obtained from short-range ordered alloys in most system but they do not provide information regarding the structural arrangement of atoms in terms of possible presence of small ordered domains. On the other hand, the structural models view the short-range ordered state in terms of a distribution of tiny, highly ordered domains. First we will try to review briefly the various structural interpretations that have been given to explain the positions and intensities of the diffuse maxima in the short-range ordered alloys in different systems and then discuss some of the criticisms of structural models.

II. STRUCTURAL MODELS

The structural model that views short-range order in terms of a contiguous distribution of small, highly ordered microdomains is commonly referred to as the microdomain model. In stoichiometric alloys these ordered microdomains can occupy the entire volume of the sample and are separated from one another by antiphase domain boundaries or twin boundaries or other discontinuities. The structure of microdomains is usually the same as the long-range ordered structure below the critical temperature (Tc). The size of microdomains is usually considered to range from about one superlattice unit cell to several hundred angstroms. A somewhat different version of this model considers short-range ordered alloys as consisting of a very high density of small, highly ordered regions (or particles) embedded in a nearly random matrix. In fact, for many systems it has been suggested[7-10] that the short-range ordered state consists of a coherent two-phase mixture in which the ordered phase is limited to a small equilibrium particle size. This description of short-range order may be referred to as the particle model. In this case, also, the structure of the coherent ordered particles is usually the same as the long-range ordered structure. There is yet another variation of this model that has been suggested recently.[11] Electron diffraction results[11] and some recent theoretical calculations[12] suggest that the short-range ordered state may contain microdomains possessing not one but many different superstructures. For a particular alloy, the most frequently occurring microdomain may or may not correspond to the equilibrium long-range ordered structure of that alloy. This description will be referred to as the multiple microdomain model.

The information about the presence of domains or particles in the short-range ordered state comes mostly from X-ray, electron and neutron diffraction, transmission electron microscopy and field ion microscopy. We will now review briefly how the information obtained from the above mentioned techniques can be used for structural interpretation of the short-range order state.

1. Structural Models Based on X-Ray Diffraction Data

X-ray diffraction gives quantitative data on the diffuse scattering intensities, from which the short-range order parameters can be calculated. Now one tries to develop a model to calculate the short-range order parameters and tries to match them with the experimentally determined values. There are many ways to proceed and some important ones will be mentioned here.

Moss[13] developed an antiphase domain model in which one starts with the ordered material and slices it up into domains of increasingly smaller size until one gets an agreement with the experimental short-range order results. With this model he was able to account for the disk-shaped diffuse peaks observed in Cu_3Au[14] and the experimentally measured[15] short-range order parameters. The structural picture that emerges from this is the microdomain model described earlier.

Gehlen and Cohen[16] developed a computer simulation method where atoms were selected at random and interchanged. The interchange between pairs of sites is guided by comparing calculated short-range order parameters with those determined by experiments. The interchanging procedure is allowed to continue until the two agree. Using the experimental short-range order parameters for Cu_3Au[13] above T_c, the final atomic arrangement obtained by this method showed very small, highly ordered

regions to be present in a nearly random matrix. The structure of short-range ordered Cu_3Au that emerges from this model is similar to the particle model described earlier. More recently, Lin et al.[17] have used a similar computer simulation technique to interpret the diffuse scattering results in short-range ordered Au-40 at % Pd alloy. This alloy is fcc when disordered and shows peculiar short-range order diffuse peaks that are split into two components such as those at positions close to (1,3/8,0) and (1,5/8,0) in reciprocal space. This is not commensurate with the superlattice peak positions of any known structure. The computer simulation results indicate a tendency towards the formation of a long period superlattice structure of $L1_0$(M=1) type, which has never been observed before.

Greenholz and Kidron[18] also developed a diffraction model and tried to match the calculated short-range order parameters with the experimental data on CuAu[19] and Cu_3Au.[15] For data on CuAu[19] quenched from above T_c and also for Cu_3Au data taken above T_c,[15] the model showed the presence of ordered domains in a disordered matrix. The mean size of the ordered domains for CuAu were about (3×3×3) unit cells and they occupied about one-third of the volume of the crystal. For Cu_3Au they found 60% of the ordered volume to be made of domains with a size of (1×1×1) unit cells, and about 34% were domains with a size of (3×3×3) unit cells.

It may be noted that all the structural models of short-range ordered alloys described above predict the presence of microdomains of various sizes with different volume fractions, but all the microdomains in a particular alloy have the same ordered structure, namely the long-range ordered structure observed below T_c in that alloy. No microdomain

with structure other than the long-range ordered structure of the alloy is predicted from the above models. More recently, Clapp[12] has used a Probability Variation Method (PVM)[20] to determine the complete frequency distribution of nearest neighbor configurations (clusters) for a number of cubic alloys from their experimentally determined short range order parameters. He used the work "cluster" in the general sense of the term to mean a group of atoms A and B arranged in some ordered fashion, and is not much different from a microdomain that is only one unit cell in size. He calculated the "enhancement factor," defined as the ratio of a cluster's population in the short range ordered state to its population in an ideal random alloy of the same composition, and also the percentage of atoms in the lattice of a given kind (say A or B), that have that particular cluster configuration. His calculations showed that the most enhanced configuration relative to the random state often corresponded to the perfectly ordered state, although its volume fraction may be very small. There were also departures from this simple expectation, although it is not clear how many cases (if any) result from data limitation. For Cu_3Au alloy above T_c the most enhanced cluster is Cu_3Au type and the next most enhanced cluster has the DO_{22} structure. His calculations on Au_3Cu ($L1_2$ structure) show the rather surprising result that the most enhanced clusters are the $L1_0$ (CuAu) type and the fourth most enhanced clusters are the Cu_3Au type. The calculations on Au-40 at % Pd alloy suggest that $L1_0$ (M=1) may not be the only structure as the computer simulation experiments[17] seemed to indicate, but $L1_0$ (M=2) structure is just as likely to be present. These calculations imply that the short-range ordered state may consist

of a distribution of ordered microdomains possessing different but closely related structures and the most frequently occurring microdomain may or may not correspond to the equilibrium long-range ordered structure of the alloy. The structural model of the short-range ordered state that emerges from this model is similar to the multiple microdomain model referred to earlier.

2. Structural Interpretation of Electron Diffraction Data

The diffuse scattering intensity measurements by X-ray diffraction are very laborious and time-consuming, but they give valuable quantitative data which can be modeled to obtain structural information, as described in the previous section. The short-range order diffuse scattering information from electron diffraction is relatively easier to obtain but it is very qualitative in nature. Absolute intensity measurements in electron diffraction patterns are difficult, if not impossible to obtain. However, qualitative electron diffraction has revealed important information about the short-range order state that is difficult to resolve by X-rays. For example, the presence of weak splitting in the diffuse scattering from disordered Cu_3Au was first observed in electron[21-23] rather than by X-ray diffraction. The earlier X-ray work by Cowley[14] and by Moss[15] did not reveal this splitting, but was later confirmed[13] by using a high resolution X-ray rotation photographic method. Such splittings were also observed in disordered CuAu in the high temperature electron diffraction patterns taken up to 50°C above the transition temperature,[24] but the previous X-ray results[19] failed to detect them. These split maxima in CuAu were qualitatively interpreted[24] as short chains of antiphase domains with

degenerate CuAu(II) like structure being present in the short-range ordered state above T_c. Watanabe and Fisher[25] also observed the splitting of diffuse peaks in the electron diffraction patterns of $CuAu_3$ taken above T_c, but this was not observed in the X-ray work of Batterman.[26] This splitting was taken as evidence for some form of antiphase relationship (of the same two-dimensional character as the ordered state) being present in the short-range ordered state above T_c.

A major limitation of the above qualitative interpretation of electron diffraction results is that no quantitative information about the size, shape and distribution of the microdomains can be predicted easily. All the structural descriptions of the short-range ordered state in copper-gold alloys described above are in qualitative agreement with most of the structural models described earlier in Section 1 with the exception of Clapp's model[12] based on PVM calculations. The electron diffraction studies on copper-gold alloys described above do not suggest a multiple microdomain configuration that one would expect from PVM calculations. This raises the question: do such microdomains with different superstructures indeed exist in short-range ordered alloys? Some recent electron diffraction work by Okamoto and Thomas[27] and by Das et al.[11] on a number of alloy systems suggests that multiple microdomain configurations may in fact exist in alloys above T_c and in those quenched from above T_c. These results provide qualitative support to Clapp's theoretical calculations[12] and will be reviewed in some detail.

In the qualitative interpretation of electron diffraction results described above, the diffuse short-range ordered maxima are considered

to be broad superlattice reflections, the broadening being essentially a small particle-size effect. This implies that the positions of the short-range ordered maxima in reciprocal space should always coincide with those of the superlattice reflections. Difficulties arise when the diffuse scattering maxima are observed at positions which do not correspond to the positions of the superlattice reflections of the ordered phase below T_c. Examples of such systems are Ni_4Mo,[11,27,28] Ni_3Mo,[11,29] Ni-10wt%W,[30] Ni_4W,[31] Au_4Cr,[11] Au_3Cr,[11,32] Au_4Mn,[11] Au_3Mn,[32] Au_4V,[11] Au-40 at % Pd,[17] certain titanium oxides[33] and possibly some vanadium and niobium carbides.[34] The system that is particularly interesting is Ni-Mo and has drawn considerable attention in recent years. In the Ni-Mo alloys diffraction patterns of the α-phase in the short-range ordered state exhibit diffuse peaks at all equivalent $\{1\frac{1}{2}0\}$ positions in the fcc reciprocal lattice over a wide range of compositions (8-33 at % Mo).[11,27-29] Within this composition range several ordered structures have been observed.[35] In addition to the Ni_4Mo and Ni_3Mo phases, a metastable phase Ni_2Mo possessing a Pt_2Mo superlattice has been observed.[29,36] None of these ordered phases produce superlattice reflections at the $\{1\frac{1}{2}0\}$ positions. The other systems such as Ni-W, Au-Cr, Au-V and Au-Mn also exhibit similar phenomena. It is obvious that the microdomain or the particle model which requires the presence of ordered domains of the same structure as that of the ordered phase below the critical temperature cannot explain the short-range order diffuse scattering in these alloys.

It was first discovered by Okamoto and Thomas[27] that Ni_4Mo samples quenched from above T_c contained very weak scattering near superlattice

positions corresponding to the ordered Ni_4Mo structure. This had not been observed before in the X-ray work of Spruiell and Stansbury.[28] More recently, Das et al.[11] reported the presence of diffuse scattering near Ni_2Mo superlattice positions in addition to those at Ni_4Mo positions in both Ni_4Mo and Ni_3Mo specimens severely quenched from above Tc. Interestingly enough, no diffuse scattering was observed near superlattice positions corresponding to an ordered Ni_3Mo structure in the Ni_3Mo specimen. It may be emphasized that the detection of such weak spots depends critically on the examination of proper reciprocal lattice sections. For example, Fig. 1a is a [001] diffraction pattern of a Ni_4Mo sample after quenching in iced brine from 1100°C. If one simply looks at the [001] diffraction pattern, it is not possible to tell whether there is any diffuse scattering present near Ni_4Mo and Ni_2Mo superlattice positions, because the diffuse peaks near $\{1\frac{1}{2}0\}$ positions dominate. However, a plot of isointensity contour maps indicates the presence of the diffuse scattering present near the Ni_4Mo and Ni_2Mo superlattice positions (marked by filled circles and filled triangles respectively in Fig. 1b). For the detection of these weak superlattice reflections one needs to go to certain favorable orientations where only the superlattice reflections lie on the Ewald sphere. The [110] orientation is very favorable for detecting Ni_2Mo spots and $[\bar{1}30]$ is suitable for detecting Ni_4Mo spots. Firgure 2 shows an example of a [110] diffraction pattern taken from a Ni_3Mo sample after quenching from 1270°C in iced brine. The diffuse scattering near Ni_2Mo superlattice positions (marked by arrows) can be clearly seen. There are no Ni_4Mo or $\{1\frac{1}{2}0\}$ reflections present in this orientation. A qualitative interpretation of the diffuse

scattering observed near Ni_2Mo and Ni_4Mo positions will be that there are microdomains present in the short-range ordered state with Ni_2Mo and Ni_4Mo structures. The presence of diffuse $\{1\frac{1}{2}0\}$ peaks in short-range ordered Ni-Mo alloys have been interpreted[27] to be due to the presence of imperfectly ordered domains with DO_{22} superstructure, resulting from the occurrence of non-conservative antiphase boundaries within the microdomains. These qualitative observations imply that the short-range ordered Ni-Mo alloys (in the composition range 8-25 at % Mo) contain microdomains with not one, but three different superstructures, namely DO_{22}, Ni_4Mo and Ni_2Mo. These results lend support to the multiple microdomain model described earlier.

It has been shown[27,36] that the three structure DO_{22}, Ni_4Mo and Ni_2Mo are very closely related to each other and starting with any one of these, the two others can be derived simply by introducing a periodic distribution of $\{420\}\frac{1}{2}\langle\bar{1}10\rangle$ type antiphase boundaries. In the short-range ordered state above T_c, if diffusion is fast enough, tiny regions with these various types of closely related ordered structures may be continuously forming and disappearing in order to establish an equilibrium distribution. Statistically there exists a finite probability for some other closely related structures to exist in the short-range ordered Ni-Mo alloys, but their volume fraction may be too small to give rise to any appreciable diffraction effect. At a particular stoichiometry the entire sample may consist of such imperfectly ordered microdomains with various volume fractions, as is predicted by PVM calculations[12] for many alloys.

The question is often raised as to whether the quenched alloys represent the short-range ordered state above Tc or not. If quenching is fast enough, the high temperature distribution of various types of microdomains may be essentially retained, particularly if the ordering kinetics are slow. However, depending on the quenching rate, the degree of order within these microdomains may be slightly enhanced, which is reflected in the diffuse scattering patterns. Figures 3a and 3b compare [001] electron diffraction patterns taken from a sample quenched from above T_c and from a sample held at a temperature above T_c. The two patterns are essentially the same and exhibit diffuse scattering near $\{1\frac{1}{2}0\}$ positions of the fcc reciprocal lattice, but the intensities of the $\{1\frac{1}{2}0\}$ spots are much greater in the quenched sample (Fig. 3a) than in the sample held above T_c (Fig. 3b). Quenching from above T_c retains the short-range order state fairly well in this alloy. In thin films the equilibrium long-range order structure of Au_3Cr is isotypic with Ni_4Mo (D1a superstructure).[32] Examination of high temperature electron diffraction patterns from various reciprocal lattice sections has established[11] that there is weak scattering present near the D1a superlattice positions in the short-range ordered state. Thus the short-range ordered Au_3Cr alloy may contain predominantly microdomains of imperfectly ordered DO_{22} and D1a structures.

The electron diffraction results described above have given considerable new information about the short-range order state in Ni-Mo and Au-Cr alloys and suggest multiple microdomain configurations to be present in these alloys. However, there are no three-dimensional short-range order parameters available for these systems to perform PVM calculations to independently check if such microdomains are

predicted or not. Three-dimensional short-range order parameters are badly needed for these alloys. In any case, the qualitative data from electron diffraction and the PVM calculations for various alloys[12] suggest that multiple microdomain model may be a better structural description of the short-range order state in most alloys.

III. DIRECT OBSERVATION OF MICRODOMAINS

From the discussion so far it is clear that the structural information about the presence of microdomains in the short-range order state is obtained in an indirect way from the X-ray and electron diffraction data. The direct evidence for microdomains can be obtained from dark field transmission electron micrographs of the diffuse short-range order spots. Dark field images of diffuse {100} spots in equiatomic Pt-Co alloy, severely quenched from above T_c, show[37] microdomains with average diameter of 20-40Å. Evidence for microdomains in Ni_4Mo[38] and in Ni_3Mo[11] has been obtained from dark field images of $\{1\frac{1}{2}0\}$ spots. These dark field electron micrographs of diffuse spots exhibit bright spots in images which are interpreted as microdomains. Recently, Cowley[39] has pointed out that without a careful analysis of the imaging conditions such interpretations may be invalid. He showed that under certain imaging conditions such spotty contrast can be obtained even for a completely random arrangement of atoms. Hence, care must be exercised in the interpretation of such dark field images. Further direct evidence of the presence of microdomains in short-range ordered alloys has come from field ion microscope images. Southworth and Ralph[40] observed tiny, highly ordered domains embedded in a random matrix in Pt-Co alloys quenched from above T_c. Similar ordered regions have also been observed[27] in the field ion microscope images of Ni_4Mo quenched from above T_c. These results provide direct evidence of microdomains at least in alloys quenched from above T_c. To date, direct evidence of microdomains in samples held at temperatures above T_c has never been reported. Attempts to obtain dark field images of

diffuse spots in samples held at temperatures above T_c have largely been unsuccessful. At high temperature excessive thermal diffuse scattering becomes important, and also the degree of order within the microdomains may be so small that no satisfactory images are obtained. Recently attempts are being made at Berkeley using the high voltage microscope to image the ordered domains in copper-gold alloys by direct lattice imaging techniques. These experiments may provide direct evidence of microdomains above T_c.

IV. CRITICISMS OF STRUCTURAL MODELS

One of the major criticisms of the structural models in the past had been that it could not explain the position of the short-range order diffuse maxima in alloys, where the diffuse maxima were non-coincident with the superlattice positions. This is because the microdomain model or the particle model tried to interpret the diffuse maxima as small particle-size broadening of the superlattice reflection. This difficulty has been overcome by the recent multiple microdomain model described in this paper. Recently it has been shown[41] that the splitting of the diffuse peaks in Cu_3Au and $CuAu_3$, which had been previously interpreted as direct evidence for the existence of antiphase microdomains above T_c, can now be directly related to the shape of the Fermi surface, in particular to the flat regions of the surface. Also recent studies[42] on Cu_3Au alloys suggest that there may be no direct relation between the short-range order state above T_c and the antiphase domain structure below T_c. Some of the earlier results on Cu-Al alloys[7] have now been re-interpreted[43] with the halp of Fermiology. Some of the recent electron diffraction results[33] on short-range ordered titanium oxides ($TiO_{0.9}$ to $TiO_{1.25}$) show bands of diffuse scattering which can be correlated with the Fermi surface of TiO. Whether any structural model can account for the bands of diffuse scattering observed in these titanium oxides and in some transition metal carbides[34] remains to be seen. The direct evidence for microdomains in these systems has yet to be shown.

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FIGURE CAPTIONS

Fig. 1. (a) [001] electron diffraction pattern of Ni_4Mo quenched in iced brine from 1100°C. (b) Iso-intensity contour map of [001] electron diffraction pattern of quenched Ni_4Mo. The dotted line outlines the portion traced. The notations are: ● Ni_4Mo superlattice positions; ▲ Ni_2Mo superlattice positions.[11]

Fig. 2. [110] electron diffraction of Ni_3Mo after quenching in iced brine from 1270°C.[11]

Fig. 3. [001] electron diffraction patterns of Au_3Cr taken (a) from a foil quenched from above T_c; (b) from a foil at 330°±10°C.

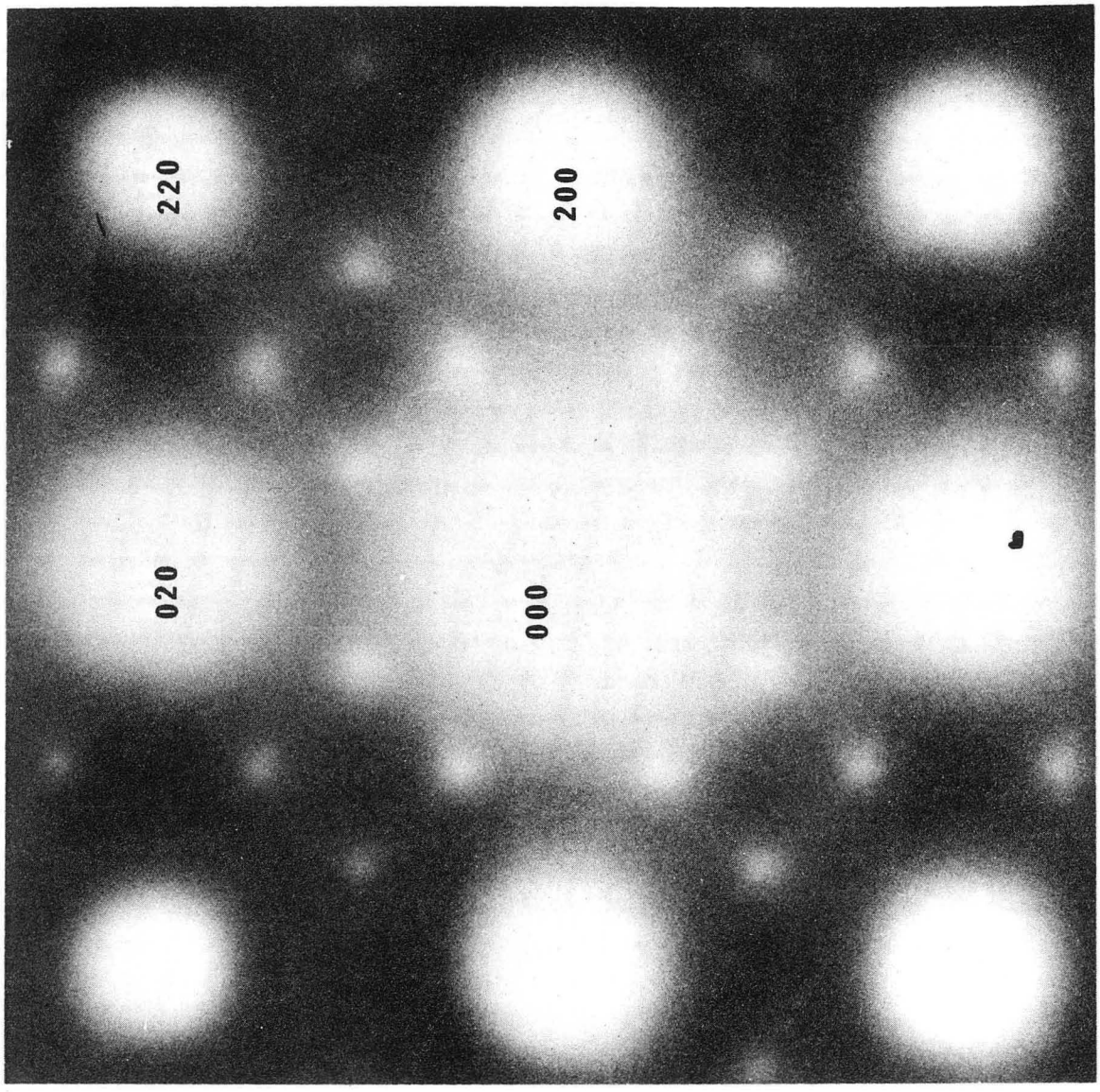

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Fig. 1a

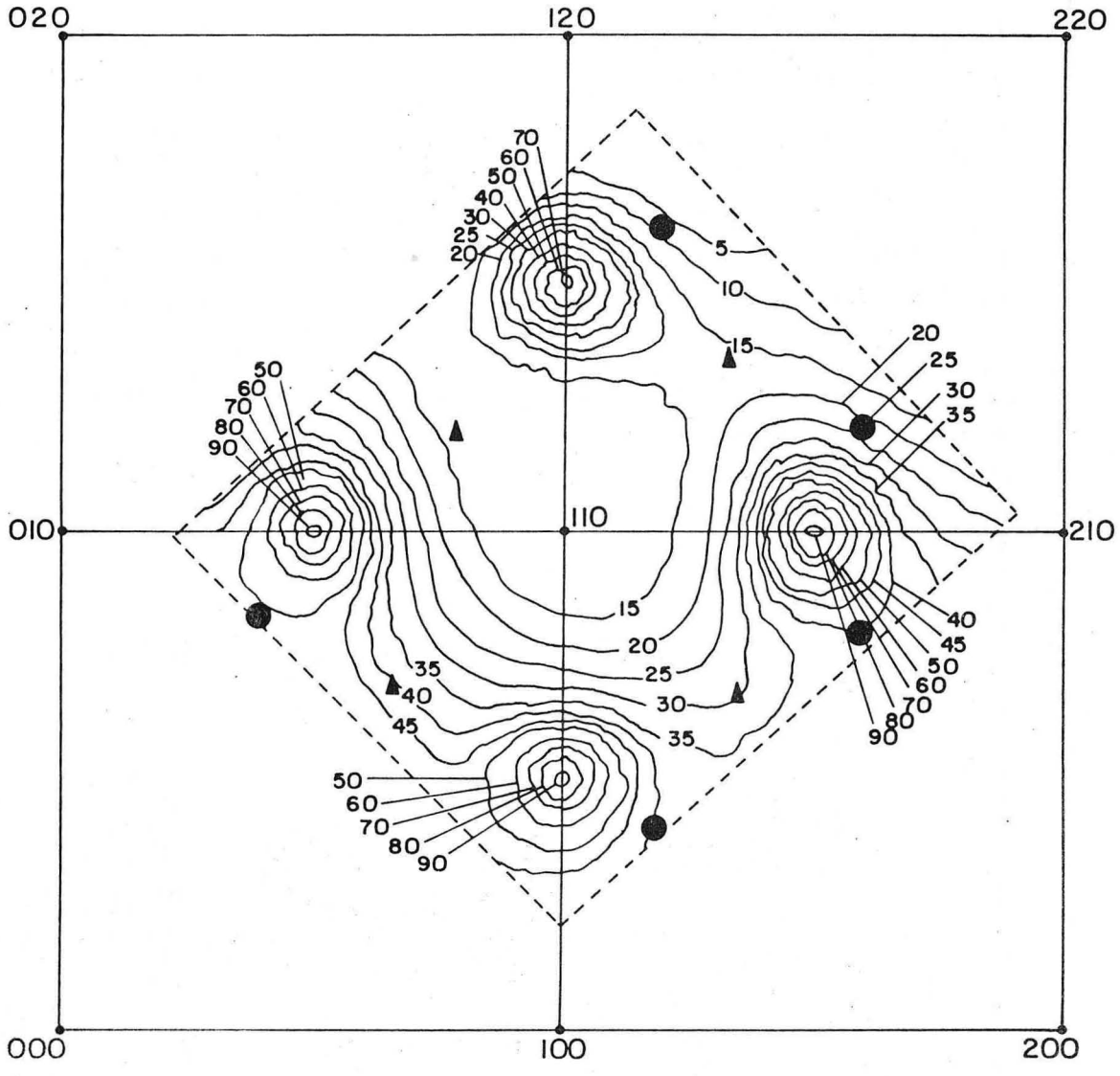
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Fig. 1b

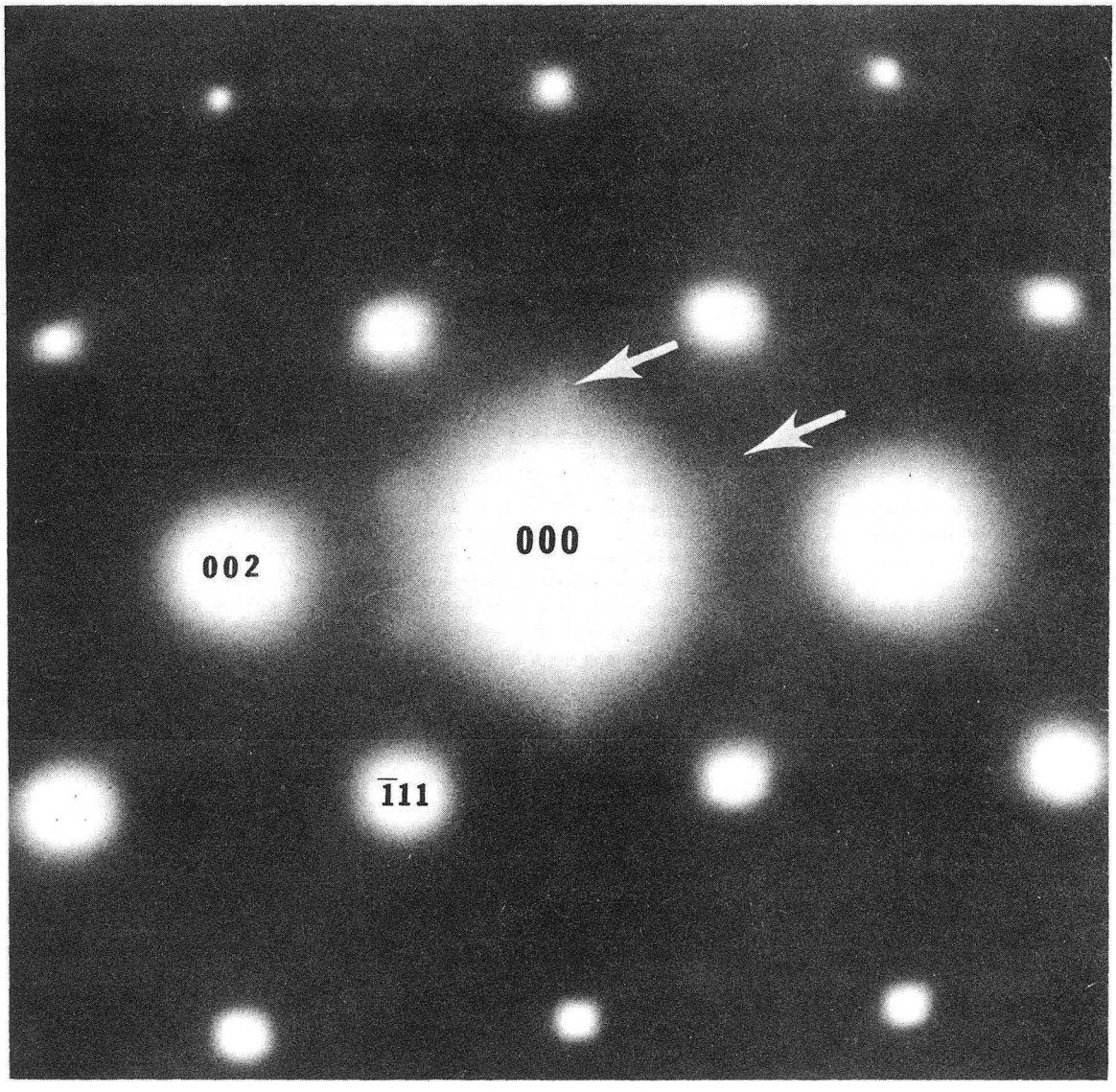

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Fig. 2

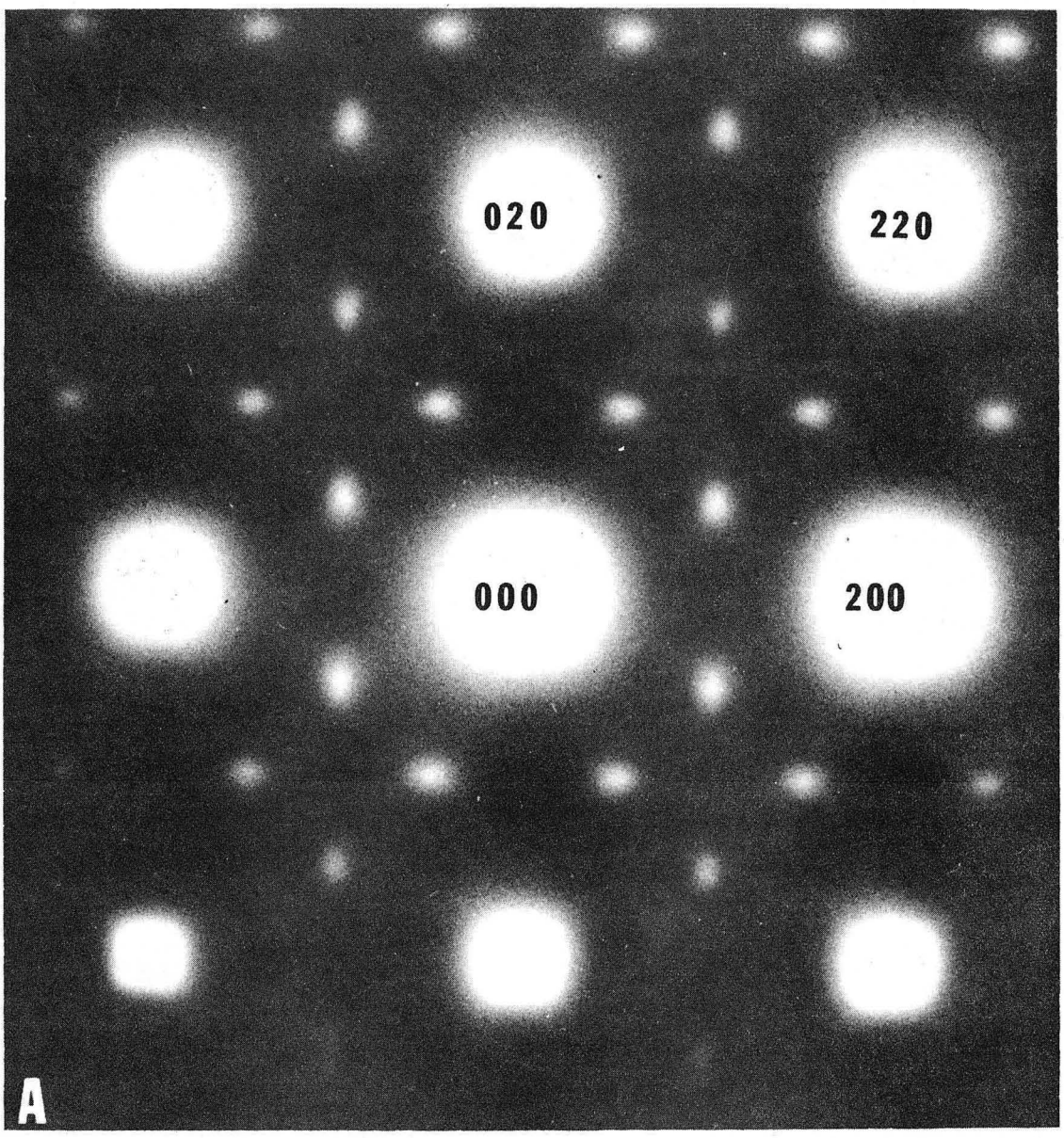

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Fig. 3a

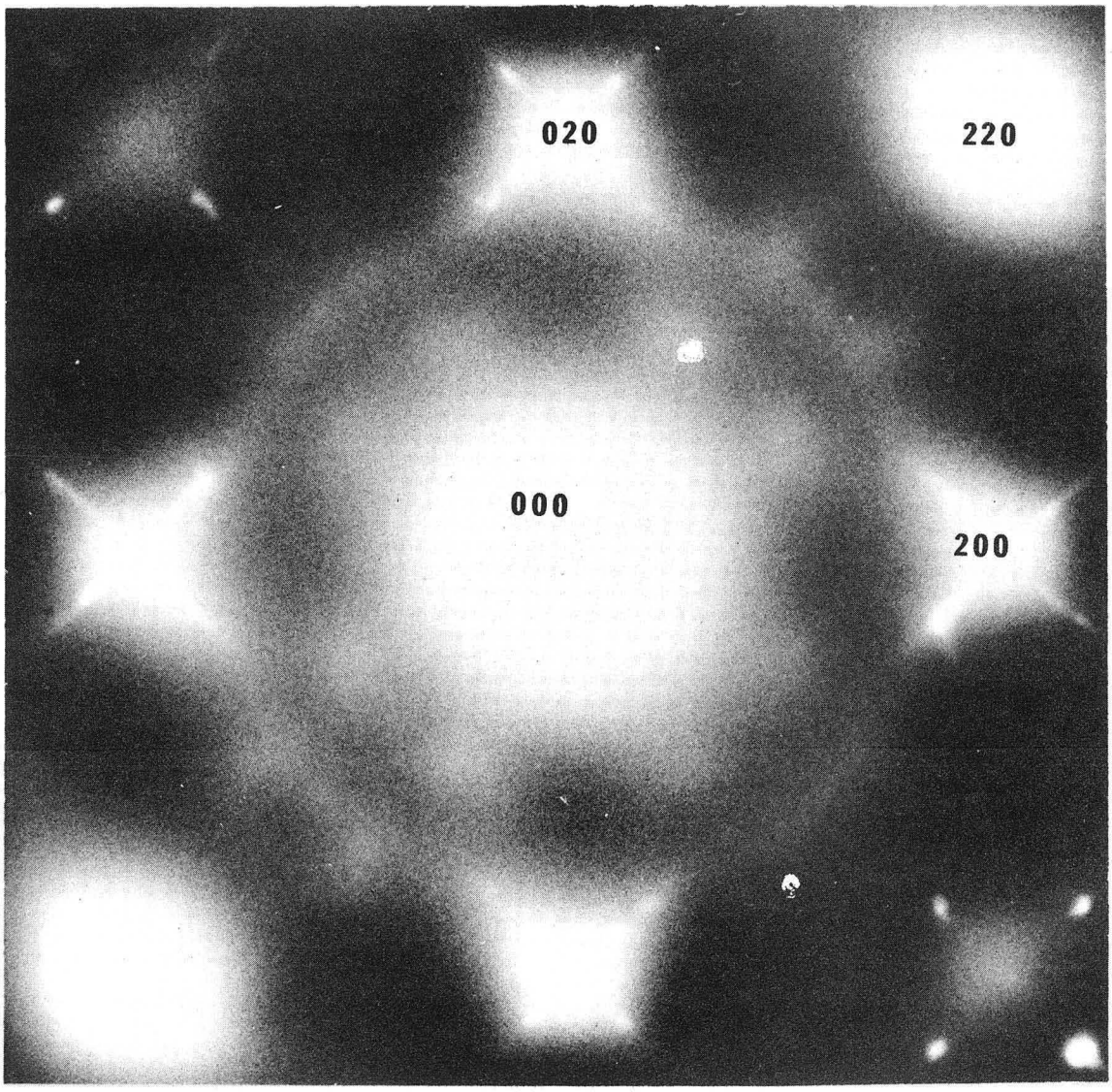

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Fig. 3b

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