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for

Effect of Annealing on the Substructure
of
Cold Worked fcc Metals and Alloys

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I. INTRODUCTION

The aim of this research is to obtain information concerning the relation between the structure and properties of metals and alloys. This is being accomplished primarily through study of the effect of annealing on the substructure of cold-worked metals and alloys. The observed changes in substructure as shown by x-ray diffraction and transmission electron microscopy are then related to physical properties. Materials currently being studied include α -Cu-Ge solid solutions, and the ordering alloys Cu_3Au , Ni_3Al , and Pt_3Co .

The experimental approach of the research on the solid solution alloys is to study as quantitatively as possible the changes in substructure that occur during annealing. This provides information about the processes by which the defects introduced by plastic deformation are rearranged and/or eliminated. In addition, because the various defects disappear and become rearranged at different rates, simultaneous measurements of physical properties may provide a means of sorting out the contributions of specific defects and arrangements of defects to the physical properties. There have been only a very limited number of combined structure and property studies, and in most of these the structure portion of the work has been qualitative. The understanding of data on physical properties will not be complete until the contribution of various defects to the properties have been better defined.

The study of the annealing behavior of the ordering alloys

makes use of the fact that both the x-ray and electron diffraction patterns from an ordered alloy contain information about the locations of the atom species. This information is not available with random solid solutions and allows one to begin with a well-defined atom arrangement and observe changes in the arrangement caused by plastic deformation and/or heat treatment. In addition, the behavior of the ordering alloys themselves is of considerable interest because they have structure-sensitive properties that can be controlled by heat treatment and they are the basis of many important commercial alloys.

II. RESEARCH STATUS

A. Annealing Behavior of Cu-base Solid Solutions

This part of the research has been concerned primarily with the study of the changes in the substructure during annealing of α -Cu-Ge solid solution alloys. A thorough diffraction study of the annealing of filings of Cu-8.5 at. % Ge has been completed.¹ Additional work on the composition dependence of the annealing behavior has also been carried out² and is being assembled for publication. During the past year, work has been initiated on the annealing behavior of solid samples of the same alloys. The solid samples will allow measurements of properties such as hardness, electrical resistivity, etc. to be made along with the studies of substructure. As noted previously,² the basic information obtained from the studies on the filings will provide a sound basis for the work on the solid samples which have certain experimental disadvantages for studies

of structure.

The Cu-Ge alloy to be used in the study was prepared by Engelhard Industries. Approximately 400 grams of a mixture of Cu with nominally 9.75 wt. pct. Ge was melted in a graphite crucible and poured into a graphite mold. Both elements were initially 99.999% pure. Melting was done under vacuum and the chamber was backfilled with helium while pouring into the mold. The ingot was then rolled approximately 50 pct. R.A. and samples were cut from both sides for chemical analysis. Results of chemical analysis showed 9.8 wt. pct. Ge (8.6 at. pct. Ge) with 0.001 wt. pct. Pt the principal impurity.

The ingot as received from Engelhard was homogenized in an argon atmosphere by heating at 600°C for one day, 625°C for one day, 725°C for two days, 800°C for two days, and 825°C for five days followed by furnace cooling to room temperature. The material was then cross-rolled to a thickness of 0.045 in. in steps of 50 pct. R.A. with intermediate anneals at 375°C for one hour. The resulting material had a fine grain size with very little texture as required for good diffraction analysis.

Nine 3 x 1 x 0.045 in. specimens of the alloy were machined from the as-rolled sheet and lapped flat with one micron Alundum powder. These specimens were then given a final annealing treatment and then subjected to explosive loading at pressures of 100, 180, and 350 kbars at the Eastern Laboratories of E. I. duPont deNemours and Co. Explosive loading was chosen because it provides a means of achieving a fairly large amount of deformation with no large dimension changes and with little

grain rotation. The latter point is particularly important because the presence of large amounts of texture interferes with structural studies as well as influencing property measurements. Consideration is being given to studying samples subjected to deformation by rolling once the work on the shock loaded samples has been completed.

A special apparatus has been assembled to make precise measurements of the changes in the electrical resistivity of the specimens during annealing. The basic unit is a Leeds and Northrup Precision Kelvin Bridge and Ratio Box purchased with AEC and University funds. This unit is coupled to a D-C Null Detector giving a system which can make measurements in the range of 0.01 microhm to 1.0 ohm with a readability to any four significant figures. The entire apparatus is housed in an aluminum box to shield against stray field current effects. The specimens on which measurements will be made have resistances of $\sim 1,000$ microhms. The sensitivity of the equipment is less than 0.01 pct. with a current of 0.3 amp. Under balanced conditions no current flows through the specimen so that the complication of Joule heating is minimized. In addition, the measurements made with the system are independent of lead and contact resistances. Construction of a special specimen holder has made it possible to reproduce measurements to better than 0.03 pct.

The specimens have just been returned from duPont in the explosively loaded condition and we have begun making measurements.

B. Annealing Behavior of Cu_3Au

In this portion of the research we are studying the annealing behavior of the classical ordering alloy Cu_3Au . Our initial work characterized the process of ordering and antiphase domain growth in a quenched powder of the alloy.³ Subsequent studies established the effect of initial cold work on the same processes.⁴ During the past year we have studied the kinetics of antiphase domain growth and the details of the antiphase domain configuration by means of transmission electron microscopy. This has provided some new information and also allowed a quantitative comparison to be made with the x-ray data. (Very few comparisons of this type can be found in the literature.)

The work on the kinetics of domain growth has been completed and prepared for publication. The results of the earlier x-ray work³ were confirmed. That is, the domains were found to follow a $D^2 = t$ relation during growth and the activation energy for the process was found to be 42 kcal/mole vs. 44 kcal/mole from the x-ray measurements. There was no deviation from the $D^2 = t$ relation at domain sizes as large as 2700 Å. Changing the quenching temperature from 450° to 600°C had no effect on subsequent domain growth at 350°C. Considering the general behavior found for precipitation processes this is not unusual. It might be expected that changes in the quenching rate would have an effect on the domain growth.

Determination of the size distributions of the growing domains showed that certain sizes are preferred giving rise to a periodicity in the size distributions. This was found for

annealing at both 350° and 375°C. Analysis of the positions of the peaks in the distributions indicated that a unit domain size of about 20-25 Å forms the basic pattern for domain growth. It is felt that the as-quenched alloy forms a very fine, uniform, contiguous domain structure very rapidly on heating in the range 300°C to T_c . The domain size associated with this structure is approximately 20-25 Å. Subsequent domain growth then occurs by a process in which neighboring domains are combined to form new domains with sizes which are multiples of the unit domain size. Because of the manner in which the process takes place and the behavior of the size distributions during growth, there appears to be a close connection between the early stages of ordering and the early stages of precipitation. It has been shown theoretically that many characteristics of ordering can be treated as the inverse of those for spinodal decomposition.^{6,7}

As shown by Fisher and Marcinkowski,⁸ the antiphase domain configuration in Cu_3Au forms a so-called "maze pattern." If the APDB were all {100} Type I as expected from energy considerations,⁹ the APDB would form a grating. Fisher and Marcinkowski⁸ explained the "maze pattern" on the basis of the presence of appreciable amounts of {100} Type II APDB. Under these conditions certain lines of the grating disappear because they cannot form contrast and the "maze pattern" is formed.

Yamaguchi et al.¹⁰ explained the "maze pattern" with a combination of dislocations and {100} Type I APDB. Because the Burgers vector of the dislocations is equal to the antiphase displacement vector, dislocations can remove, replace, or change

APDB. They estimated the dislocation density required to form the "maze pattern" to be $5 \times 10^{10}/\text{cm}^2$ for Cu_3Au annealed at 330°C for 32 hrs. They concluded that this was a reasonable value for their alloy which was prepared by evaporation, but they did not measure the dislocation density.

We have used the model of Yamaguchi et al. to estimate the dislocation densities required to explain the "maze patterns" that we have observed. The values we found (which are, of course, dependent on the domain size) are in range 10^{10} - $10^{11}/\text{cm}^2$. This is in good agreement with Yamaguchi et al.; however, when we measured the actual dislocation densities for several specimens, we found them to be in the range 10^8 - $10^9/\text{cm}^2$. This difference is too large to be accounted for by invisible dislocations, etc., so that we have concluded that the "maze pattern" cannot be explained by dislocations alone. Also, because evidence for appreciable amounts of $\{100\}$ Type II APDB has not been found by electron microscopy or x-ray diffraction,³ we feel that $\{100\}$ Type II APDB is not a primary contributor to the formation of the "maze pattern."

We have looked at the antiphase domain structure in a general way and have concluded that there is another phenomenon which contributes to the formation of the "maze pattern." A brief summary of our considerations will be given here.

An APDB in the ordered alloy is defined as the surface between two neighboring ordered regions having a mutual displacement equal to the antiphase vector. First, consider an arbitrary displacement between a region A within a region B as

shown in Fig. 1a. Assume the antiphase displacement of A with respect to B across the surface a between A and B is given by $\vec{v}_a$. By definition, this vector is conserved along the surface a. Then, if the surface divides into two surfaces b and c with displacement vectors $\vec{v}_b$ and $\vec{v}_c$ along a line l, as shown in Fig. 1b, a new region C between b and c will be formed. Because the displacement between points A₁ and B₁ must be the same as that between A₂ and B₂ (i.e., the displacement vector is conserved), we can write:

$$\vec{v}_a = \vec{v}_b + \vec{v}_c \quad (1)$$

We can also take the line l as the line of intersection of boundaries as shown in Fig. 1c. In general, the sense of the displacement vector between any two regions is arbitrary. But, if in Fig. 1c we define $\vec{v}_a$ as the displacement vector of region A relative to fixed region C, the sense of the remaining displacements $\vec{v}_b$ (B relative to A) and $\vec{v}_c$ will be defined. Then the total displacement around l can be written:

$$\vec{v}_a + \vec{v}_b + \vec{v}_c = 0 \quad (2)$$

This is the same as Eq. (1) and can be extended to boundaries intersecting on the line l:

$$\sum_{i=1}^n \vec{v}_i = 0 \quad (3)$$

where $\vec{v}_i$ is the displacement vector for the i^{th} region relative to the $i-1^{\text{th}}$ region. This general expression must be satisfied by the domain configuration in the ordered alloy.

Consider the APDB configuration in Cu_3Au as seen in a 110 dark field image. For an APDB to be visible, $2\vec{R} \cdot \vec{g}$ (in the relation $\alpha = 2\vec{R} \cdot \vec{g}$) must be an odd integer. This means that with $\vec{g} = [110]$ the antiphase displacement vectors of the visible APDB will be one of $\pm \frac{a}{2} [101]$, $\pm \frac{a}{2} [\bar{1}01]$, $\pm \frac{a}{2} [011]$, or $\pm \frac{a}{2} [0\bar{1}1]$ and those of the invisible APDB will be $\pm \frac{a}{2} [110]$ or $\pm \frac{a}{2} [1\bar{1}0]$. We can divide these antiphase vectors into three components designated u , v , and w where the w components of the antiphase vectors of the invisible APDB are zero. Then for m visible APDB and n invisible APDB intersecting on a line we can write according to Eq. (3):

$$\sum_{i=1}^m u_i + \sum_{j=1}^n u_j' = 0$$

$$\sum_{i=1}^m v_i + \sum_{j=1}^n v_j' = 0$$

$$\sum_{i=1}^m w_i + \sum_{j=1}^n w_j' = 0$$

where u_i , v_i , and w_i are the u , v , w components of the displacement vector associated with the i^{th} visible APDB and u_j' , v_j' , and w_j' are those associated with the j^{th} invisible APDB. By definition $w_j' = 0$, so that:

$$\sum_{i=1}^m w_i = 0 \quad (4)$$

Now, since $w_i = \pm \frac{a}{2}$, Eq. (4) requires that m be an even number. That is, if the antiphase displacement is conserved (i.e., there are no dislocations, stacking faults, etc. present) the APDB

intersections must consist of even numbers of APDB. This is true for the image formed by any one supperlattice reflection.

Consider the two-dimensional APDB configuration shown schematically in Fig. 2a. The intersection A shown in Fig. 2b satisfies the requirement of an even number of boundaries and also satisfies Eq. (3) if the boundaries are all {100} Type I. For example, one can set $\vec{v}_a = \frac{a}{2} [101]$, $\vec{v}_b = \frac{a}{2} [011]$, $\vec{v}_c = \frac{a}{2} [\bar{1}0\bar{1}]$, and $\vec{v}_d = \frac{a}{2} [0\bar{1}\bar{1}]$.

The intersection B shown in Fig. 2c consists of an odd number of APDB, and therefore, the displacements are not conservative.

For an intersection of type C (involving APDB a and b) shown in Fig. 2d, if one of the APDB is a {100} Type I, the other must be a {100} Type II APDB. For example, if we let $\vec{v}_a = \frac{a}{2} [101]$ then $\vec{v}_b = -\vec{v}_a = \frac{a}{2} [\bar{1}0\bar{1}]$ which is a {100} Type II APDB. Similarly, if b is taken as a {100} Type I APDB, then a is a {100} Type II APDB. Therefore, with this type of intersection it may be necessary to introduce a dislocation to convert the Type II APDB into Type I APDB as was suggested by Yamaguchi et al.¹⁰ However, there is another configuration involving this intersection which should be considered. Assume that an invisible APDB c exists at the intersection. The anti-phase displacement $\vec{v}_c$ of the invisible boundary (shown as the dotted line in Fig. 2d) is $\pm \frac{a}{2} [110]$ or $\pm \frac{a}{2} [\bar{1}\bar{1}0]$. Taking $\vec{v}_a = \frac{a}{2} [101]$, {100} Type I, and $\vec{v}_c = \frac{a}{2} [\bar{1}\bar{1}0]$ then from Eq. (3):

$$\begin{aligned}\vec{v}_b &= -\vec{v}_a - \vec{v}_c \\ &= \frac{a}{2} [\bar{1}0\bar{1}] + \frac{a}{2} [1\bar{1}0] = \frac{a}{2} [0\bar{1}\bar{1}]\end{aligned}$$

Therefore, b can be a $\{100\}$ Type I APDB. If the invisible APDB c lies on $\{100\}$, it will be a $\{100\}$ Type II APDB and the result is similar to that proposed by Fisher and Marcinkowski.⁸ However, because the invisible APDB can reduce its energy by deviating from $\{100\}$, the presence of $\{100\}$ Type II seems unlikely. Also, the original two boundary intersection of type C may not be stable with a $\{100\}$ Type I and a $\{100\}$ Type II APDB because the energy can be reduced by changing the $\{100\}$ Type II APDB to another plane.

Experimental verification of the occurrence of the various possible APDB intersections is quite difficult. However, one can note that the x-ray diffraction data³ show the existence of appreciable amounts of APDB which is not $\{100\}$ Type I or $\{100\}$ Type II. The best fit to the x-ray data is achieved with a model in which $\{100\}$ Type I APDB is combined with either $\{111\}$ shear type APDB or APDB with random orientation.

During the current work, images of $\{111\}$ APDB were obtained quite often. In Fig. 3, which is a micrograph of a specimen annealed 20 hrs. at 350°C , several points are marked where the APDB are parallel to the trace of $\{111\}$. (This is also the trace of $\{110\}$; however, the APDB are inclined to the foil surface so that they are most likely on $\{111\}$.)

Examples of the various types of APDB intersections can be seen in Figs. 4a and 4b. Figure 4a is a 110 dark field image of a specimen annealed at 350°C for 50 hrs., while Fig. 4b is the same area shown as a $\bar{1}02$ dark field image. There

are several instances where an intersection appears to involve only two APDB in one image, but is shown by the other image to consist of more than two APDB. For example, see A, B, C, D, and E in Fig. 4a or F and G in Fig. 4b.

From these considerations we feel that invisible non-{100} APDB make an important contribution to the observed "maze pattern." Although dislocations and {100} Type II APDB also contribute to the formation of the "maze," their effect is not as important.

It was generally observed that the regularity of the domain structure changed during domain growth. At small domain sizes the APDB appeared to be more straight and more nearly parallel to {100}, while at large sizes the APDB were not as straight and the amount of non-{100} APDB increased. This is in agreement with the x-ray observations³ where the relative amount of {100} Type I APDB was found to increase at domain sizes less than about $100 \text{ \AA}$ and then to decrease as the domains grew larger. Some possible explanations for this have been discussed previously;³ however, we have now considered one other aspect of the growth process which may be an important factor in determining the APDB configuration. It was suggested by Marcinkowski¹¹ that the elimination of the high energy {100} Type II APDB serves as the primary driving force for domain growth. This cannot be correct according to our conclusion that the amounts of APDB other than {100} Type I APDB increase during growth for sizes above $100 \text{ \AA}$. Consider an idealized domain configuration, as shown in Fig. 5a, where all of the

APDB are {100} Type I except those surrounding one domain which are {100} Type II. If that one domain grows by consuming neighboring domains, as shown in Figs. 5b and 5c, the amount of {100} Type II APDB actually increases. Of course, there are other configurations in which the growth can result in a decrease in the amount of {100} Type II APDB. For example, Fig. 6a shows a case where {100} Type II APDB is annihilated during growth. However, in the general case no matter how large the relative amount of {100} Type I APDB is initially, the process of growth by consumption of neighbors in a random manner will cause the relative amount of {100} Type II APDB to increase and approach two-thirds. As noted previously, the {100} Type II APDB will probably be replaced in the actual configuration by lower energy APDB.

The cause of the initial increase in the relative amount of {100} Type I when the domain size is less than $100 \text{ \AA}^3$ is not clear. However, as illustrated in Fig. 6b, it may result from the occurrence of a special arrangement of domains very early in the ordering process when the domains first become contiguous.

Other work on Cu_3Au included an analysis of the data on x-ray peak positions to look for additional evidence for {111} APDB. The analysis gave values for the {111} APDB probability only at very short annealing times; however, as noted above, additional evidence for {111} APDB was found by electron microscopy.

Measurements of the hardness changes during recrystal-

lization at temperatures just below and just above the critical temperature have just been started. The samples were deformed by rolling and the heat treating is being done in a salt bath to minimize the heat-up time. Preliminary results indicate that the temperature dependence of the annealing behavior around T_c is more uniform than shown by the data of Arunachalam and Cahn.¹² They made measurements for only two annealing temperatures and concluded that a sharp change in recrystallization behavior occurred at the critical temperature.

C. Other Efforts

X-ray diffraction measurements have been made on Ni_3Al cold worked by filing and then annealed. The stacking fault probability measured for the filings is 0.034 which is quite high compared to Cu_3Au .¹³ Whether this is simply a result of a lower stacking fault energy is not yet clear and will require more study. The faults annealed out rapidly on heating at $300^\circ C$ or higher for one hour.

Simultaneous measurements of the line broadening of the 100 and 110 superlattice peaks and 111 and 200 fundamental peaks were made for filed samples of Ni_3Al given one hour isochronal anneals at eight temperatures in the range of 300 to $1050^\circ C$. These showed that the broadening caused by cold work disappeared quite uniformly up to about $800^\circ C$. At that temperature an anomalous increase in the broadening of the 100 superlattice peak occurred. Although further study is necessary to establish this behavior, it is interesting to note

that a minimum in ductility occurs at about 800°C in Ni_3Al and in alloys containing precipitated Ni_3Al .¹⁴

The long-range order parameter was about 0.85 for all of the samples given the isochronal anneals. This indicates that the ordering process in the alloy may be more rapid than in Cu_3Au . The critical temperature for the alloy has not yet been established, but it is more than 1100°C .

Preliminary observations of the ordering behavior of the alloy Pt_3Co have been made. The data are not complete at this point so that no conclusions can be made. However, the ordering process is such that highly ordered samples can be produced quite readily. Attempts to electropolish thin foils from the alloy have been unsuccessful to date. Further work is planned for this alloy and the alloy Pt_3Fe .

The development of a computer program for analysis of x-ray data with the University's IBM 360 computer is in its final stages. When finished, the program will allow the raw x-ray data to be given directly from the diffractometer in the form of punched paper tape to the computer for analysis. The computer will plot the data for visual observation, perform the necessary corrections, carry out the analysis, and then report the results in tables and graphs. We are using some parts of the computer program developed by Prof. C. Wagner and his students at Yale University. The important innovations included in the program are:

- (a) correction of the intensity for the variation in the atomic scattering factor.
- (b) a subroutine for determination of the proper

background intensity for the entire diffraction pattern. This is done by making a least squares fit of a linear (or parabolic) function to the entire spectrum including the peaks. All data points more than one standard deviation above the line are then removed and the fit repeated. This procedure is repeated until the standard deviation of all points from the line is less than a predetermined value. At that point one final fit is made in which the poorest points below the line are removed. The results of this simple procedure are very encouraging.

- (c) a subroutine for unfolding neighboring diffraction peaks which overlap. There are instances with heavily cold worked materials or partially ordered alloys where the overlap of diffraction peaks is so great that it cannot be neglected. The overlap is particularly evident when the background has been precisely determined as described in (b) above. The routine determines the point at which each peak contributes one-half of the intensity above background and then fits a parabolic function from that point to the point at which the intensity goes to zero. This latter point is determined from the corresponding point on the other side of the peak (where there is no overlap) weighted by an asymmetry factor calculated at one-half peak height.
- (d) a subroutine for determining the precise peak position by fitting a parabola to the smoothed raw data at the top of the peak. This is an improvement of a routine included in Wagner's program

III. PERSONNEL

In addition to the principal investigator, two graduate research assistants, D. J. Borich and M. Sakai, have been engaged in research supported by this contract during the past year. A new research assistant, C. Rase, began his M.S. thesis research this summer.

IV. PRESENTATIONS

"Effect of Annealing on Cold-Worked Cu_3Au "

Presented by A. L. Ward at TMS-AIME Spring Meeting,
Pittsburgh, May, 1969.

D. E. Mikkola served as chairman of Session III of
TMS-AIME Symposium on Stacking Faults at TMS-AIME
Spring Meeting, Pittsburgh, May, 1969.

V. PUBLICATIONS

"Effect of Annealing on the Substructure of Cold-Worked
 $\text{Cu-8.5 at. \% Ge.}$ " W. G. Truckner and D. E. Mikkola
To be published in J. Appl. Phys., December, 1969.

"Antiphase Domains in Cu_3Au " M. Sakai and D. E. Mikkola
To be submitted for publication

"Ordering of Cold-Worked Cu_3Au " A. L. Ward and D. E.
Mikkola. In preparation.

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(a)

(c)

(b)

Fig. 1 General displacements between ordered regions.

Fig. 2 Two-dimensional antiphase domain configurations.

Fig. 3 110 dark field image of Cu_3Au quenched from 450°C and annealed at 350°C for 20 hrs. Marks indicate {111} APDB.

Fig. 4a 110 dark field image of Cu_3Au quenched from 450°C and annealed at 350°C for 50 hrs.

Fig. 4b $\bar{1}02$ dark field image of same area as Fig. 4a.

IV	II	IV	II	IV	II	IV
III	I	III	I	III	I	III
IV	II	IV	II	IV	II	IV
III	I	III	IV	III	I	III
IV	II	IV	II	IV	II	IV
III	I	III	I	III	I	III
IV	II	IV	II	IV	II	IV

(a)

IV	II	IV	II	IV	II	IV
III	I	III	I	III	I	III
IV	II				II	IV
III	I	IV			I	III
IV	II				II	IV
III	I	III	I	III	I	III
IV	II	IV	II	IV	II	IV

(b)

IV	II	IV	II	IV	II	IV
III	I	III		III	I	III
IV	II				II	IV
III			IV			III
IV	II				II	IV
III	I	III		III	I	III
IV	II	IV	II	IV	II	IV

(c)

Fig. 5 A model showing antiphase domain growth beginning with one domain surrounded by Type II APDB (heavy lines) in a matrix of domains having Type I APDB (light lines). I, II, III, and IV refer to Type I, II, III, and IV antiphase domains respectively.

Fig. 6 Models showing antiphase domain growth under circumstances where Type II APDB (heavy lines) is replaced by Type I APDB (light lines).