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August 1970

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Spinodally Decomposed Cu-Ni-Fe Alloys of Asymmetrical Compositions

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ABSTRACT

Two Cu-Ni-Fe alloys of asymmetrical compositions (i.e. with unequal volume fractions of the two low temperature phases) were heat treated to produce spinodal structures with various wavelengths or characteristic particle spacings. Transmission electron microscopy and diffraction were used to study the spinodally decomposed microstructures and to measure the wavelengths in the specimens. The change in composition of the Ni-Fe rich phase with aging time was determined by measuring the Curie temperature; and the results show that the decomposition kinetics for the asymmetrical alloys are an order of magnitude slower than for the Cu-Ni-Fe alloy of symmetrical composition. Results of mechanical testing, Curie temperature and wavelength measurements indicate that, while coherency is maintained, the yield stress is directly proportional to the difference in compositions of the two precipitating phases and is independent of the wavelength and the volume fractions.

1. INTRODUCTION

The theoretical aspects of spinodal decomposition are now well understood (see e.g. Cahn (1)). In elastically anisotropic materials, spinodal decomposition occurs along elastically "soft" directions; and for Cu-Ni-Fe alloys such directions are the cube directions. The resulting periodic structure produces side bands in diffraction patterns, and this effect has been studied extensively by x-ray investigators (2).

Most experimental studies of spinodal decomposition have utilized x-ray diffraction methods and there has been limited application of high resolution metallography. Neglecting grain boundaries, the microstructure of a spinodally decomposed alloy can be characterized by three parameters; amplitude, wavelength and volume fraction of the phases present. Butler and Thomas (3) studied a Cu-Ni-Fe alloy of symmetrical composition (i.e. at the center of the miscibility gap) and followed the changes in wavelength and amplitude as a function of aging time. They also related the changes in microstructure to the age-hardening response of the alloy. The objective of the present investigation was to determine experimentally the effect of the volume fraction of the phases on the transformation kinetics and on the age-hardening response of Cu-Ni-Fe alloys of asymmetrical composition, which were on the same tie-line as the alloy studied by Butler and Thomas (3) (see fig. 1 of ref. 3).

2. EXPERIMENTAL PROCEDURES

The experimental alloys were prepared using 99.999% purity Cu, 99.85% purity Ni and 99.6% purity Fe. The compositions of the two alloys, in atomic percent, were:

alloy 1, 32.0Cu-45.5Ni-22.5Fe;

alloy 2, 64Cu-27Ni-9Fe.

0.5% Mn was added to each melt to act as a deoxidizer to aid fabrication. The charges were melted in a large induction furnace in an helium atmosphere and were chill cast into copper molds to reduce segregation. X-ray fluorescent analysis verified to within $\pm 1\%$ the compositions given above. After homogenization, the ingots were hot forged and rolled at 950°C to a thickness of 40 mils. Small pieces from each ingot were then annealed and further cold rolled to 8 mils thickness for electron microscopy specimens.

The heat treating procedure used by Butler and Thomas was also followed in this study. After this heat treatment, the average grain sizes were 0.07 mm dia (alloy 1) and 0.12 mm dia (alloy 2). Then groups of specimens were aged at 625°C for 1 minute, 0.1, 1, 10, 100 and 1000 hours. For aging times less than one hour, a salt bath was used.

An Instron testing machine was used to measure the yield stress and ductility of flat tensile specimens which were 40 mils thick; and all specimens were pulled at a constant strain rate of 0.02 cm/min. The yield stress was measured at 0.2% strain offset, and the total elongation to fracture was determined by the change in spacing of two indentations made on the gage length. The work hardening rate (i.e. the slope of the stress-strain curve) was calculated at 2% strain

for all specimens tested. A scanning electron microscope was used for examining the fracture surfaces to determine the mode of fracture.

To determine the composition of the Ni-Fe rich phase, the Curie temperatures of the aged specimens were measured using apparatus developed for the study of high temperature magnetic phase changes (4). This apparatus is essentially a transformer in which the specimen is the core between the primary and secondary coils. An X-Y recorder was used to measure the change in induced voltage, which is a function of the magnetic permeability, as a function of temperature. Two heating and cooling cycles were run for each specimen, and the average value of the measured temperatures was taken as the Curie temperature. The specimens used for this measurement were 1" x 1/4" x 40 mils.

Thin foils for transmission electron microscopy were prepared using the procedure described by Butler and Thomas (3). The wavelengths (λ) or particle spacings of heat treated specimens were measured directly from enlarged prints of micrographs. All micrographs were taken in the $\langle 110 \rangle$ orientation with a strong 200 reflection operating. At least one hundred measurements, taken from five different areas, were made to determine the average wavelength for each aging time. Because a 15% magnification error can occur when using a double tilt specimen stage, a carbon replica of a ruled grating was used to calibrate the magnification for different objective lens currents at fixed settings of the intermediate and projector lenses. For specimens with $\lambda < 150\text{\AA}$, the sideband spacings on 400 reflections were also used to calculate λ as described previously (3).

3. EXPERIMENTAL RESULTS

The experimental results for the two alloys studied are summarized in the graphs shown in figures 1 and 2. For comparison, the results of Butler and Thomas⁽³⁾ for the Cu-Ni-Fe alloy of symmetrical composition aged at 625°C are given in figure 3. The compositions of the Ni-Fe rich phase were determined from the Curie temperature measurements using the information given in ref. 3. Distinct sideband peaks were not observed on the microdensitometer traces, but rather a general flattening of the main diffracted peak was observed to occur over some finite distance on the diffraction pattern (see fig. 4). Thus, the values of λ calculated from the sideband spacings represent some average wavelength in the microstructure. The wavelength values determined from the sidebands give a more accurate measurement of the actual wavelengths than the micrographs because a diffraction pattern represents a larger statistical sampling of the specimen than the one hundred measurements taken from micrographs. Also, there is no magnification error in the sideband measurements.

The changes in microstructure with aging time for alloy 2 are shown in the transmission electron micrographs of figure 5. Alloy 1 and the symmetrical alloy studied by Butler and Thomas showed similar developments of microstructure. For $\lambda < 150\text{\AA}$, the microstructure consists of wavy, irregular plates or rods lying primarily along (100) planes. Even in the specimen aged ten hours, it is difficult to discern a distinct interface between the two decomposing regions. The last micrograph of a specimen aged 1000 hours shows that definite, coherent interfaces parallel to (100) planes have developed

between the Cu rich and Cu poor phases. Also, the two phases in the microstructure shown in fig. 5c appear to be highly interconnected when one takes into account that the micrograph is a two dimensional projection of a three dimensional structure. Micrographs of alloy 1 and the symmetrical alloy are given in figure 6 for comparison of the three microstructures.

The contrast in the electron micrographs of the spinodal structure was enhanced as the amplitude of the composition fluctuations increased with aging. The lattice parameter variation associated with the composition fluctuations causes a phase displacement of the diffracted electrons thus giving the observed contrast. Other factors that could affect the contrast, such as variations in structure factor, diffraction vector and extinction distance, are expected to be negligible for these Cu-Ni-Fe alloys. Cadoret and Delavignette (5) have done a detailed study, including kinematical calculations, of the contrast in spinodally decomposed Cu-Ni-Fe alloys.

A comparison of the measured Curie temperatures and the wavelengths show that the wavelength did not grow significantly until the equilibrium tie-line compositions were reached. Then particle coarsening occurred as shown in figure 5b-c, and the two phases remained coherent after aging for 1000 hours at 625°C. Optical metallography revealed some discernible grain boundary precipitation after aging for one hour, a few discrete grain boundary precipitates after 100 hours and some continuous grain boundary precipitation after 1000 hours.

The yield stress of the spinodally decomposed alloys increased three-fold with long aging times until the equilibrium tie-line compositions were reached; and there was a corresponding 75% decrease in the total strain to fracture. For alloy 1, the yield stress decreased slightly for specimens aged longer than 10 hours; whereas alloy 2 showed no change in yield stress for specimens aged from 10 hours to 1000 hours.

The work hardening rate ($d\sigma/de$) measured at 2% strain increased with aging time for both alloys studied (see fig. 7). While alloy 1 showed a twofold increase in the work hardening rate, alloy 2 had only a 25% increase after aging for 1000 hours. The scatter in the data for aging times up to 10 hours shows no distinct trend; but for specimens aged longer than 10 hours there is a definite increase in the work hardening rate. This increase coincides with the transition from a wavy, irregular morphology to one with distinct, well-defined interfaces. The difference in work hardening behavior of the two alloys is probably due to the difference in the relative volume fractions of the two phases. Since alloy 1 has 75% of the Ni-Fe rich phase and since this alloy work hardened more rapidly than alloy 2, we conclude that more energy is required to deform the Ni-Fe rich phase probably because it has the larger shear modulus.

Fractography revealed that the as quenched specimens and the specimens aged one minute fractured in a completely ductile manner. With increased aging time, the fracture mode became intergranular with some small regions of ductile failure. This transition in fracture mode occurred before grain boundary precipitation was resolved by optical metallography, e.g. fig. 5a showing the spinodal structure after aging for six minutes.

4. DISCUSSION

4.1 Kinetics and Morphology of Decomposition

The rates of spinodal decomposition for the symmetrical and asymmetrical Cu-Ni-Fe alloys were found to be significantly different (see figures 1, 2 and 3). Butler and Thomas⁽³⁾ found that for the symmetrical alloy the equilibrium compositions were reached after aging for one hour at 625°C; whereas the asymmetrical alloys attained the equilibrium compositions after aging for ten hours at 625°C. This order of magnitude difference in decomposition kinetics can be understood by considering the free energy curve and the driving force for the transformation. Since spinodal decomposition is essentially a diffusional process, the rate of decomposition is proportional to the interdiffusional flux ($\tilde{J}$) which is given by the expression⁽¹⁾

$$-\tilde{J} = M \frac{d^2 f}{dc^2} V_c$$

where M is the diffusion mobility and $f(c)$ is the free energy of homogeneous material of composition c . Because $d^2 f/dc^2$ has a maximum value at the critical composition, spinodal decomposition occurs more rapidly in the symmetrical alloy than in the asymmetrical alloys.

Once the asymmetrical alloys reached the equilibrium tie-line compositions, particle coarsening occurred (see figures 1 and 2). Within experimental error, the asymmetrical alloys showed the same coarsening behavior as the symmetrical alloy studied previously⁽³⁾ for which particle coarsening could be described by the rate law $\lambda \propto kt^{1/3}$, which is consistent with the theories of diffusion controlled coarsening where large spherical particles grow at the expense of small ones.

A comparison of the Curie temperature data (figures 1 and 2) with

the pseudo-binary diagram (see fig. 1 of ref. 3) indicates that the as quenched specimens of both alloys had partially decomposed to the spinodal composition, as given by the tie-line, that was nearest to the original alloy composition. For both as quenched alloys, the measured compositions of the Ni-Fe rich phase were $\pm 8\%$ Cu from the original compositions. Thus, during the quench, the fundamental composition waves developed. The fundamental can be described by a sine wave form and is symmetrical about the original composition. As the fundamental grows in amplitude upon aging, the non-linear terms in the solution of the diffusion equation for spinodal decomposition become important and they produce harmonic distortions of the fundamental composition waves.

As discussed by Cahn,⁽⁶⁾ the amplitudes of these harmonic distortions are proportional to the square or higher power of the fundamental amplitude; and when this amplitude is initially small, these distortions are not important. The even harmonics, which have non-zero amplitudes only in asymmetrical alloys, distort the fundamental sine wave to conform to the lever rule by increasing the amplitude of the minor phase while decreasing its spatial extent and vice versa for the major phase. This effect of the even harmonics is clearly shown in the Curie temperature data for alloy 2 aged from one minute up to ten hours. The odd harmonics convert the fundamental sine wave into a square wave, i.e. a composition profile more characteristic of a two phase structure, by flattening the peaks of the fundamental and sharpening the gradient between the extremes in composition. As the gradient at the interface increases, the contrast between adjacent particles also increases and the interface becomes more

distinct. This effect is qualitatively seen in the series of electron micrographs of the aged specimens (fig. 5a-c).

This simple explanation of spinodal decomposition describes the average or overall decomposition process occurring throughout the specimen. But within a localized region the actual decomposition process is more complicated than suggested above because there are several wavelengths present and the effects of the harmonics on the fundamental wave cannot be so precisely described. The microdensitometer trace of a 400 diffraction spot given in figure 4 shows three distinct sideband peaks to the right of the main peak. These diffraction rocking curves suggest that there is a spectrum of wavelengths centered about the dominant wavelength that received maximum amplification. Also, during the initial stages of spinodal decomposition when the wave amplitudes are small, the elastic strain energy term is not very large for Cu-Ni-Fe alloys; and thus the composition waves may not necessarily be along the cube directions.

The decomposition process in the symmetrical and asymmetrical alloys differed significantly in that the wavelength remained constant during the first hour of aging of the asymmetrical alloys whereas the wavelength began increasing immediately upon aging the symmetrical alloy. This difference in the modes of decomposition is probably related to the difference in thermodynamic driving forces since the total free energy change is greater for the decomposition of the symmetrical alloy than for the asymmetrical alloys. Another possible factor is that the even harmonic composition waves, which have non-zero amplitudes only in asymmetrical alloys, may inhibit the growth of the initial fundamental wavelength until the appropriate volume fraction of the two phases,

as given by the lever rule, is attained and the equilibrium compositions are reached.

The wavy appearance of the particles during the first ten hours of aging, as shown in fig. 5a-b, can be understood qualitatively from the following. Khachaturyan⁽⁷⁾ has done an elastic energy analysis of a homogeneously decomposing solid solution and has shown that the shape of the particles is determined by the minimum elastic energy associated with the difference in specific volumes of the precipitating phases. This elastic energy mechanism differs from Cahn's theory which is only valid during the initial stages of spinodal decomposition when the compositional amplitude is small.

Based on Khachaturyan's analysis of decomposition along the cube axes in a face centered cubic solid solution, the particle morphology with the largest free energy is a three dimensional array of cuboidal inclusions. The subsequent microstructure having lower energy is a distribution of rod-shaped particles; and then a distribution of regularly spaced platelets of the equilibrium phases has the lowest free energy of the three types of morphology. The distributions consisting of cuboidal inclusions and rod-shaped particles contain some partially decomposed matrix; and thus, in addition to larger elastic energies, these particle distributions have larger chemical free energies than the platelet morphology. Initially during the decomposition process, composition waves form in all three dimensions along the cube axes as noted by Cahn.⁽⁸⁾ But the total free energy can be reduced by the transformation of this initial cuboidal morphology to a distribution of rod-shaped particles and finally to a platelet morphology.

For the asymmetrical alloys studied, the equilibrium compositions were reached after ten hours of aging. Thus, in specimens aged less than ten hours, partially decomposed matrix was still present and the microstructure consisted of cuboidal inclusions and rod-shaped particles. Because the particle morphology was transforming to platelets of the equilibrium composition, no distinct (100) habit planes developed until the specimens had been aged 100 hours. Consequently, the microstructure had a wavy appearance as observed in the electron micrographs shown in fig. 5a-b.

4.2 Age Hardening

The asymmetrical alloys have age hardening responses similar to that of the symmetrical alloy, i.e. the yield stress increased rapidly with aging time and then became constant for aging times greater than ten hours once the equilibrium compositions were attained. However, there are significant differences between the symmetrical alloy and the asymmetrical alloys. For the asymmetrical alloys, the wavelength initially remained constant while the yield stress doubled. But for the symmetrical alloy, the wavelength grew initially as the yield stress increased (cf. figures 1, 2 and 3). Then as the wavelength increased for aging times greater than ten hours, the yield stress remained constant. But the variation of yield stress for the asymmetrical alloys followed approximately the change in Curie temperature (i.e. composition) of the Ni-Fe rich phase. Thus, the yield stress of the spinodally decomposed Cu-Ni-Fe alloys is independent of the wavelength and is directly proportional to the difference in compositions of the two coherent phases. Also, the symmetrical and asymmetrical alloys aged

at 625°C attained about the same maximum yield stress (35-39 kg/mm²). Therefore, the yield stress is not a function of the volume fractions of the precipitating phases for the case when the volume fraction of the minor phase is not less than one-fourth.

As long as the minor phase remains interconnected, it is physically reasonable to expect the yield stress to be independent of the volume fraction. because, given the interconnectivity of the minor phase, the dislocations must pass through the coherent interfaces and cannot cross slip around the particles. Cahn⁽⁹⁾ did a computer simulation for the spinodally decomposed microstructure of an isotropic material and found that with 0.24 volume fraction the minor phase remained interconnected. The transition to isolated particles was found to occur in the volume fraction range of 0.15 ± 0.03 . The electron micrographs in figure 5 show that the minor phase in the Cu-Ni-Fe alloys was highly interconnected at a volume fraction of 0.25.

These experimental results amplify the suggestions of Butler and Thomas⁽³⁾ concerning the inapplicability of hardening theories based on wavelength changes. Furthermore, the present results show that the Mott and Nabarro theory as originally conceived⁽¹⁰⁾ whilst appearing to apply to the symmetrical alloy does not apply in general to spinodal alloys because there is no direct dependence of strength on volume fraction. The variation of yield strength with composition implies that the important factors affecting the dislocation behavior are changes in lattice parameter, shear modulus and stacking fault energy of the two phases. Another factor contributing to the age hardening may be the energy required to create a new interface between the sheared precipitate and the matrix as discussed

by Kelly and Nicholson⁽¹¹⁾. The graph of yield stress versus the difference in lattice parameters (as estimated from the Curie temperature data) given in figure 8 shows that the yield stress does not vary linearly with the change in lattice parameters. Thus it appears that more than one strengthening mechanism is operative during various stages of age hardening and that the relative importance of the various mechanisms changes as the equilibrium compositions are approached. At the present time there is no satisfactory theory that accounts completely for the observed results. However, it is useful to consider those theories that appear to have some application and which include parameters that are composition dependent.

A theory based on coherency strains in spinodal alloys, which is equivalent to the Mott and Nabarro theory for spherical precipitates, has been developed by Dahlgren⁽¹²⁾ who concluded that the yield stress is independent of the wavelength and of the volume fraction of the minor phase, within certain limits, and is directly proportional to the difference in lattice parameters of the two coherent phases. He derived the following expression for anisotropic crystals with coherent lamellar microstructures:

$$\sigma_y = \frac{\bar{m}}{3\sqrt{6}} \frac{1}{(S_{11} + S_{12})} \frac{\Delta a}{\bar{a}}$$

where $\bar{m}$ is an average Schmid factor for polycrystalline specimens, S_{11} and S_{12} are elastic compliances, Δa is the difference in lattice parameters of the two phases and $\bar{a}$ is the average lattice parameter of the as quenched solid solution. Because the elastic constants show a small composition dependence, this result is an approximation. For randomly oriented grains,

$\bar{m} = 3.06$; but because a preferred orientation was developed during fabrication such that some grains had a [100] axis parallel to the tensile axis, it is reasonable to use the Schmid factor for [100] crystals which is 2.45. For Cu-Ni-Fe alloys on this tie-line, $S_{11} + S_{12} \sim 0.65 \times 10^{-12} \text{ cm}^2/\text{dyne}$, $\Delta a = 0.03\text{\AA}$ and $\bar{a} = 3.58\text{\AA}$. Substituting these values into the above expression, the calculated yield stress is $\sigma_y \sim 44 \text{ kg/mm}^2$ which agrees fairly well with the maximum yield stress measured for these alloys. Theories based on coherency strains can also account for the larger yield stress observed for spinodally decomposed Au-Pt alloys. (13)

No evidence of strain contrast was observed in the electron micrographs of these spinodal structures. However, one would not expect such small coherency strains ($\sim 1\%$) to cause strain contrast. Along a [100] direction the displacement is $|\bar{R}| = \frac{\Delta a}{\bar{a}} \sim 0.009$ and for the diffraction vector $\bar{g} = 200$, then $\bar{g} \cdot \bar{R} \sim 0.02$, which is considerably less than 0.3 which is the value associated with poor or zero dislocation visibility. Consequently, this coherent spinodal structure shows no strain contrast, and it is not possible to verify the coherency strain theory directly. However, as fig. 8 shows, there is not a linear variation between yield strength and Δa .

A theory based on stacking fault strengthening has been developed by Hirsch and Kelly⁽¹⁴⁾. They derive a simple equation for the yield strength of spinodal alloys to be $\tau \approx (\gamma_2 - \gamma_1)b$ which is qualitatively in agreement with our results viz. that the yield strength varies with stacking fault energy differences which are proportional to compositional differences. Estimating that the stacking fault energy difference between

the two phases in Cu-Ni-Fe is approximately 100 ergs/cm^2 , which is reasonable from the present knowledge of stacking fault energies,⁽¹⁵⁾ and taking a mean value for $|\bar{b}|$ to be $2.53\text{\AA}$, we obtain $\tau \sim 41 \text{ kg/mm}^2$ which is the right order of magnitude. However, this stacking fault strengthening mechanism cannot explain the age hardening of Au-Pt alloys which have twice the yield strength of Cu-Ni-Fe alloys,⁽¹³⁾ whereas the stacking fault energies of gold and platinum differ by less than 50 ergs/cm^2 ⁽¹⁶⁾.

The equation derived by Fleischer⁽¹⁷⁾ for strengthening arising from the difference in shear moduli of the two phases also gives the correct magnitude for the yield stress of Cu-Ni-Fe alloys. But this theory is unable to account for the larger yield stress of Au-Pt alloys. For if one assumes that the shear modulus varies linearly with composition, then the maximum possible difference in shear moduli between the two coherent phases is approximately the same for both Cu-Ni-Fe and Au-Pt alloys.

From the above and based on the present experimental data, it is not possible to conclude definitely which strengthening mechanisms give rise to the age hardening of spinodal alloys, although the measured yield stresses of Cu-Ni-Fe and Au-Pt alloys strongly suggest that internal stresses in the coherent microstructure are a significant factor in the age hardening response of spinodal alloys.

5. SUMMARY

The spinodal decomposition of asymmetrical Cu-Ni-Fe alloys was similar in many respects to the symmetrical alloy studies by Butler and Thomas but with some significant differences. The as quenched specimens

contained the fundamental composition waves which formed during the quench. The quenched-in fundamental wavelength did not grow until the equilibrium tie-line compositions were reached. In comparison, the wavelength in the symmetrical alloy began growing immediately upon aging before the equilibrium compositions were reached. The rate of decomposition for the asymmetrical alloys was one order of magnitude slower than for the symmetrical alloy due to the difference in thermodynamic driving forces.

The observed age hardening response of these asymmetrical alloys substantiates the conclusions reached by Butler and Thomas. For this coherent spinodal microstructure, the yield stress is directly proportional to the difference in compositions of the two phases and is independent of the wavelength and the relative volume fractions within the limits $1/4 \leq f_1 \leq 3/4$. The work hardening rate increases when the initially diffuse interfaces become distinct planar (100) interfaces. The age hardening response of spinodally decomposed Cu-Ni-Fe (and Au-Pt) alloys seem to be best explained by theories based on internal stresses due to the difference in lattice parameters of the coherent phases, although it is likely that a single mechanism is not responsible over the whole aging range.

ACKNOWLEDGEMENTS

We wish to thank Dr. A. Goldberg of LRL, Livermore for the loan of the magnetic coil assembly and the United States Atomic Energy Commission through the Inorganic Materials Research Division of the Lawrence Radiation Laboratory for continued financial support of our research. This work was done in partial fulfillment of the M.S. requirements by one of the authors (RJL).

REFERENCES

1. J. W. Cahn, Trans. TMS-AIME, 242, 166 (1968).
2. A. Guinier, Sol. S. Phys. 9, 356 (1959).
3. E. P. Butler and G. Thomas, Acta Met. 18, 347 (1970).
4. A. Goldberg, W. U. Dent and J. H. Miller, J. Sci. Instrum. 44, 200 (1967).
5. R. Cadoret and P. Delavignette, Phys. Stat. Sol. 32, 853 (1969).
6. J. W. Cahn, Acta Met. 14, 1685 (1966).
7. A. G. Khachaturyan, Phys. Stat. Sol. 35, 119 (1969).
8. J. W. Cahn, Acta Met. 9, 795 (1961).
9. J. W. Cahn, J. Chem. Phys. 42, 93 (1965).
10. N. J. Mott and J. R. N. Nabarro, Proc. Phys. Soc. 52, 86 (1940).
11. A. Kelly and R. B. Nicholson, Progress in Mater. Sci. 10, 330 (1963).
12. S. D. Dahlgren, Ph. D. Thesis, University of California, Berkeley, UCRL Report #16846 (1966).
13. R. W. Carpenter, Acta Met. 15, 1297 (1967).
14. P. B. Hirsch and A. Kelly, Phil. Mag. 12, 881 (1965).
15. J. P. Hirth and J. Lothe, Theory of Dislocations, p. 763, McGraw-Hill (1968).
16. I. L. Dillamore, R. E. Smallman and W. T. Roberts, Phil. Mag. 9, 517 (1964).
17. R. L. Fleishher in Electron Microscopy and Strength of Crystals, p. 980, Wiley (1963).

FIGURE CAPTIONS

- Fig. 1. Summary of the changes that occur in yield stress, Curie temperature and wavelength for alloy 1 aged at 625°C. Note that the wavelength remains constant until the equilibrium Curie temperature is attained.
- Fig. 2. Summary of the changes that occur in yield stress, Curie temperature and wavelength for alloy 2 aged at 625°C. Note that the wavelength remains constant until the equilibrium Curie temperature is attained.
- Fig. 3. Summary of the changes that occur in yield stress, Curie temperature and wavelength for the symmetrical alloy aged at 625°C; taken from ref. 3. Note that the wavelength increases immediately upon aging.
- Fig. 4. Microdensitometer trace showing the sidebands on the 400 reflection along the [100] direction for alloy 2 aged 1 hour at 625°C. The three sideband peaks on the right correspond to three wavelengths in the crystal.
- Fig. 5. Transmission electron micrographs of alloy 2 aged at 625°C for (a) 6 minutes $\lambda \sim 75\text{\AA}$, (b) 10 hours $\lambda \sim 135\text{\AA}$ and (c) 1000 hours $\lambda \sim 900\text{\AA}$. Note the change from the wavy, irregular morphology in (b) to the distinct planar interfaces shown in (c).
- Fig. 6. Transmission electron micrographs of (a) alloy 1 aged 10 hours at 625°C $\lambda \sim 130\text{\AA}$ and (b) the symmetrical alloy aged 5 hours at 625°C $\lambda \sim 180\text{\AA}$. Note the difference in morphologies between the symmetrical and asymmetrical alloys.

Fig. 7. Work hardening rate ($d\sigma/de$) vs. log. aging time for the two asymmetrical alloys measured at 2% strain. The transition to distinct, planar interfaces occurred between 10 and 100 hours with a corresponding increase in the work hardening rate.

Fig. 8. Yield stress (σ_y) vs. difference in lattice parameters (Δa) of the two coherent phases as estimated from the Curie temperature data. The wavelength remained constant up to point A on the curves, and to the right of point A the Curie temperature remained about constant. At point B the transition to planar, distinct interfaces occurred.

XBL 702-449

Fig. 3

Fig. 4

XBB 708-3704

Fig. 5

XBB 708-3703

Fig. 6

XBL 708-1915

Fig. 7

XBL 708-1916

Fig. 8

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