Electron Microscopic Observation on the Precipitation Sequence in Cu-Be Alloys*

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Electron microscopic observation on the precipitation sequence in Cu-Be alloys was carried out. Clear images of G.P. zones on {100} planes, the ordered phase, the intermediate γ' -phase, the equilibrium γ -phase and then the informations about the size, shape and distribution in each stage were obtained more clearly than before. Strains associated with precipitates in the early stages of aging and some lattice defects such as dislocations, twins and stacking faults were observed, and these mean that the precipitation in Cu-Be alloys is associated with an exclusively large strain. These electron microscopic results were discussed as compared to those obtained by other methods.

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I. Introduction

Many investigations have been reported on the Cu-Be alloys. In 1944, Guinier et al.⁽¹⁾ reported the presence of beryllium rich platelets (i.e., G.P. zones) on {100} planes in the early stages of aging. Oscillating crystal films of the alloys aged at 190°, 220°, 300° and 420°C suggested that the platelets developed directly into the equilibrium precipitate γ . In 1948, Guy et al.⁽²⁾ published the results of work on the 1.73% Be alloy, which were in very good agreement with those of Guinier et al.⁽¹⁾ In 1951, Gruhl et al.⁽³⁾ proposed two phases γ' (ordered α) and γ (equilibrium precipitate) based upon the results of an examination by the Debye-Scherrer method of polycrystalline samples of 1.8% Be alloy. In 1952, Geisler et al.⁽⁴⁾ advocated the sequence of the precipitation to be γ''' (body-centered monoclinic, $a=b=2.54\text{Å}$, $c=3.24\text{Å}$ and $\beta=85^\circ 25'$) — γ' (body-centered tetragonal, $a=b=2.79\text{Å}$ and $c=2.54\text{Å}$) — equilibrium γ , based upon a detailed review of the pertinent literature on the precipitation. Since thin foil techniques attained a significant development, these earlier X-ray works were re-examined by electron microscope. However, the existence of all of these precipitates has not necessarily been confirmed. Further, no clear images of individual precipitates have been obtained even though their existence has been confirmed.

It is the object of this work (1) to investigate the structure of precipitates and their orientation relationship with the matrix and then the existence of the precipitates for a better understanding of the sequence of precipitation in this alloy, (2) to obtain clear images of the precipitates and then the information about their size, shape and distribution by electron microscopic study, and (3) to correlate the results with those obtained previously by other methods.

II. Experimental Methods

The alloys used were prepared from a mother alloy containing 4 wt% Be. The chemical compositions of these alloys are listed in Table 1. Foil specimens 0.1 mm thick were homogenized at 800°C for 10 hours under hydrogen atmosphere and then water-quenched from 780°C. These specimens were aged at 200°C in a silicon oil bath, and at 350°C and 450°C in a salt bath respectively. Electrothinning was performed on the specimens in phosphoric acid saturated with chromium trioxide at 0°C. The specimens were examined with a JEM-120 type electron microscope operated at 120kV, using a tilting device and a beam deflecting device.

Table 1 Chemical compositions of specimens

No. of specimens	Chemical analysis (wt%)				
	Be	Si	Al	Fe	Cu
V-8090	2.01	0.07	0.02	0.03	rest
V-8091	1.41	0.04	0.01	0.01	rest
V-8092	0.99	0.04	0.01	0.03	rest

III. Results and Discussions

In age-hardenable alloys, the solid solution becomes supersaturated with respect to both vacancies and solute atoms by quenching. Thus, the alloy will tend to precipitate out the excess vacancies and solute atoms in some form or another. It has been established experimentally that for aluminium, aluminium alloys and other metals excess vacancies either condense into dislocation loops or induce the climb of dislocations^{(5)~(9)}.

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(5) P. B. Hirsch, J. Silcock, R. F. Smallman and K. H. Westmacott : Phil. Mag., 3 (1958), 897.

(6) G. Thomas : Phil. Mag., 4 (1959), 606, 1213.

(7) G. Thomas and M. J. Whelan : Phil. Mag., 4 (1959), 511.

(8) R. E. Smallman, K. H. Westmacott and J. A. Coiley : J. Inst. Metals, 88 (1959), 127.

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(2) A. G. Guy, C. S. Barrett and R. F. Mehl : Trans. AIME, 175 (1948), 216.

The absence of loops and climb of screw dislocations into helices seems to be particularly important in some concentrated alloys, since this indicates that most of the vacancies are held in solution probably as a result of the interaction with solute atoms⁽⁶⁾⁽⁷⁾⁽¹⁰⁾. The examination of Cu-Be alloys by transmission electron microscopy shows that after quenching from the region of solid solubility the microstructures do not exhibit any of the expected dislocation configurations resulting from vacancy condensation. Thus, it is concluded that most of the quenched-in vacancies are held in supersaturation and are probably associated with beryllium atoms. This conclusion is supported by the resistivity investigations which have already been published⁽¹⁰⁾. In other words, it has been clarified that an appreciable clustering occurs during quenching and vacancies are trapped at these clusters if there exists the binding energy ($E_b = 0.22 \sim$

0.24 eV)⁽¹¹⁾ between vacancies and clusters.

Upon aging below $\sim 300^\circ\text{C}$ the microstructural changes are similar to those observed during G.P. zone formation in Al-Cu alloys, in that the electron diffraction patterns show continuous streaks through reciprocal lattice points in $\langle 100 \rangle$ as reported by Henmi et al.^{(12)~(14)}, but the clear image of individual G.P. zone has not been obtained in high beryllium content (2wt%) alloys^{(15)~(17)}. The less concentrated alloys were used to distinguish G.P. zones individually. Examples are shown in Photo. 1 (a) (bright field) and Photo. 1 (b) (dark field micrograph using the streaked (220) reflection). The plate-like shapes of the zones and their $\{100\}$ orientations confirm the conclusions already deduced from the diffraction pattern by Guinier et al.⁽¹⁾ and Tanaka et al.⁽¹⁶⁾ The plates of beryllium atoms are $\sim 30 \text{ \AA}$ in thickness and $\sim 500 \text{ \AA}$ in diameter measured in the

Photo. 1 Bright (a) and dark (b) field images using (220) reflection of G. P. zones in Cu-0.99%Be alloy aged at 250°C for 120 hr and the corresponding diffraction pattern (c).

(10) Y. Murakami, H. Yoshida and S. Yamamoto : Trans. JIM, 9 (1968), 11.

(11) A. Lawley, K. F. Lukens, H. Kimura and R. Maddin : Trans. ASM, 60 (1967), 516.

(12) Z. Henmi and T. Nagai : J. Japan Inst. Metals, 30 (1966), 400.

(13) Z. Henmi and T. Nagai : J. Japan Inst. Metals, 30 (1966), 857.

(14) Z. Henmi and T. Nagai : Trans. JIM, 10 (1969), 166.

(15) G. Thomas : *Electron Microscopy and Strength of Crystals*, interscience publisher, John Wiley and Sons, (1963), p. 793.

(16) K. Tanaka, M. Mannami and K. Izumi : Acta Met., 11 (1963), 79.

(17) P. Wilkes and M. M. Jackson : Metal Science Journal, 3 (1969), 130.

micrographs, while the calculated thickness from the length of the streaks is $\sim 4\text{Å}$. This disagreement between these values and the shape of the zones may be explained according to the size-factor hypothesis put forward by Guinier⁽¹⁸⁾. Since the radii of copper and beryllium atoms are 1.28 and 1.11 Å respectively, the zones would then form with about a 10% contraction of the surrounding lattice in a direction normal to the plane of the zones. Such thin zones may be explained by the kinematical theory of diffraction contrast by Hirsch et al.⁽¹⁹⁾ These workers showed that if the atoms in the lattice are displaced from their normal position by a vector $\mathbf{R}$, then a phase factor $\alpha = 2\pi \mathbf{g} \cdot \mathbf{R}$ is introduced in the diffracted wave $\mathbf{g}$. This factor is greatest when $\mathbf{R}$ lies perpendicular to the electron beam. Thus, although theoretically each particle may be too small to be resolved, it can be detected because of the associated strain field. Aging of the more concentrated 2.01% Be alloys for 4 days at 250°C did not reveal the zone images as mentioned above, but developed an unique striated microstructure instead. This is illustrated in Photo. 2 (a) (bright field) and Photo. 2 (b) (dark field image using the $(02\bar{2})$ reflection). The striations do not possess such

2 (b). This may suggest that the striated structure in Photo. 2 is caused not only by a strain contrast effect but also by solute segregations to the strained field. Photo. 2 (c) shows extra reflections of indices $\{110\}$, etc. in the pattern. These extra spots have already been explained in a previous work⁽²¹⁾ in terms of ordered structure which is similar to that discussed by Gruhl et al.⁽⁸⁾, Pfeiffer⁽²²⁾, and Henmi et al.^{(12)~(14)} and can be regarded as being formed as a result of piling up of G.P. zones where beryllium atoms occupy alternate $\{100\}$ layers. Aging at 350°C for 2 hours of 2.01% Be alloys after 19% roll, developed γ' -precipitates having a body-centered tetragonal unit cell with $a = b = 2.79\text{Å}$ and $c = 2.54\text{Å}$. The electron diffraction effects associated with the formation of γ' are similar to those found by X-ray diffraction⁽⁴⁾ and also confirmed by electron diffraction by Nakagawa⁽²³⁾. The diffraction patterns can also be explained by the same orientation relation, $(112)_\alpha // (120)_{\gamma'}$ and $[\bar{1}\bar{1}0]_\alpha // [001]_{\gamma'}$. The streaking as observed at $\{010\}_{\gamma'}$ spots etc. in Photo. 3 (c) is due to the thin nature of the γ' plate-like precipitate and its direction shows that the plates of γ' -precipitates are formed parallel to the $\{112\}_\alpha$ habit planes proposed

Photo. 2 Characteristic "tweed" structure or striation in Cu-2.01%Be alloy aged at 250°C for 4 days after being $\sim 16\%$ rolled. Bright (a) and dark (b) field images using $(02\bar{2})$ reflection in the diffraction pattern (c).

a distinct image as in the case of dislocations or stacking faults, but are a rather diffuse basket-weave of light and dark contrast. Their density is quite high and their distribution is extremely uniform throughout the microstructure. Structures of this type were formed during aging over a wide range of temperature from 200° to 300°C. It has been clarified by Tanner⁽²⁰⁾, that the coherency of G.P. zones includes elastic shear strain on $\{110\}$ planes in $\langle 1\bar{1}0 \rangle$ directions, i.e., at angle of 45° formed with the plane of the zones and that this strain distribution is a consequence of the highly anisotropic elastic behaviour of copper. Thus, the striated structure is a strain contrast effect. In the present work, the results of stereographic analysis are similar to those obtained by Tanner⁽²⁰⁾ in that the striations are parallel to $\{110\}$ traces and that each of the reciprocal points possesses $\langle 110 \rangle$ reldods, but the results are dissimilar in that the striations have not necessarily disappeared even in the condition $\alpha = 2\pi \mathbf{g} \cdot \mathbf{R} = 0$ as shown in Photo.

previously⁽⁴⁾ within a few degrees, i.e. the plane of the γ' plate-like precipitates lies between $\{112\}_\alpha$ and $\{113\}_\alpha$. This can be seen in the bright (Photo. 3(a)) and dark (Photo. 3 (b)) field images. Though the bright field images of these intermediate precipitates are obscured by the $\{110\}$ striations, the dark field micrograph reveals the precipitate particles. These γ' -precipitates were observed to grow to a larger size in the specimens aged at 450°C. This is shown in Photo. 4 (a) with the corresponding dark field micrograph (Photo. 4 (b)) using the $\{010\}_{\gamma'}$ spot and the electron diffraction pattern (Photo. 4 (c)). In Photo. 4 the habit plane is easily confirmed by trace analysis to lie between $\{112\}_\alpha$ and $\{113\}_\alpha$. At a lower aging temperature, the equilibrium phase γ is formed as the nodular products of discontinuous precipitation that occurs predominantly at grain boundaries and spreads into the matrix as reported by Henmi et al.^{(12)~(14)} and Nakagawa⁽²³⁾. Bright (Photo. 5 (a)) and dark (Photo. 5 (b)) field images of γ -precipitates

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(20) L. E. Tanner : *Phil. Mag.*, **14** (1966), 111.

(21) S. Yamamoto and Y. Murakami : *Mem. Fac. Eng., Kyoto Univ.*, **31** (1969), 576.

(22) I. Pfeiffer : *Z. Metallk.*, **56** (1965), 465.

(23) M. Nakagawa : *Japan. J. Appl. Phys.*, **4** (1965), 760.

Photo. 3 Bright (a) and dark (b) field images of intermediate γ' -precipitates in Cu-2.01%Be alloy aged at 350°C for 2 hr after being $\sim 19\%$ rolled. The corresponding diffraction pattern (c) and its indexed pattern (d).

Photo. 4 γ' -precipitates in the Cu-2.01%Be alloy aged at 450°C for 21 hr. Bright (a) and dark (b) field images using $\{010\}_{\gamma'}$ spot in the diffraction pattern (c).

formed during aging at 300°C are shown with the corresponding electron diffraction (Photo. 5 (c)). The γ -precipitates have an ordered body-centered cubic unit cell (CsCl type) with $a = 2.70 \text{ \AA}$. Discontinuous precipitation has been observed by transmission electron microscopy in various alloy systems. The driving force for the discontinuous precipitation in Al-Ag alloy⁽⁹⁾ has been concluded to be purely chemical since no elastic strains (i.e., contrast effects) in grains outside the discontinuous area were observed, while it is thought that in Fe-W and Fe-Mo alloys⁽²⁴⁾, the driving force is provided by the internal strains and that discontinuous precipita-

tion is only likely to occur from the supersaturated iron solid solution containing solute atoms greatly differing in size from iron. In Cu-Be alloys, G.P. zones and intermediate phases prior to the formation of the discontinuous precipitates accompany remarkable elastic strains. Therefore, the strain energy may be more important than the chemical effects for the driving force in Cu-Be alloys. A different morphology of γ -precipitates was obtained when the specimens were aged at 450°C, as shown in Photo. 6. Unlike the specimens aged at 300°C where the shapes of the precipitates were irregular, the specimens aged at 450°C show the precipitates having smooth interfaces.

(24) E. Hornbogen : Z. Metallk., 52 (1961), 47.

Photo. 5 Cu-1.41% Be alloy aged for 20 hr at 300°C showing equilibrium γ -precipitates (a). Dark field image (b) was taken using $\{110\}_{\gamma}$ spot in the diffraction pattern (c).

Photo. 6 Showing γ -precipitates in Cu-1.41% Be alloy aged at 450°C for 141 hr.

In this study, especially in the aging process at 450°C lattice defects such as dislocations, twins and stacking faults were observed in the precipitates or in the matrix around precipitates although the precipitates have been regarded as relatively free from the lattice defects. Photo. 7 (a) shows the diffraction contrast characteristic of stacking fault formed in the precipitates. Bright (Photo. 7 (b)) and dark (Photo. 7 (c)) field images have been obtained using the strong $\{11\bar{1}\}$ reflection shown in the diffraction pattern (Photo. 7 (d)) (The precipitates shown in Photo. 7 seem to have CuAu-I type ordered structure because the diffraction pattern of the $[11\bar{2}]$ orientation (Photo. 7 (d)) shows superlattice spots). The result of the analysis shows that the fault is extrinsic in nature according to the diffraction theory^{(25)~(28)}. It is believed that the energy of the extrinsic fault is probably

higher than that of the intrinsic fault, so that the equilibrium separation of the two partial dislocations will be smaller if an extrinsic fault is formed. In fact, the nature of stacking faults has been investigated in various alloys and in almost all cases the faults were found to be of the intrinsic type⁽²⁹⁾. And the following facts are found in Photo. 7. (1) The dislocations run on both sides of the stacking fault, terminating at the fault, and never pass through it. (2) Only the faulted region in the precipitates is at the Bragg reflecting position similarly as the neighbouring matrix. From the above considerations, a more reasonable interpretation may be given as follows: The nature of the fault in Fig. 7 is not extrinsic and the contrast is arising from two separated overlapping intrinsic faults on the planes which are at short intervals but not neighbouring. Twins were also often observed. An example is shown in Photo. 8. The result of trace analysis shows that the precipitates are formed along the twin boundaries although the plane of the twin boundaries is not $\{111\}$ as distinct from that of the boundary where no precipitates lie. It seems unlikely that the conventional electrolytic polishing of the thin sheet introduces lattice defects into a foil but the subsequent handling of the specimen frequently leads to accidental deformation. But the lattice defects caused by mechanical deformation during thinning or by careless handling of the foil can be avoided in the ordinary handling of specimens. The lattice defects are observed only in the specimens aged at a relatively higher temperature such as 450°C and they can hardly be seen in the alloys (2% Be) aged at a lower temperature such as 350°C. Thus it is safe to say that these lattice defects are formed during precipitation at 450°C. The detailed mechanism of the formation of these defects and their role during precipitation are now under investigation and the result will be reported elsewhere in the near future.

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Photo. 7 The diffraction contrast characteristic of stacking fault can be observed in the precipitates in Photo. 7(a). Bright (Photo. 7 (b)) and dark (Photo. 7 (c)) field images using the strong (111) reflection shown in the diffraction pattern (Photo. 7 (d)). Moiré patterns (Photo. 7 (a)) and dislocations are observed. The diffraction pattern of [112] orientation shows superlattice spots such as (110), etc.

Photo. 8 Twins and the precipitates formed along twin boundaries in Cu-1.41%Be alloy aged at 450°C for 30 min.

The precipitation sequence in Cu-Be alloys in this investigation is: flat G.P. zones — ordered phase — γ' b.c. tetragonal precipitates — equilibrium phase γ . The information about the size and distribution of the precipitates is as follows:

(1) G.P. zones on {100} planes are formed homogeneously in the matrix, the diameter of the zone being less than about 500 Å and the thickness less than 10 Å.

(2) Ordered structure is formed as a result of the piling-up of G.P. zones and has a period about three times greater than that of the face-centered cubic lattice⁽²¹⁾.

(3) Intermediate γ' -phases are directionally arrayed and their sizes, depending upon aging temperatures, vary over a range of 80~300 Å in thickness and 300~20000 Å in length.

(4) Equilibrium γ -precipitates are formed as products of discontinuous precipitation below 350°C. On the other hand, at a high aging temperature (450°C), they are formed in a rather different way and their size, shape and distribution are different from those of the specimen aged below 350°C.

It is well known that the degree of dispersion of the precipitates is one of the fundamental parameters which determine the strength of age-hardening alloys. Comparing above results with mechanical properties⁽¹⁰⁾⁽³⁰⁾, it is concluded that the uniform dispersion of fine intermediate γ' -precipitates or the mixture of G.P. zones and

(30) Y. Murakami, H. Yoshida, T. Kawashima and S. Yamamoto: J. Japan Inst. Metals, 30 (1966), 508.

γ' -precipitates corresponds to the maximum hardness. In the later stage of the equilibrium γ -phase formation, the alloy becomes softened again, but its hardness is still higher than that of the supersaturated solid solution.

It is noticed that the elastic strains play an important role during precipitation in Cu-Be alloys because of the large difference of atomic radii between Be atom and Cu atom. In the early stages of the precipitation at low temperatures, the zones would form with about a 10% contraction of the surrounding lattice in a direction normal to the plane of the zone. As mentioned already, this strain gives rise to contrast effects in the micrographs of the low-beryllium alloys, giving evidence in support of the existence of coherency strains in the alloys. In the more concentrated alloys, the characteristic striations or "tweed" structure in the micrographs and $\langle 110 \rangle$ reldos in the diffraction patterns were observed, and these have been explained in terms of a splitting of the strain field of the zones. The strain field arising from segregation on $\{100\}$ planes of a disc of solute atoms of different size from the solvent atoms may be regarded as partial edge dislocations with the Burgers vector along $\langle 100 \rangle$. The splitting of the strain field proposed is equivalent to the splitting into two edge dislocations with the $\langle 110 \rangle$ Burgers vector⁽¹⁷⁾. The intermediate γ' phase is formed in directional array and it has a definite orientation relationship. Further, the equilibrium γ -phase is formed accompanying lattice defects especially when specimens are aged at high temperatures. During formation of the intermediate γ' -phase (body-centered tetragonal structure) or the equilibrium γ -phase (body-centered cubic structure), the structural change must take place. Since the transformation of a face-centered cubic structure into a body-centered tetragonal or cubic one is associated with an exclusively large strain, the transformation products must have a special shape and orientation relationship, and moreover various lattice defects may be formed to avoid the large strain energy. At any rate, the elastic strains or various lattice defects may play an important role during precipitation in Cu-Be alloys and these effects must explicitly be taken into consideration to clarify the mechanism of the precipitation and to explain the strength of this alloy comparable with that of special steels.

IV. Conclusion

The precipitation sequence in Cu-Be alloys were investigated. The crystal structures of the precipitates, their orientation relationships with the matrix, and the shape, size and distribution of the precipitates were

studied by means of transmission electron microscopy and diffraction. The results of the present work are summarized as follows:

(1) The precipitation sequence in Cu-Be alloy was confirmed to be flat G.P. zones — ordered structure — γ' b.c. tetragonal precipitates — equilibrium γ -phase. The clear images of these precipitates and the information about the size, shape and distribution in each stage were obtained more clearly than before.

(2) The first G.P. zone is the beryllium-rich region on $\{100\}$ matrix planes. It is coherent on $\{100\}$ matrix planes and produces reldos along $\langle 100 \rangle$ directions. Transmission electron microscopy and diffraction reveal that the disc-shaped G.P. zones on $\{100\}$ planes are formed homogeneously in the matrix, with the zone diameter of less than about 500 Å and the thickness of less than 10 Å.

(3) The second ordered structure is formed as a result of piling-up of G.P. zones and has a period about three times greater than that of the face-centered cubic lattice. It is coherent on $\{100\}$ matrix planes and produces extra reflections in the diffraction pattern.

(4) The third precipitate γ' has a body-centered tetragonal unit cell with $a=b=2.79$ Å and $c=2.54$ Å. It is coherent on $\{112\}$ matrix planes and produces two dimensional diffraction effects by anisotropic growth on $\{112\}$ matrix planes. Its orientation relationship is approximately the same as that reported by Geisler et al.⁽⁴⁾ and Nakagawa⁽²³⁾: $(112)_a // (120)_{\gamma'}$ and $[\bar{1}\bar{1}0]_a // [001]_{\gamma'}$. Transmission electron micrographs show that the intermediate γ' -phase are directionally arrayed and their sizes, depending upon aging temperature, vary over a range of 80~300 Å in thickness and 300~20000 Å in length.

(5) The fourth γ -precipitate is an equilibrium phase having an ordered body-centered cubic unit cell with $a=2.70$ Å (CsCl-type). Equilibrium γ -precipitates are formed as products of discontinuous precipitation below 350°C as already reported^{(12)~(14)}. On the other hand, at a high temperature such as 450°C, they are formed in a rather different way, and their size, shape and distribution are different from those of the specimens aged below 350°C.

(6) Strains associated with precipitates in the early stages of aging and some lattice defects such as dislocations, twins and stacking faults were observed, and these mean that the precipitation in Cu-Be alloy is associated with an exclusively large strain. It has been pointed out that these effects must explicitly be taken into consideration to clarify the mechanism of the precipitation and to explain the strength of this alloy.