Composition Dependence of Bainite Morphology in Cu-Zn-Al Alloys

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The morphology of bainite structure is examined by optical and electron microscopy for Cu-Zn-Al alloys with various compositions. It depends not only on the heat treatment for the bainite formation but also strongly on the electron atom ratio, e/a , and the M_s temperature of the alloy. In alloys with e/a smaller than 1.46, the 9R bainite plates are formed with a pair of the variants having (551) and (55 $\bar{1}$) habit planes in a form of long hinge. As e/a increases, the numbers of bainite plates decrease with increasing the size. The trace of habit plane keeps linear until the second structural change, 9R to fcc, takes place in the plates. As the M_s temperature decreases or the aging temperature increases, this change becomes easier to take place. If e/a is larger than 1.46, the bainite plate is transformed with γ_2 particles or a complex eutectic structure consisting of α and γ_2 phases is constructed. In a specimen with $e/a=1.47$, incommensurate γ phase is observed in electron microscopy and the bainite plates are considerably smaller than those in the specimen with $e/a=1.44$. The observed morphological change due to the alloy composition is discussed by using a pseudo-binary phase diagram constructed with the form of Cu-(Zn $_k$ -Al $_1$), where the ratio k of the atomic concentrations of Zn and Al is assumed to be unchanged.

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I. Introduction

The bainitic transformation takes place in Cu-Zn⁽¹⁾⁻⁽⁶⁾, Cu-Al⁽⁷⁾ and Cu-Sn⁽⁸⁾ alloys during cooling from the high temperature β phase or on heating the metastable β_1 phase at a moderate temperature. In the case of Cu-Zn alloys, the plate-shaped product with 9R close-packed structure is simply formed in this transformation. As for Cu-Al alloys, however, a complex eutectic bainite structure composed of the 9R plate with γ_2 precipitate is usually observed. Since the difference in morphologies between these cases is quite remarkable, one might doubt if the bainites are formed with the same mechanism in both alloys.

In Cu-Zn alloys, the bainite plate is believed to be formed with the mode of shear transformation as in the martensitic transformation. The solute concentration is lower in the plate than that in the matrix⁽²⁾⁽⁴⁾⁽⁹⁾⁻⁽¹¹⁾, and the second structural change, from 9R to fcc, occurs during the growth of plate⁽⁶⁾⁽⁷⁾. When the aging temperature is higher than the ultimate temperature for the formation of bainite, B_s , the precipitation of a rod-shaped α phase[†] becomes predominant. Even when the temperature is slightly lower than B_s , the rod-shaped α is often observed to precipitate initially and the bainite plate nucleates preferentially around the rod in Cu-Zn-Al alloys⁽¹²⁾⁽¹³⁾. However, only a few studies have been performed on the crystallography of α precipitation⁽¹⁴⁾⁽¹⁵⁾, and we have not

yet known the formation mechanism of bainite plate around the rod α precipitate in detail. The B_s temperature varies with the solute concentration of the alloy similar to the M_s temperature⁽²⁾⁽⁷⁾. This fact is compatible with the previously obtained experimental results which reveal the martensitic nature⁽¹⁶⁾ in this transformation.

As for Cu-Al alloys, on the other hand, the γ_2 phase with a complex bcc structure⁽¹⁷⁾ or Cu₃Al superlattice domains⁽⁷⁾ are produced initially, and then the bainite plates with a close-packed structure are induced in the surrounding regions of these precipitates. Therefore, the plate is thought to nucleate with a less solute concentration, similarly as in the upper bainite in carbon steels, although no measurement of solute concentration has been made on individual plates of these structures.

In order to understand a general feature of the bainitic transformation in non-ferrous alloys, we examined in the present study the morphology of bainites of many Cu-Zn-Al alloys with various kinds of compositions, situated between Cu-Zn and Cu-Al alloys, by optical and electron microscopy. The observations are made on two series of specimens, i.e., one with the same e/a value of alloy composition and different M_s temperatures, and the other with the same M_s and different e/a . A discussion is made on the difference in the formation mechanisms of plain bainite structure and complex eutectic bainite structure by utilizing pseudo-binary equilibrium phase diagrams⁽¹⁷⁾.

II. Experimental Procedure

Ingots of Cu-Zn-Al alloys were prepared by melting 99.99% pure materials at 1473 K in quartz capsules in argon atmosphere. The nominal compositions of

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† This α has been called the Widmanstätten α ⁽²⁾.

specimens were determined from the following equations⁽¹⁷⁾.

$$M_s(K) = 3250 - 80c_{Zn} - 110c_{Al}$$

$$e/a = 1 + (2c_{Al} + c_{Zn})/100, \quad (1)$$

where c_{Zn} and c_{Al} are the atomic percent of Zn and Al, respectively. Table 1 represents the alloy compositions of the two series of specimens prepared in the present study.

Plate specimens 2 mm thick were prepared by repeating cold- and hot-rolling of ingots, for alloys with e/a between 1.40 and 1.46. For brittle alloys with e/a larger than 1.47, disk-shaped specimens 1 mm thick with 16 mm in diameter were cut directly from the ingot by a high speed cutting machine. These specimens were heated for 300 s at 1143 K, and quenched into water at room temperature. Isothermal agings for the bainite process were carried out for a desired time at various temperatures in a silicone oil or salt bath. Optical micrographic observations were made of the aged specimens after polishing in a CrO_3 - H_3PO_4 solution followed by etching in a $FeCl_3$ - HCl - H_2O solution. Thin specimens for electron microscopic observation were prepared by jet polishing. A Hitachi HU 650 electron microscope was used for observation.

III. Experimental Results

1. Optical microscopic observations

Typical micrographs taken of specimens with different e/a and M_s values aged at 493 K are shown in Figs. 1 ~ 3

Table 1 Alloy compositions of two series of specimens.
Series 1: $e/a=1.40$

M_s (K)	Composition (at%)		
	Zn	Al	Cu
263	20.6	9.7	69.7
303	19.0	10.5	70.5
333	17.8	11.1	71.1

Series 2: $M_s=263$ K

e/a	Composition (at%)		
	Zn	Al	Cu
1.40	20.6	9.7	69.7
1.41	18.4	11.3	70.3
1.44	11.8	16.1	72.1
1.45	9.6	17.7	72.7
1.47	5.2	20.9	73.9
1.48	3.0	22.5	74.5

to survey the basic morphological change. Figure 1 shows bainite plates produced in a specimen with $e/a=1.45$ and $M_s=263$ K, aged for 27 ks. The plates are often observed as a pair of variants having particular habit planes as shown by single arrows in Fig. 1. These pairs have previously been observed in Cu-Zn⁽¹⁾⁽⁴⁾⁽⁵⁾, Cu-Zn-Al⁽¹⁸⁾⁽²¹⁾ and Ag-Cd⁽¹⁹⁾⁽²⁰⁾ alloys.

In Fig. 1, the bainite pairs have nearly the same sizes each other. This fact indicates that the pairs nucleated at almost the same time and not much nucleations took

Fig. 1 Optical micrograph showing bainite plates in the specimen with $e/a=1.45$ and $M_s=263$ K, heated for 27 ks at 493 K.
↗: Variant pair with (551) and $(5\bar{5}1)$ habit planes. ↗: Nucleation site where a large number of bainites are nucleated.

place after that time. Moreover, concerning the shape of pairs, if one assumes a butterfly-shape with half circular wings⁽¹⁹⁾, one might observe different pairs with different sizes depending on the depth of the nucleation site. Consequently, we thought that the shape of wings is not half circular but it is more or less modified by the possible inhomogeneity associated with the crystallographic growth nature.

Concerning this problem, we tried to determine the three dimensional shape of bainite pairs in another experiment using the same specimen as that in the above experiment. Figure 2 shows a part of micrographs[†] successively taken after removing surface layer by 5 μm in depth every time by polishing. The arrows in (a) to (e) indicate the same bainite pair. The pair is clearly observed in the micrographs to move to the right direction as removing the surface deeper, but no remarkable change in the length of one variant of the pair was recognized in (b) to (e). The wings were symmetrical in general, and asymmetry was observed only where the growth of one variant crystal of the pair was suppressed by surroundings. Based on these micrographs, we concluded that the pair has a

long hinge-shape as schematically shown in (f). The analysis also indicated that the bainite was formed heterogeneously at a nucleation site. That is, we often found that bainites separated enough each other converge together to a point as removing the surface deeper. Examples of the preferred nucleation sites are shown in Fig. 1 by double arrows⁽¹²⁾⁽¹³⁾.

Figure 3 indicates different intergranular bainite structures in alloys with different compositions, aged at 493 K. (a) is a micrograph taken from a specimen with $e/a=1.40$ and $M_s=263$ K, aged for 1.5 ks. Numerous bainite plates with particular crystal habits are seen in the micrograph. (b) shows a similar micrograph from a specimen having the same M_s temperature with $e/a=1.45$, larger than in (a), aged for 27 ks. The number of bainite plates is much less than that in (a), although the aging time is increased. The plates grow remarkably with straight habit traces. In (c) are shown numerous thin and small bainite plates transformed by aging a specimen

[†] The photographs were taken covering an area about 8 times larger than that shown in Fig. 2.

Fig. 2 A series of optical micrographs successively taken after removing the surface layer, 5 μm thick in every time, (a)–(e). $\nearrow$: The same bainite pair. (f) Shape of bainite pair.

Fig. 3 Optical micrographs showing the composition dependence of bainite structure. All specimens are heated at 493 K. (a) $e/a=1.40$, $M_s=263$ K (b) $e/a=1.45$, $M_s=263$ K (c) $e/a=1.40$, $M_s=303$ K (d) $e/a=1.40$, $M_s=333$ K.

with $e/a=1.40$ and $M_s=303$ K for 600 s. (d) represents a mixed structure of bainites and martensites in an alloy with $e/a=1.40$ and $M_s=333$ K, 30 K higher than that of the specimen in (c). In the as-quenched state, one observes numerous self-accommodated martensite groups and after aging for 3.6 ks at 493 K, the morphology changes as in (d).

Systematic observations as shown in Fig. 3 on the effect of alloy compositions of specimens with e/a between 1.40 and 1.47 clearly gave the following results: (1) As the e/a value increases, the incubation period of the bainite formation increases and the numbers of bainite plates decrease with increasing size of plate. (2) As far as the M_s temperature is concerned, the incubation period varies in opposite sense.

Figure 4 shows micrographs of bainite in an alloy with $M_s=263$ K and $e/a=1.44$, aged at 623 K, which is higher than that in Fig. 1. (a) represents bainites having wavy habit traces and α rod precipitates in a specimen aged for only 120 s. The bainite pattern was modified to (b) by an additional aging, for 600 s in total. No remarkable growth of bainite was observed in the longitudinal direction from (a) to (b). The arrows in (a) show α rod precipitates at the edge of bainite plates, which would suppress the growth of plates in this direction. On the other hand, the thickness clearly increased and the habit trace became wavy. In this stage of transformation, the crystal structure of the plate is changed to fcc⁽²¹⁾. Finally, the following change was recognized in a

specimen having $M_s=263$ K with e/a between 1.40 and 1.46, as the aging temperature was increased: (1) The incubation period is decreased. (2) The habit trace becomes wavy with ease. (3) The structural change, 9R to fcc, takes place easily.

As for a specimen with e/a larger than 1.46, the γ_2 precipitation[†] becomes predominant and the morphology changes drastically. Figure 5 shows an example of optical micrographs from specimens with $e/a=1.47$, aged at 673 K. (a) and (b) are taken after aging for 2.7 ks and 5.1 ks, respectively. Near the boundary at the center in Fig. 5(a), we can see a massive complex pattern of eutectic decomposition consisting of γ_2 and α phases. In addition, many small bainite plates are seen inside the grain, which look very fine and homogeneous compared with those in alloys with $e/a=1.44$. Figure 5(b) shows complex eutectic decompositions with a granular shape coexisting with the bainite plates inside the grain.

In alloys with higher e/a and aged at lower temperature, the γ_2 precipitates are individually formed after a longer incubation period inside the grain, and eutectic decomposition to γ_2 and α often takes place at the grain boundary. With increasing temperature, the incubation period is shortened and complex morphology with decomposed regions and plate bainites is observed at the end of transformation.

[†] Since the matrix crystal has an ordered D0₃ structure in this range of ternary alloys, the precipitates are expected to have the γ_2 structure.

Fig. 4 Bainite plates with wavy habit traces and α rods in the specimen with $e/a=1.44$ and $M_s=263$ K, heated at 623 K. (a) 120 s (b) 600 s. $\nearrow$: α rods which disturb the growth of bainite plates.

2. Electron microscopic observations

Figure 6(a) shows an electron micrograph showing initially formed bainite plates with straight habit traces in a single crystal specimen with $e/a=1.41$, aged for 1.8 ks at 493 K. The orientation of specimen surface has been determined by the X-ray Laue method prior to the observation. The index of habit plane can definitely be determined by the trace analysis together with the measurement of the angle between traces of the bainite pair⁽⁵⁾. The index analysis indicated that the variant crystals, named A and C in Fig. 6(a), have habit indices of (551) and $(5\bar{5}1)$, respectively, rather than $(12\ 11\ 2)$ and $(12\ 11\ \bar{2})$ so far reported⁽¹⁾⁽⁵⁾. However, by prolonged aging, the habit traces became wavy, accompanying the structural change from 9R to fcc, as shown in Fig. 6(b). The specimen with $e/a=1.44$ was aged for 3.5 ks at 623 K in this case. Consequently, determination of the habit plane indices becomes difficult as the bainite reaction proceeds. The arrow shows an α rod precipitate appearing in addition to the plate bainites.

Figure 7 indicates an electron micrograph and electron diffraction pattern taken from a specimen with $e/a=1.47$ after aging for 11.4 ks at 573 K. One can see granular precipitates about $0.3\ \mu\text{m}$ in size in (a). In the corresponding electron diffraction pattern in (b), weak addi-

Fig. 5 Eutectic decompositions coexisting with bainite plates in the specimen with $e/a=1.47$, heated at 623 K. (a) 2.7 ks (b) 5.1 ks.

tional spots were observed at about $1/3\ g_{220}$ of $D0_3$ as shown by arrows. A careful measurement shows that these spots are not exactly at $1/3$ positions but slightly shifted to the direction of g_{220} . Therefore, we expect that the precipitates are the incommensurate γ phase reported by Lovey *et al.*⁽³⁰⁾

Figure 8(a) is an electron micrograph showing a bainite plate appearing between two γ_2 granular precipitates. The micrograph was taken after heating a specimen with $e/a=1.47$ for 68 ks at 523 K. The plate is thought to be formed through a possible reduction of solute concentration caused by γ_2 precipitation. The micrograph in Fig. 8(b) was taken for a specimen with $e/a=1.48$ aged for 900 s at 673 K. A large bainite band at the center seems to appear first and then the precipitation of γ_2 follows in the surrounding matrix. The crystal structure of bainite was determined to be 9R, although the martensite in the alloy has the 2H structure.

IV. Discussions

In the early stage of transformation, the bainite plates are often observed as a pair with two variant crystals having (551) and $(5\bar{5}1)$ habit planes. The shape of the pair is hinge-like with a long axis in $[1\bar{1}0]$ direction. The two variant crystals are those having almost the same transformation shear among the four variants of self-accommodation⁽²²⁾⁽²³⁾ in the martensitic transformation. The shear directions are observed to be in common in the

Fig. 6 Electron micrographs showing bainite plates in the initial and last stages of transformation. (a) $e/a=1.41$, heated for 1.8 ks at 493 K. (b) $e/a=1.44$, heated for 3.6 ks at 623 K. A, C: Variant pair. ∇ : α rod precipitates.

both variants and almost parallel to $[1\bar{1}0]$.

The growth of bainite is controlled by the concentrated stress field⁽¹²⁾⁽¹³⁾ which would be produced at the tip by the transformation shear and accordingly, the growth could not be isotropic. Actually, it has been verified that the velocity of growth is three times larger in the direction parallel to the transformation shear than that perpendicular to it[†]. This would be the reason why one obtains the hinge-shaped bainite pairs.

In order to discuss the composition dependence of the bainite morphology, we must take account of the equilibrium phase diagram of the present alloys. Unfortunately, however, no reliable phase diagrams covering whole range of Cu-Zn-Al ternary alloys are available at present. Since then, we try here to construct a pseudobinary phase diagram by using so far reported ternary diagrams in a limited range⁽²⁴⁾ with those of the Cu-Zn and Cu-Al binary systems⁽²⁵⁾.

We assume that Zn and Al atoms are mixed randomly in the alloy and also the ratios of Zn and Al contents are unchanged during the transformation. Under these assumptions, the solute contents in the present alloy can

[†] A measurement on the anisotropic growth of bainite plate has recently been made in the author's laboratory. A paper thereupon is to be read at the ICOMAT-92 in Monterey.

Fig. 7 Electron micrograph and diffraction pattern of incommensurate γ phase in the specimen with $e/a=1.47$, heated for 11.4 ks at 573 K. ∇ : Spots due to incommensurate γ .

Fig. 8 Electron micrographs of bainite plates and γ_2 precipitates in the specimens with higher e/a values. (a) $e/a=1.47$, heated for 68 ks at 523 K. (b) $e/a=1.48$, heated for 900 s at 673 K.

be expressed as $\text{Cu}-(\text{Zn}_{kx}-\text{Al}_k)$, in the binary form, where $x=c_{\text{Al}}$ and $kx=c_{\text{Zn}}$ are the atomic concentrations in % of Al and Zn, respectively. The parameter k can be written as $k=c_{\text{Zn}}/c_{\text{Al}}$, which is different for every alloy ingot. When the M_s temperature is set to 263 K, k can be obtained as 7.39, 2.13 and 0.82 for alloys with $(e/a)_0=1.40, 1.44$ and 1.48 , respectively. Since the electron atom ratio can be expressed as

$$e/a = 1 + (2+k)x/100, \quad (2)$$

the pseudo-binary phase diagrams are constructed by taking e/a as abscissa as shown in Fig. 9. The diagrams display a clear increase in the eutectic temperature with decreasing k , i.e., relatively increasing the Al content. On the other hand, the phase boundary of α phase is rather insensitive to k . We will discuss the observed morphology by using these diagrams in the following.

We start from the point p in the diagrams, setting the aging temperature at 573 K. Suppose that a reduction of solute concentration takes place for some reason or other, from p to q, in a localized region. Then, the transformation would appear martensitically, because the M_s reaches to the aging temperature. As shown in Fig. 3, the closer is the M_s to the aging temperature, the easier is the bainite formed. Consequently, the bainite can be nucleated at a region where the solute atoms are deficient, provided that the size of nucleus is as large as a critical size of bainite nucleus. The critical size is expected to be about 10 nm, assuming that the sizes of bainites and martensites⁽²⁶⁾ are the same. The bainite is heterogeneously nucleated at a site, shown in Figs. 1 and 3, where the reduction of solute concentration takes place and where α rod precipitates. Accordingly, the concentrated stress field around the α rod precipitate is

thought to be the origin producing the reduction of solute concentration.

Figure 9(a) is the diagram for an alloy with $e/a=1.40$. The point q is inside the stable α range, so that the nucleated bainite with the 9R structure will become immediately disordered and may quickly transform to the fcc structure. This is the reason why the bainite plate in this alloy has always been observed with a disordered 9R structure⁽⁴⁾⁽⁶⁾⁽¹⁸⁾, or with wavy habit traces having the fcc structure. When the bainite has a disordered structure, the lattice invariant shear is not necessarily of stacking faults but the slip deformation can operate instead. Then, the habit plane $\{551\}$ would change to $\{321\}$, across which the two lattices might fit well⁽²⁷⁾⁽²⁸⁾. Actually, Srinivasan and Hepworth⁽⁵⁾ have measured the movement of habit plane index from $\{551\}$ to $\{321\}$ following the progress of transformation. Since q is inside the α range, the solute concentration is expected to be smaller in the bainite than in the stable α phase and this is consistent with the results obtained by Lorimer *et al.*⁽¹⁰⁾ With increasing aging temperature, this tendency becomes more predominant and this can also be explained by the fact that the point q is displaced to the left in Fig. 9(a).

For a specimen with M_s little higher than 263 K and the same e/a , for example $M_s=303$ K, i.e. $k=5.90$, between 7.39 and 2.13 in Fig. 9(a) and (b), respectively, the eutectic temperature as well as the M_s line will shift upward slightly. We think that the morphology will be influenced by the latter change more explicitly than the former, i.e., the line pq is shortened and the bainite will be formed easier by a change in solute concentration. In fact, the morphology in Fig. 3(c) indicates a clear difference from (a).

Figure 9(b) represents the diagram for $k=2.13$. The

Fig. 9 Pseudo-binary phase diagrams of the Cu-Zn-Al alloys with $M_s=263$ K. (a) $(e/a)_0=1.40$, $k=7.39$ (b) $(e/a)_0=1.44$, $k=2.13$ (c) $(e/a)_0=1.48$, $k=0.82$.

points p and q are displaced to the higher e/a side, apart from the phase boundary of α . As shown in Fig. 3, the incubation period of transformation increases with e/a . As the point q is apart from the solidus line, it becomes hard to nucleate the α phase and it would take more time to prepare a site suitable for bainite nucleation. The bainite with flat habit plane is stable upon heating in alloys with large e/a . This fact is also confirmed in Fig. 9(b); the point q is in the coexisting range of α and β_1 phases and the 9R is rather stable similarly as in the case of martensite there. A detailed discussion has been made on the relation between the transformation products, 9R bainite rod α , and equilibrium α phase in the previous paper⁽²⁹⁾.

Figure 9(c) is the diagram for $k=0.82$. The alloy with $e/a=1.48$ is situated near the eutectic position and the typical eutectic structure is usually observed in a quickly diffused region, for example, near the grain boundary as shown in Fig. 5. An electron microscopic examination shows that these structures are of alternatively arranged lamellae shape with α and γ_2 phases, like the pearlite in steels, and so the structure should be classified as "pearlite". However, as for the eutectic structure appearing with bainite in Fig. 5(b), a mixture of granular γ_2 and plate-shaped bainite is detected by electron microscopy, and this is to be called "bainite"⁽⁷⁾.

On the other hand, in the region with low diffusion rate of solute atoms, such as intergranular ranges, the γ_2 phase initially precipitates when the aging temperature is not so high. Since the concentration is changed from p to q' in Fig. 9(c), solute poor regions must be formed around the γ_2 precipitates, and after the concentration q is reached, the bainite crystals are martensitically produced covering the γ_2 crystals, as seen in Fig. 5(b). The bainite structures in Fig. 5(b) and Fig. 8(a) are similar to the upper bainite structure in steels, which are known to be constructed by alternative precipitations of carbide and ferrite.

Prior to the precipitation of γ_2 phase, an incommensurate γ phase is generally produced in the matrix as in Fig. 7. This fact suggests that the lattice vacancies would play an important role in the bainite transformation. The electron diffraction spots due to the incommensurate γ phase in Fig. 7(b) are displaced from the $1/3$ positions in the opposite direction from those reported by Lovey *et al.*⁽³⁰⁾ The reason may be attributed to the aging procedures. That is, Lovey *et al.* quenched the specimen directly to the aging temperature, while in the present study, the bainite formation treatment was made after keeping the quenched specimen for a certain period at room temperature. The number of vacancies would be much larger in the former specimens than the latter. A large number of very small bainite plates have been observed as in Fig. 5(a), even in the present specimen,

although a matrix region containing incommensurate γ phase was selectively photographed in Fig. 7. Such a small bainite may be originated from the possible fluctuation in solute concentration in the matrix caused by the formation of the incommensurate γ phase.

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