High Resolution Analytical Electron Microscopy Study on the Bainitic Transformation in a Ag-45 at% Cd Alloy*

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The bainitic transformation in a Ag-45 at% Cd alloy at 433 K has been examined by means of optical microscopy, high resolution analytical electron microscopy and electron diffraction. As a result, the following were clarified: Crystal structure and morphology of the bainitic product (α_B) in an early stage of growth were an internally faulted (orthorhombic) N9R type long period stacking order structure and a plate shape with parallel straight interfaces, respectively. They were very similar to those of (monoclinic) M9R or M18R type martensite plates in other noble metal base β phase alloys. However, they were different from an internally twinned 2H type stacking order structure and a spear shape, respectively, of the normal martensites produced in the sub-zero cooled Ag-Cd alloy, and moreover no ordered structure was recognized in the N9R α_B plates even in the very early stage of growth. The α_B plates approached the equilibrium α phase with the progress of growth, changing the faulted N9R to a non-faulted fcc structure and the straight interfaces to the swollen ones. Composition of the α_B plates was almost the same as that of the equilibrium α phase from the very early stage of growth (more than 30 nm width). From these observations, it was concluded that diffusion of Cd atoms first occurred in the β_2 matrix, approaching the composition of the equilibrium α phase, and then martensitic transformation occurred due to a rise of M_s in the regions reduced in Cd content. This conclusion was consistent with the formation of 9R α_B plates which were different from the normal 2H martensites in the Ag-Cd alloy.

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I. Introduction

The bainitic transformation has been known to occur not only in ferrous alloys but also in non-ferrous alloys such as Cu-Zn binary⁽¹⁾⁽²⁾, Cu-Zn base ternary⁽³⁾⁻⁽⁵⁾ and Ag base binary⁽⁶⁾⁽⁷⁾. According to previous studies⁽⁸⁾⁻⁽¹¹⁾, the bainitic products in those non-ferrous alloys are very similar to diffusionless martensitic products with respect to the crystallographic features such as the surface relief effect, the habit plane, internal defects and the orientation relationship. On the other hand, the bainitic

transformations exhibit a C-curve behavior in the T - T - T diagram⁽²⁾⁽¹²⁾, which is characteristic of the diffusion control transformations. Then some workers considered that the bainitic transformations should be of dual nature, diffusionless and diffusional, although their relations were not clarified. That is, some workers proposed that the diffusionless martensitic transformation first occurred in the matrix without any compositional change and then diffusion occurred to approach the composition of the equilibrium α phase accompanying a structural change into a fcc lattice⁽¹⁾⁽²⁾⁽⁸⁾⁻⁽¹²⁾, but some others did that diffusion first occurred in the matrix to approach the composition of the α phase and then martensitic transformation occurred due to a rise in M_s in the matrix regions low in solute content⁽¹³⁾⁽¹⁴⁾. Some workers of another group⁽¹⁵⁾⁽¹⁶⁾, however, were opposed to the above proposition asserting that the bainitic transformation was a diffusion control precipitation phenomenon of the transitional α phase, and

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that the α precipitates simply exhibited similar crystallographic features to martensitic products.

In the present study, then, the bainitic transformation in a Ag-45 at% Cd alloy has been examined to clarify the above obscure points, although similar studies have so far been made by a few workers⁽¹¹⁾⁽¹⁷⁾. The present study has been performed by using a newly developed high resolution analytical electron microscopy with a field emission electron gun, which enables us to take electron diffraction patterns with a high angular resolution from 5 nm diameter areas in minimum and to analyze the constituent elements from energy dispersive X-ray spectroscopy of those areas.

II. Experimental Results

A Ag-45 at% Cd alloy ingot was prepared by melting high purity Ag (99.999%) and Cd (99.999%) in a sealed quartz tube filled with argon gas, the tube being placed in a furnace at 1273 K. The ingot was again melted in another sealed quartz tube (12 mm diameter) filled with argon gas, and casted into a 3 mm diameter quartz tube which was connected to the 12 mm diameter tube. The 3 mm diameter ingot was annealed at 823 K for 86.4 ks in a sealed quartz tube filled with argon gas. The weight loss during the heat treatments was less than 0.5%, indicating that the ingot had the same composition as scheduled. From the 3 mm diameter ingot, 0.3 mm thick sheets were cut and supplied for examinations.

The 0.3 mm thick sheets were then betatized at 955 K for 300 s in air, and rapidly quenched to 260 K by compulsory dropping. The M_s temperature of the quenched alloy was measured by differential scanning calorimetry (DSC) to be 218 K. The quenched sheets were then heat-treated for the bainitic transformation in a silicon oil bath at 433 K for various periods up to 1468.8 ks (17 d).

The bainitic products thus obtained have been observed by optical and transmission electron microscopy (TEM). The specimens for optical microscopy were electrolytically etched by a jet method, using a 10% KCN solution at the voltage of 15 V. The thin foils for the TEM

observation were prepared by normal electropolishing after the specimens were jet-polished. Jet-polishing was done in the solution of 10% KCN and 1% K_2CO_3 at room temperature under the condition of 15 V and 80 mA, and normal electropolishing in the solution of 5% KCN and 1% gelatine at room temperature at the voltage of 2.5 V. Some of the as-quenched specimens before the bainitic transformation were electropolished in the solution of 5% KCN and 1% gelatine at room temperature, and then sub-zero cooled after they were mounted on the cooling stage of the optical microscope. The martensitic structure during the sub-zero cooling was supplementally observed in the temperature range from 183 to 173 K.

The electron microscopes used were a Hitach H-700SS operated at 200 kV and a Hitachi H-600FE operated at 100 kV. The latter⁽¹⁸⁾ is a specially ordered high resolution analytical electron microscope with a field emission electron gun, which enabled us to take electron diffraction (ED) patterns with a high angular resolution from 5 nm diameter areas in minimum and to analyze the constituent elements of the areas by the energy dispersive X-ray (EDX) spectroscopy method. Because of the use of a field emission electron gun, the column of the microscope is of an ultra high vacuum, being about 10^{-5} Pa in the specimen chamber, and therefore no contamination was produced on the surface of the examined specimens. The thin foil specimens for analytical electron microscopy were sputtered by argon ions to remove some persistent surface films formed during electropolishing by using an ion beam thinning apparatus (Japan Oxygen-Edward E306 type IBT 200) operated at 2 kV.

III. Results

1. Optical microscopy observation

Figure 1 shows optical micrographs of the β_2 matrix and bainitic (α_B)[†] and martensitic products in the Ag-45 at% Cd alloy. (a) is of the β_2 matrix, which was taken from the polished surface of an as-quenched specimen at room

Fig. 1 Optical micrographs of a Ag-45.0 at% Cd alloy as-quenched, (a), the α_B plates formed in the alloy subsequently heat-treated at 433 K for 3.6 ks, (b), and the γ_2 martensites produced in the as-quenched alloy upon subzero cooling to 183 K, (c).

temperature. No specific structure is observed in (a). (b) is of the α_B product, which was taken from the etched surface of a specimen isothermally heat-treated at 433 K for 3.6 ks and then quenched to room temperature. In (b) are seen α_B plates with characteristic habit planes and massive products without any specific habit plane (arrowed), the α_B plates being of an orthorhombic N9R long period stacking order (LPSO) structure, as will be verified later. (c) is of the normal martensitic products, which was taken from the unetched surface of an as-quenched and then sub-zero cooled specimen at 183 K. There are seen a surface relief effect due to the martensitic transformation and internally striated ($\uparrow$) spear shape martensite plates. According to a previous study⁽¹⁹⁾, the spear shape martensites are γ_2 and probably of a 2H stacking order structure, and the internal striations are twins. It should be noted here that the structure and morphology of the α_B plates in Ag-45 at% Cd alloy are different from those of the normal γ_2 martensite plates produced in the sub-zero cooled alloy, although such a difference has not been observed in Cu-Zn(-Al) alloys⁽³⁾⁽⁸⁾⁻⁽¹¹⁾⁽²⁰⁾⁻⁽²²⁾.

[†] In the present paper, the bainitic product will be denoted as $\alpha_B^{(23)}$, since the suffix 1 of the α_1 used so far gave us the impression that the matrix phase was ordered into the D0₃ type superlattice, not into the B2 type one in the present alloy.

2. Electron microscope observation

Figures 2(a) and (b) are TEM images of α_B plates in the Ag-45% Cd alloys heat-treated at 433 K for 5.4 and 169.2 ks, respectively. The α_B plate in (a) is supposed to correspond to an early stage of growth, since the heat-treated period at 433 K is not so long and the width of the plate is not so wide. The plate has parallel straight interfaces and internal defects with homogeneous and high density. This morphology is very similar to that of monoclinic M9R or M18R type martensites in other noble metal base β phase alloys such as Cu-Zn(-Al)⁽³⁾⁽⁸⁾⁻⁽¹¹⁾⁽²⁰⁾⁻⁽²²⁾, and therefore the α_B plate is supposed to be also of a 9R type LPSO structure, as will be verified later. Upon the progress of growth, α_B plates increase in thickness and the density of internal defects decreases, the distribution of the defects becoming inhomogeneous, as seen in (b). At the same time, interfaces lost the parallelism, partly showing a swelling. A similar morphological change with the progress of growth has also been observed in Cu-Zn(-Al) alloys⁽¹⁴⁾. It should be noted in Fig. 2 that a basket weave structure is recognized in the β_2 matrix regions, as clearly seen in a magnified image at the lower right corner, and this will be discussed later.

Figures 3 and 4 are the TEM images and ED patterns taken from the α_B plates of 50 and

Fig. 2 (a) and (b): TEM images of the α_B plates formed in a specimen heat-treated at 433 K for 5.4 and 169.2 ks, respectively, showing a morphological change of α_B plates with the progress of growth.

200 nm width, respectively, in a specimen heat-treated at 433 K for 5.4 ks. In both figures, ED patterns (b) and (c) were taken from the black spot areas (~ 5 nm diameter) in the α_B plates and the adjacent β_2 matrix regions, respectively, in TEM images (a). (b) shows that the α_B plates are of a 9R type LPSO structure, their c^* axis appearing on the patterns, and (c) does that the β_2 matrices are of a B2 type structure, revealing their $[\bar{1}00]$ zone's, in which 010 type superlattice reflections ($\uparrow$) are clearly observed, although their intensity is not so strong because of a small difference between the atomic scattering factors of Ag and Cd atoms. In addition to the B2 superlattice reflections, anomalous reflections are seen as indicated by double arrows, and this will be discussed later.

Both of (b) are of the $[010]$ zone's of 9R α_B plates, because 009 and 600 reflections are seen. $[009]^*$ and $[600]^*$ reciprocal directions

Fig. 3 (a): A TEM image of an α_B plate of about 50 nm width in an early stage of growth, observed in a specimen heat-treated at 433 K for 5.4 ks. (b) and (c): ED patterns taken from the areas indicated by black spots in the α_B plate and the adjacent parent phase in (a), respectively.

Fig. 4 (a): A TEM image of an α_B plate of about 200 nm width in an intermediate stage of growth, observed in the same specimen as in Fig. 3. (b) and (c): ED patterns taken from the areas indicated by black spots in the α_B plate and the adjacent parent phase in (a), respectively.

make the right angle to each other, and superlattice reflections of $h + k = 2n \pm 1$ (n : integer) indices which are inherited from those of the B2 type matrix are not observed in the pat-

terns. These facts indicate that the α_B plates in the Ag-45 at% Cd alloy has a disordered orthorhombic N9R LPSO structure although the matrix is ordered. A similar result has very recently been obtained by Wu *et al.*⁽²⁴⁾. These results are largely different from those of Cu-Zn(-Al) alloys in which the α_B plates have been observed to possess an ordered monoclinic M9R LPSO structure⁽¹⁴⁾⁽²¹⁾⁽²²⁾. Moreover, as seen in Fig. 5 taken from a bainite plate of 120 nm width, the $1/3$ and $2/3$ reflections along the $[009]^*$ reciprocal axis in (a) disappeared when the specimen was tilted around the axis, as known from (b). This indicates them to be double reflections, not intrinsic ones. This result is also different from the case of Cu-Zn(-Al) and Ag-Zn alloys⁽⁹⁾⁽²⁵⁾, in which the $1/3$ and $2/3$ reflections were intrinsic. Thus, no periodic segregation of Cd atoms on every third layers parallel to the basal plane is recognized in the N9R α_B plates of the Ag-45 at% Cd alloy.

The striations seen in the α_B plates of Figs. 3(a) and 4(a) are perpendicular to the c^* axis in (b), and all the reflections in (b) are streaked perpendicular to the striations and parallel to the c^* axis. Therefore, the internal striations are probably stacking faults on the basal planes, as in monoclinic M9R or M18R type LPSO martensites in other noble metal base β

Fig. 5 (a): $[010]$ zone ED pattern of the N9R structure taken from an α_B plate of about 120 nm width. (b): ED pattern obtained by tilting the specimen around the c^* axis in (a) so that all the reflection rows other than those with $00l$ in (a) disappear.

Fig. 6 (a): A TEM image of an α_B plate of several hundreds of nm in width in a later stage of growth, observed in a specimen heat-treated at 433 K for 1468.8 ks. (b) and (c): ED patterns taken from the areas indicated by black spots in the α_B plate and the adjacent parent phase, respectively.

phase alloys.

Figure 6(a) is a TEM image of an α_B plate whose thickness and stacking fault density increased and decreased with the progress of growth, respectively, when heat-treated at 433 K for 1468.8 ks. Figure 6(b) and (c) are corresponding ED patterns of the plate and the adjacent matrix, respectively. As mentioned already, the plate in (a) shows swollen interfaces in the later stage of growth. The pattern (b) can be explained to be of the [110] zone's of a fcc structure, and there is seen neither superlattice reflection nor tetragonality. Therefore, the plate may be said to change from a disordered N9R to a disordered fcc. In α_B plates of Cu-Zn(-Al) alloys⁽¹⁴⁾ an ordered M9R to disordered fcc transition via a disordered N9R state is observed. On the other hand, (c) shows an ordered B2 structure in the adjacent matrix, because of the appearance of its superlattice reflections, as in the as-quenched state. The disordered fcc structure may probably correspond to that of the equilibrium α phase.

3. Analytical electron microscopy

Figures 7 and 8 are a series of TEM images and EDX spectra, which were taken from the α_B plates of 30 and 150 nm width, respectively, and their adjacent matrices in a specimen heat-treated at 433 K for 2.4 ks^{††}. They may, therefore, give us the compositional information on the early and middle stages of bainite growth, respectively. The black spots shown in Figs. 7(a) and 8(a) indicate the areas from which EDX spectra (b) and (c) have been taken, (b) and (c) corresponding to the α_B plates and their adjacent matrix regions, respectively. The size of the black spots is far smaller than the width of the α_B plates, and moreover the spacial resolving power of the present EDX analysis is estimated to be about 30 nm from a simple approximation⁽²⁶⁾ as to the spreading of electron beam in the specimens, the specimen thickness and the accelerating voltage of the electron microscope being assumed to be about 100 nm and 100 kV, respectively. Therefore,

^{††} No bainite plates were observed in the specimens heat-treated for a period shorter than 1.8 ks.

Fig. 7 (a): A TEM image of an α_B plate in an early stage of growth, observed in a specimen heat-treated at 433 K for 2.4 ks. (b) and (c): EDX spectra taken from the areas indicated by black spots in the α_B plate and the adjacent parent phase, respectively.

Fig. 8 (a): A TEM image of an α_B plate in an intermediate stage of growth, observed in the same specimen as in Fig. 7. (b) and (c): EDX spectra taken from the areas indicated by black spots in the α_B plate and the adjacent parent phase, respectively.

the EDX spectra obtained from the α_B plates larger than 30 nm in width are the characteristic ones of the plates, being independent of the surrounding matrices. The EDX spectra of (b) and (c) are of the K_α from Ag (the left hand side) and Cd (the right hand side) atoms. According to a thin foil approximation⁽²⁷⁾, the intensity of characteristic X-ray spectra is proportional to the compositions of the constituent elements. It may thus be known from (b) and (c) of both figures that the Cd content in the α_B plates is lower than that in the surrounding matrix. The Cd content in the surrounding matrix in the specimens heat-treated for shorter than 7.2 ks at 433 K was almost the same as that of the matrix in the as-quenched specimens because of the small amount of bainite plates.

Figures 9(a) and (b) show the EDX spectra, which were taken from the fcc α plate and its surrounding matrix in Fig. 6, respectively. Figure 9 indicates that the Cd content in the fcc

α plate is lower than that in the matrix. The Cd content in the matrix of this specimen was of the equilibrium β_2 phase with a much higher Cd content. All of EDX spectra in Fig. 9 as well as Figs. 7 and 8 were counted until they exhibited the Gaussian distribution, the integrated count number being about 10^4 . The composition thus analyzed is shown in Fig. 10, where the difference in Cd content between the α_B or α plate and the surrounding matrix is plotted as a function of width of the bainitic plates. The difference in Cd content at the most right hand side in Fig. 10 is of the fcc α phase, and it is shown on the basis of the Cd content of the matrix in the as-quenched specimens that is, by subtracting the Cd content of the bainite from that of the as-quenched matrix. It is thus known from the figure that the Cd content of the N9R α_B plates is nearly the same as that of the fcc α phase from the very early stage (about 30 nm width) of growth. It may therefore be concluded that 9R α_B plates are

Fig. 9 (a) and (b): EDX spectra taken from the areas indicated by black spots in the α_B plate and the adjacent parent phase in Fig. 6(a), respectively.

Fig. 10 The deficiencies of solute content in the α_B plates, compared with that in the surrounding parent phase, as a function of plate width.

martensitically created in the β_2 matrix regions low in Cd content, and that they grow holding the reduced Cd content or changing a little the

content at the growing interfaces due to a transformation stress⁽¹³⁾, finally transforming to the fcc α phase.

IV. Discussion

1. Martensitic nature of N9R α_B plates

The bainitic products in the Ag-45 at% Cd alloy at 433 K have been verified to possess an internally faulted N9R type LPSO structure and a plate shape with parallel straight interfaces. This crystal structure and morphology are very similar to those of monoclinic M9R or M18R martensites in other noble metal base β phase alloys. Recently, ordered domains in the 9R α_B plates of Cu-Zn-Al alloys have been demonstrated to be inherited from those in the parent phase⁽²²⁾. Therefore, the 9R α_B plates are considered to be a martensitic product, and then other observed features of the α_B plates will be discussed from such a point of view.

The observed N9R structure and plate morphology of α_B plates, however, were obviously different from an internally twinned 2H type structure and a spear morphology of the normal martensite produced in the sub-zero cooled Ag-Cd alloy. This means that the N9R product is not the characteristic martensite of the alloy and may be of a modified martensite. The modification from 2H to 9R martensites may come from a compositional change in the β_2 matrix prior to the transformation. It has been known in many alloy systems that the structure of martensites changes depending on the composition of the alloys. For example, Cu-Al alloys with higher Al content (26~28 at%) exhibit a 2H type γ'_1 martensite, while those with lower Al content (22~26 at%) do an 18R type β'_1 martensite⁽²⁸⁾⁽²⁹⁾. Similarly, Ag-Cd alloys with lower and higher Cd contents may possibly exhibit 9R and 2H type martensites, respectively. In fact, a 9R martensite has recently been verified to be stress-induced in the Ag-45 at% Cd alloy⁽³⁰⁾. These results, therefore, suggest that the 9R martensite can be formed even in the Ag-45 at% Cd alloy whose characteristic martensite is of a 2H structure, if the Cd content is locally decreased, raising the M_s temperature, or the M_s temperature

is increased by stress. In the present case, the diffusion of Cd atoms seems to occur first in the β_2 matrix at 433 K, reducing the Cd content, and then a martensitic transformation into the 9R structure occurs due to a rise of the M_s temperature in the regions reduced in Cd content. The results obtained by EDX analysis seems to support the local reduction in Cd content, because the Cd content in the 9R α_B plates was lower than that in the adjacent β_2 matrix from the very early stage of growth and was nearly equal to that of the equilibrium α phase.

As seen in Fig. 1, the 9R α_B plates is very small in size compared with that of 2H martensites. If the 9R α_B plates were formed in the original and homogeneous composition of the β_2 matrix, their sizes would be much larger like the 2H martensites. So, the small size of bainite also seems to support the local reduction in Cd content. Incubation periods forming a C curve in the T - T - T diagram for the bainitic transformation⁽²⁾⁽¹²⁾ might be needed for such a local diffusion of Cd atoms in the β_2 matrix.

2. Disordered structure of 9R α_B plates

Superlattice reflections characteristic of 9R structure and 1/3 and 2/3 reflections have not been observed in the ED patterns taken from α_B plates in the Ag-45 at% Cd alloy (Figs. 3 and 4), indicating that no ordered structure is inherited from the parent phase and no Cd atom is segregated on the every third layers of the α_B plates. An ordered structure and a third layer segregation of solute atoms have previously been observed in the 9R α_B plates in Cu-Zn(-Al)⁽⁸⁾⁻⁽¹¹⁾⁽²⁰⁾⁻⁽²²⁾ and Ag-Zn⁽²⁵⁾ alloys. Therefore, an ordered structure might be inherited and a third layer segregation might occur similarly in the 9R α_B plates of the present Ag-Cd alloy. However, they might be immediately destroyed after the martensitic transformation into 9R α_B , due to quick disordering and diffusion of Cd atoms in the plates. On the other hand, in Cu-Zn(-Al) and Ag-Zn alloys, they might be maintained for a while, due to not so quick disordering and diffusion of Zn atoms. In fact, it has been reported that diffusion rate of Cd solute in Ag solvent is faster

than that of Zn solute in Cu or Ag solvent⁽³¹⁾. Therefore, the fact that no ordered structure and no Cd atom segregation are observed in the 9R α_B plates in the Ag-Cd alloy does not deny the martensitic nature of the 9R α_B formation.

3. Anomalous reflection from β_2 matrix phase

In the above, local reduction of Cd content has been described to occur in the β_2 matrix prior to the formation of 9R α_B . Diffusion of Cd atoms in a more fine scale may also occur in different regions of the β_2 matrix, and may cause a spinodal-like decomposition or a precipitation phenomenon in those regions. Anomalous reflections observed in ED patterns, Figs. 3(c) and 4(c), in especially in 4(c), as mentioned already, are not of the CsCl type superlattice reflections from the matrix, and are considered to be a verification for such a spinodal-like decomposition or precipitation. Because the anomalous reflections can be explained by assuming a spinodal decomposition⁽³²⁾, precipitation of ω -like phase⁽³³⁾⁽³⁴⁾ or a hexagonal phase⁽³⁵⁾⁻⁽³⁷⁾. A basket weave image due to such a spinodal decomposition or precipitation is actually observed in the TEM images taken from the matrix, as seen in Figs. 2(a), 3(a) and 4(a). These also support the local diffusion of Cd atoms in the matrix prior to the bainite formation.

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