Electron Microscope Study on Fine Structures of the Austenite in an Ausaged Fe-Ni-Al Alloy*

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Fine structures of the austenite in an ausaged Fe-28.9Ni-5.7Al (mass%) alloy have been examined by means of conventional and high resolution transmission electron microscopy and electron diffraction. The ageing treatment was carried out at various temperatures in the range between 673 and 923 K for up to 2.88 Ms, which gave rise to complicated changes in the martensite-start temperature, M_s , of the alloy. Upon ageing, typically at 823 K, the austenite exhibited characteristic fine structures. That is, the conventional transmission electron micrographs showed striations in them and the corresponding electron diffraction patterns did extra reflections other than the fundamental austenite ones and $\langle 011 \rangle^*$ diffuse streaks. Detailed observations suggested that these fine structures were due to the formation of coherent precipitates with a Ll_0 type ordered structure during ausageing, not with the Ll_2 or DO_3 type one previously reported. Fine structures were also observed in the grown precipitates themselves on the later stage of ageing, and they were accounted for by the Ll_0 type ordered structure. The relation between the complicated variations in M_s and the microstructural change of austenite upon ausageing was well explained.

(Received October 20, 1987)

Keywords: ausageing, iron-nickel-aluminum alloy, martensitic transformation, transformation temperature, coherent precipitate, coherency strain, electron microscopy

I. Introduction

It has been reported so far⁽¹⁾⁻⁽⁹⁾ that in Fe-Ni based ternary alloys, such as Fe-Ni-Ti and Fe-Ni-Al, γ' precipitates with the Ll_2 type ordered structure are formed upon ausageing, and that they affect significantly the martensitic transformation occurring in those alloys upon subzero cooling, in respect of the martensite start temperature, M_s , and the morphology, substructure and crystal structure of martensites. According to previous studies by conventional transmission electron microscopy (CTEM), however, it appears that the precipitation process in those alloys proceed in somewhat

different manners. That is, spherical precipitates are formed in Fe-Ni-Ti alloys, being evenly distributed in the austenite matrix⁽⁴⁾⁽⁶⁾⁽⁹⁾, while cuboid ones are formed in Fe-Ni-Al alloys⁽¹⁾⁽³⁾. The austenite in the latter alloys exhibits marked striations along certain directions in CTEM images and also "satellite" reflections in electron diffraction (ED) patterns. Hornbogen and Meyer⁽¹⁾ reported that the satellites approached the respective principal reflections as ageing proceeds, as in spinodally decomposed alloys. Meanwhile, Gun'ko *et al.*⁽³⁾ explained the satellites as a cross-sectional view of diffuse streaks along $\langle 110 \rangle^*$ directions in reciprocal space cut by the Ewald sphere, and ascribed the origin to coherent shear strains produced by the concurrent formation of another precipitate named α' with the DO_3 type ordered structure. Ausageing effects in the former Fe-Ni-Ti alloys have so far been examined in some detail⁽⁴⁾⁻⁽⁹⁾, but those in the latter Fe-Ni-Al alloys have not so much⁽¹⁾⁻⁽³⁾. Hence, the nature of the diffuse streaks and the corresponding striations in the austenite of the latter alloys have not been well understood yet. Moreover, microstructural

* A part of this paper was originally presented at the Annual Autumn Meetings of the Japan Institute of Metals, Akita and Hiroshima, (1983), (1984).

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changes of the austenite upon ausaging have not been explored in connection with the complicated variations in M_s , as previously reported⁽²⁾. Therefore, the present investigation has been conducted in order to clarify those obscure points by means of CTEM and high resolution electron microscopy (HREM). The results obtained will be reported in this paper.

II. Experimental Procedure

An alloy ingot with a nominal composition of Fe-29Ni-6Al (mass%) was made by induction-melting electrolytic iron (99.9%), Mond nickel (99.97%) and high purity aluminum (99.99%) in argon atmosphere, and by casting the melt into an iron mould. Chemical analysis of the ingot showed the composition to be Fe-28.9Ni-5.7Al (mass%), which was roughly the same as those of the alloys previously studied⁽¹⁾⁽²⁾. After being hot-forged, the ingot was homogenized at 1473 K for 86.4 ks in an evacuated quartz tube, and then hot- and cold-rolled to a sheet of 0.3 mm in thickness, from which specimens with suitable sizes were cut for the electrical resistivity vs temperature measurement to determine M_s and for CTEM and HREM observations. All the specimens were solution-treated at 1473 K for 7.2 ks in evacuated quartz tubes and then quenched into iced water. Subsequently, a variety of ageing treatments were carried out at various temperatures in the range from 673 to 923 K for various periods up to 2.88 Ms in sealed quartz tubes back filled with argon.

CTEM and HREM observations were made at room temperature by using of electron microscopes of the HU-650 and H-700SS types. The former operating at 500 kV was used for the CTEM observation, and the latter at 200 kV, for the HREM observation.

III. Results

1. Change in M_s upon ausaging

M_s of the present alloy was about 210 K in the as-quenched state, but it varied in a complicated manner with ausaging, depending on

Fig. 1 Change in the M_s temperature of an Fe-28.9Ni-5.7Al (mass%) alloy upon ausaging at various temperatures as indicated in the figure. Plotted as a function of ageing time. Open symbols mean that M_s is below 77 K.

temperature and time of the ausaging, as shown in Fig. 1. M_s is raised by ageing at 923 K, but it is depressed once below 77 K and then raised again above that temperature after a certain period by ageing at 823 K. Upon ageing at 673 K, however, it remains below 77 K even after 2.88 Ms. The overall feature of the variations in M_s of the present ausaged alloy is thus consistent with that of the previous reports⁽¹⁾⁽²⁾.

2. Microstructural change in CTEM image

Figure 2(a) and (b) show a CTEM image of the austenite in an as-quenched specimen and the corresponding ED pattern, respectively. The austenite in the as-quenched state appears to be of a homogeneous solid solution.

Figure 3(a) and (b) show a CTEM image and the corresponding ED pattern, respectively, of the austenite in a specimen subjected to ageing at 823 K for 3.6 ks, by which M_s was lowered down to about 125 K, as seen from Fig. 1. The microstructure of the aged austenite, (a), does not appear much different from that in Fig. 2(a). However, in Fig. 3(b), some extra reflections are recognised besides the fundamental austenite ones. The extra reflections have so far been interpreted as superlattice ones of coherent γ' -Ni₃Al precipitates with the L1₂ type ordered structure, which are formed upon ausaging⁽¹⁾⁽³⁾. In fact, the appearance of those extra reflections in various orientations of

Fig. 2 CTEM image (a) and the corresponding ED pattern (b) of the austenite in an as-quenched specimen, showing that the austenite is almost of a homogeneous solid solution.

austenite was apparently consistent with that from the $L1_2$ ordered structure. Then, the reflections from the precipitates would be indexed, by referring to the FCC lattice. However, close examination on other fine structures associated with the formation of the precipitates, which will be shown later, suggests that the precipitates possess the $L1_0$ type ordered structure, as will be discussed in detail later.

In Fig. 3(b), besides the extra reflections, are noted spike-like diffuse streaks around the fundamental austenite reflections, especially those off the Bragg positions in the $\{011\}$ orientation. It is thus seen that the diffuse streaks do not exist in the exact $\{011\}$ orientations. The direction along which the diffuse streaks are observed will be examined later.

Figure 4(a) is a CTEM image of the austenite in a specimen ausaged at 823 K for 90 ks. It is

Fig. 3 CTEM image (a) and the corresponding ED pattern (b) of the austenite in a specimen aged at 823 K for 3.6 ks. Note some extra reflections (not the set of arrowed ones from a surface film) other than the fundamental austenite ones and also spike-like diffuse scattering around the austenite ones.

to be noted here that the austenite exhibits a characteristic substructure, i.e., marked striations. Such a substructure has already been observed⁽¹⁾⁽³⁾, but its crystallographic properties have not been examined in detail yet. In the present study, they were explored by the trace analysis, using several CTEM images with different orientations. As a result, it was found that the striations were parallel to the $\{011\}$ plane traces. Hence, it is seen in (a), which is of nearly the $\{011\}$ orientation, that the striations are parallel to the $\langle 111 \rangle$ directions. Figure 4(b) shows an ED pattern corresponding to (a), and it is a little deviated from one of the $\{011\}$ orientations around the $\langle 001 \rangle^*$ direction. To be noted here is that satellite-like reflections appear around the austenite reflections off

Fig. 4 CTEM image (a) and the corresponding ED pattern (b) of the austenite in a specimen aged at 823 K for 90 ks. Note the severely striated substructure along $\langle 111 \rangle$ directions in (a) and also "satellite reflections" appearing along $\langle 112 \rangle^*$ directions in (b). See the text for detail.

their Bragg positions. Since substantially no satellites are visible around the systematic reflections along the $\langle 001 \rangle^*$ direction, it is seen that the satellites are a cross-sectional view of streaks cut by the Ewald sphere. Moreover, it is seen in (b) that the satellites appear along the $\langle 112 \rangle^*$ directions which are perpendicular to the striations along the $\langle 111 \rangle$ directions in the corresponding CTEM image of (a). These observations indicate that the diffuse streaks run along the $\langle 011 \rangle^*$ directions. In fact, the appearance of the diffuse streaks in various orientations may be explained by the $\langle 011 \rangle^*$ streaks. This result is consistent with that by Gun'ko *et al.*⁽³⁾

Figure 5(a) shows a CTEM image of the austenite in a specimen subjected to ageing at

823 K for 263 ks, by which M_s was raised up above 77 K again, i.e., about 160 K. The striated substructure of the austenite is similar to that shown in Fig. 4(a). Figure 5(b) shows the corresponding ED pattern nearly in the $\langle 110 \rangle$ orientation, where the austenite reflections along the $\langle 110 \rangle^*$ direction are strongly excited. Here, it is interesting to note that the streaks are absent along the specific $\langle 110 \rangle^*$ direction. This suggests that the appearance of the $\langle 110 \rangle^*$ streaks obeys an extinction rule analogous to that for thermal diffuse scattering due to transverse acoustic phonons of the $\langle 011 \rangle^* \langle 0\bar{1}\bar{1} \rangle$ mode⁽¹⁰⁾. If this is the case, the $\langle 011 \rangle^*$ streaks in the ED patterns and the corresponding striations in the CTEM images indicate that there exist $\{011\}\langle 0\bar{1}\bar{1} \rangle$ static shear strains in the austenite matrix.

Figure 5(c) shows a dark field CTEM image formed by an 110 type superlattice reflection of precipitate, which is encircled in (b). It is seen in (c) that the precipitates are still extremely fine, i.e., only a few nm in size, and that they are evenly distributed in the austenite matrix.

CTEM images taken from the austenite of a 923 K-aged specimen exhibited similar substructures to those shown in Figs. 4 and 5, when the ageing time was limited less than roughly 3.6 ks. The prolonged ageing, however, brought about a different microstructure. Figure 6 shows an example of the CTEM images, which was obtained from the austenite aged at 923 K for 54 ks. As seen in (a), the precipitates grew a little larger to form a rod-like morphology, elongating along the $\langle 001 \rangle$ directions of precipitate. The corresponding ED pattern shown in (b) consists of two sets of reflections, one being from the precipitate and the other from a BCC phase, as indexed. The BCC phase is supposed to be the martensite, judging from the rise in M_s with ageing, as shown in Fig. 1. Here, it is to be noted that the reflections from the precipitates are still accompanied by the $\langle 011 \rangle^*$ streaks in spite of the fact that the matrix phase for the precipitates has changed from austenite to martensite. This indicates that the origin of the $\langle 011 \rangle^*$ streaks still exists in the precipitates themselves. It is further to be noted in (b) that the 002 reflection of precipitate makes doublets. On the other

Fig. 5 Bright field CTEM image (a) of the austenite in a specimen aged at 823 K for 263 ks, the corresponding ED pattern (b) and dark field CTEM image formed by an 110 type (encircled in (b)) reflection of precipitate (c). Note the absence of the $\langle 011 \rangle^*$ streaks along particular $\langle 110 \rangle^*$ directions in (b), and also the extremely fine precipitates, a few nm in size, evenly distributed in the austenite matrix in (c).

hand, (c) is a dark field CTEM image from the area identical to (a), which was formed by a 001 type reflection of precipitate. It appears that the rod-like precipitates consist of fine segments with different orientations, the segments being roughly several nm in size. These fine structures in the grown precipitates will be discussed later in terms of the ordered structure of the precipitates.

3. Microstructure observed by HREM

Figure 7(a) is a lattice image by HREM of the austenite in a specimen as-quenched, which was formed with the transmitted and 111 diffracted beams in the $\langle 211 \rangle$ orientation. The spacing of the lattice fringes corresponds to 0.21 nm of $\{111\}$ planes (lattice parameter of

the as-quenched austenite was determined by X-ray diffraction to be 0.36 nm). To be noted here is that the lattice fringes are a little wavy, suggesting that the austenite lattice is distorted locally. However, since no extra reflections indicative of the formation of precipitates are observed in the as-quenched state, as mentioned before, the local lattice distortion is supposed to be due to the presence of solute atom clusters, which are possibly less than 2 nm in size.

The lattice image shown in Fig. 7(b) is of the austenite in a specimen aged at 673 K for 263 ks, which was formed by the transmitted and three $\bar{1}1\bar{1}$, $\bar{1}11$ and 002 diffracted beams. The austenite thus aged exhibits substantially the same substructures and diffraction effects as

Fig. 6 Bright field CTEM image of a specimen aged at 923 K for 54 ks (a), the corresponding ED pattern (b) and dark field CTEM image formed by a 001 type (encircled in (b)) reflection of precipitate (c).

those shown in Fig. 3. Hence, strain contrasts visible in (b) are supposed to be due to the existence of precipitates, which are still possibly less than 2 nm in size.

Figure 7(c) shows another lattice image of the austenite in a specimen aged at 923 K for 1.8 ks in the $\langle 110 \rangle$ orientation, the fringes corresponding to the $\{111\}$ planes of austenite. The CTEM images and the corresponding ED patterns of the austenite thus aged were apparently similar to those shown in Fig. 5. Here are observed regions with darker contrasts, which are elongated along the $\langle 111 \rangle$ direction and are roughly 2.5 nm wide and 5 nm long. The darker contrasts appear to correspond to the precipitates. Since the $\langle 111 \rangle$ directions are parallel to the $\{011\}$ plane traces in the $\langle 110 \rangle$

orientation, the lattice image in (c) probably indicates that the precipitates form preferentially on the $\{011\}$ planes of austenite.

IV. Discussion

1. Crystal structure of the precipitate

The precipitates formed in the ausaged Fe-Ni-Al alloys have so far been reported to be of the γ' -Ni₃Al phase with the $L1_2$ type ordered structure⁽¹⁾⁽³⁾, like γ' -Ni₃Ti precipitates in Fe-Ni-Ti alloys⁽⁴⁾. In fact, the superlattice reflections observed in ED patterns of the present ausaged Fe-Ni-Al alloy were apparently consistent with those from the $L1_2$ type precipitate. However, if this were true, isotropic volume

Fig. 7 Lattice images of the austenite in the specimen as-quenched (a), aged at 673 K for 263 ks (b) and aged at 923 K for 1.8 ks (c).

strains due to the difference in lattice parameter between the precipitate and the austenite would be produced, and a mottled structure would be observed in CTEM images of the austenite, as actually observed in Fe–Ni–Ti alloys⁽⁴⁾⁽⁶⁾. Furthermore, it has been shown that the volume strains cause diffuse scattering along the radial directions in reciprocal space around the austenite reflections⁽¹¹⁾, as observed also in Fe–Ni–Ti alloys by X-ray diffraction⁽⁷⁾. However, unlike the Fe–Ni–Ti alloys, the present Fe–Ni–Al alloy exhibited marked striations along $\{011\}$ plane traces in the CTEM images and strong $\langle 011 \rangle^*$ streaks in the corresponding ED patterns, as shown in the preceding section. These facts, together with the extinction rule observed for the $\langle 011 \rangle^*$ streaks, indicate that $\{011\}\langle 01\bar{1} \rangle$ shear strains exist in the austenite lattice of the present ausaged Fe–Ni–Al alloy, and therefore that the coherent precipitates in the alloy must produce the shear strains. Gun'ko *et al.*⁽³⁾ reached the same conclusion, and proposed a $D0_3$ type ordered structure for the coherent precipitates. However, this is not the case in the present alloy, because no superlattice reflections indicative of the formation of $D0_3$ type precipitates were observed in the ED patterns obtained, and furthermore the $D0_3$ precipitate does not seem to produce the shear strains, because of cubic symmetry. It is thus conceivable that the coherent precipitates in the

present alloy take a Ll_0 type ordered structure, because the Ll_0 structure is of the tetragonal symmetry, as explained below.

If the coherent Ll_0 type precipitates are formed in the austenite matrix, tetragonal coherency strains are built up around the precipitates along the $\langle 001 \rangle$ directions of austenite. The tetragonal coherency strains are able to be relaxed by $\{011\}\langle 01\bar{1} \rangle$ shears, because this is energetically favorable if the anisotropy factor of the austenite lattice is larger than unity. Such a relaxation of the tetragonal coherency strains probably occur and leave the shear strains in the present ausaged Fe–Ni–Al alloy, since the anisotropy factor of Fe–Ni binary alloys undergoing a martensitic transformation has been known to be about 5⁽¹²⁾.

By the way, when the Ll_0 type ordered precipitates are coherently formed in a FCC matrix, their ED patterns are indistinguishable from those of the Ll_2 type ordered ones, so far as the appearance of superlattice reflections is concerned, as mentioned in the preceding section. Such an example is found also in the early stage of ordering in Cu–Au alloys⁽¹³⁾. In the Cu–Au alloys, Ll_0 type CuAu(I) ordered domains are coherently formed, and striations parallel to $\{011\}$ planes are observed in the disordered FCC matrix, as in the present alloy. Furthermore, the Ll_0 ordered domains are observed to nucleate on the $\{011\}$ planes of the

disordered matrix⁽¹⁴⁾. This is also very similar to the present observation that the coherent precipitates are formed on $\{011\}$ planes of the ausaged matrix (see Fig. 7(c)).

The tetragonal coherency strains along the $\langle 001 \rangle$ directions of austenite are considered to increase, as the precipitates grow larger, and eventually the tetragonal coherency is lost. In fact, the precipitates shown in Fig. 6(a) appear to become incoherent with the matrix, because they have grown and moreover the matrix has become the BCC phase. It is thus considered that the split 002 reflections of the precipitates seen in Fig. 6(b) correspond to the 002 and 200 ones of the tetragonal Ll_0 type ordered (Ni, Fe) Al phase. The bright regions seen in the dark field CTEM image of Fig. 6(c) clearly suggest that the largely grown precipitates consist of fine twins on the $\{011\}$ planes. Such fine twins cause $\langle 011 \rangle^*$ streaks around reflections from the precipitates, as actually observed in Fig. 6(b). These are consistent with the Ll_0 type ordered structure of the precipitates.

The Ll_0 type (Ni, Fe)Al phase does not exist as a thermal equilibrium phase⁽¹⁵⁾. However, it possibly exist as an intermediate phase, like the Ll_2 type γ' -Ni₃Ti phase in Fe-Ni-Ti alloys, in which the thermal equilibrium phase with the composition of Ni₃Ti is of the $D0_{24}$ type⁽¹⁶⁾. It is thus reasonable to conclude that the coherent precipitates formed in the present ausaged Fe-Ni-Al alloy possess the Ll_0 type ordered structure.

2. Relation between the change in M_s and the microstructure of austenite

In the early stage of ageing at 823 and 673 K, M_s was depressed markedly as seen in Fig. 1. Meanwhile, the ED patterns taken at this stage exhibit superlattice reflections indicative of the formation of Ll_0 type precipitates in the austenite. But the precipitates are so fine that the tetragonal coherency strains built up around them are very small. Hence, the relaxation in the austenite by $\{011\}\langle 01\bar{1} \rangle$ shears does not take place predominantly. In fact, striations along $\{011\}$ plane traces were hardly observed in the CTEM images of austenite at this stage. However, the fine precipitates are considered to be highly coherent with the austenite

lattice, and so the martensitic transformation is suppressed by their existence to a great extent, because extra energy is needed so as for the precipitates to transform together with the matrix. This is believed to be the reason for the marked depression of M_s at this stage.

With further ageing, the precipitates grow larger, and consequently the tetragonal coherency strains are expected to increase. Thus, the strain relaxation by $\{011\}\langle 01\bar{1} \rangle$ shear becomes predominant, resulting in marked striations in CTEM images of the austenite matrix. The strain relaxation is supposed to make the precipitates semicoherent or incoherent with the austenite matrix. Such precipitates become ineffective in suppressing the martensitic transformation. Concurrently, the growth of precipitates causes a depletion of solute concentration in the austenite matrix. Therefore, M_s is expected to be raised again, as actually observed. The reason for the increase of M_s upon ageing at 923 K is considered in the same way as above.

V. Conclusions

Fine structures of the austenite in an ausaged Fe-Ni-Al alloy, which are associated with complicated changes of M_s upon ausaging, have been explored by CTEM and HREM. The main results obtained are as follows:

(1) The salient striations observed in the CTEM images of austenite, typically at 823 K, were parallel to $\{011\}$ plane traces. The strong $\langle 011 \rangle^*$ streaks observed in the corresponding ED patterns seemed to appear with an extinction rule analogous to that for thermal diffuse scattering due to transverse acoustic phonons of $\langle 011 \rangle^* \langle 01\bar{1} \rangle$ mode. These observations indicate that $\{011\}\langle 01\bar{1} \rangle$ shear strains exist in the austenite lattice.

(2) The generation of the shear strains was well accounted for by assuming that the precipitates formed in the ausaged alloy possess a Ll_0 type ordered structure, not the Ll_2 type one reported so far. The assumption was consistent with the observed fine structures in the austenite and the precipitate itself.

(3) The decrease in M_s , as observed in the

early stage of ageing at 673 and 823 K, was ascribed to the formation of the fine precipitates coherent with the austenite lattice. On the other hand, the increase in M_s , as observed in the later stage of ageing at 823 and 923 K, was ascribed to the coherency loss due to the relaxation of the coherent strains, which increase with the size of precipitates, by $\{011\}\langle 01\bar{1}\rangle$ shears, and to some depletion of solute atoms in the austenite matrix accompanying the growth of precipitates.

Acknowledgements

The authors appreciate a useful discussion with Professor H. Chen at University of Illinois. The present work was partly supported by the Grant-in-Aid for Fundamental Scientific Research (Ippan A, 1980–81, and Ippan B, 1985–86) from the Ministry of Education, Science and Culture, Japan.

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