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Investigation on the formation of serrated grain boundaries with grain boundary characteristics in an AISI 316 stainless steel

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ABSTRACT

The formation of serrated grain boundaries (GBs) depending on the GB characteristics has been investigated by using an electron backscattered diffraction (EBSD) technique and a transmission electron microscopy (TEM) in an AISI 316 stainless steel. It was observed that at the early stage of aging treatment, the GB morphology was changed from flat to wavy at random GBs without any indication of $M_{23}C_6$ carbide formation, and no GB serration at special GBs (lower than $\Sigma 29$) was found. The comparison study on the misorientation angle between two neighboring grains indicated that the occurrence of GB serration at random GBs is attributed to the reduction of the total GB energy. Random GBs with high energy tend to be serrated, resulting in the formation of two segments with lower energies. On the other hands, the special GBs may be less likely to form serrated GBs due to their lower GB energy.

1. Introduction

It has been reported that grain boundary (GB) serrations occur by the interaction between GB and second phases at the boundary, and are frequently observed in superalloys [1–3] and stainless steels [4,5]. Tanaka and co-workers have observed that when GB serration occurs in an austenitic stainless steel, creep property is increased and they suggested that the GB serration may disturb GB sliding [4]. Recently, it was found that the GB serration occurs at the early stage of aging treatment prior to the $M_{23}C_6$ precipitation in an AISI 316 stainless steel [6]. And, it was also reported that the GB serration strongly affects the density of GB carbides and its morphology. Moreover, it was observed in an AISI 304 stainless steel that when the GB serration occurs, carbide density is significantly decreased and carbide morphology is changed from triangle to planar shape, resulting in a remarkable improvement of the creep-fatigue resistance [7]. In spite of the fact that the GB serration is one of the most important factors to determine the mechanical properties in many alloys, most of the investigators have studied the mechanism of GB serration not for the random GBs but for the special GBs [8–10]. Hence, up to now, the investigation for GB serration mechanism for random GB has not been yet extensively carried out.

Therefore, the purpose of the present study is to investigate the GB serration both for special and random GBs in an AISI 316 stainless steel.

2. Experimental procedures

The chemical composition of the investigated AISI 316 stainless steel is given in Table 1. In order to investigate the GB serration behavior, all the specimens were solution-treated at 1323 K for 1 h under air atmosphere. The specimen was then aged at 1033 K for various time periods. And, in order to investigate the sequential development of GB serration with increasing aging time, the specimen sealed within a quartz tube under 10^{-5} torr was isothermally aged 1033 K with increasing exposure time. This allowed step-by-step observation on the serration of a selected GB and subsequent $M_{23}C_6$ carbide precipitation.

Measurements on the GB characteristics were carried out in a scanning electron microscope (SEM) Philips XL30 field emission gun (FEG) utilizing electron backscattered diffraction (EBSD) technique. Transmission electron microscope (TEM) investigations were conducted to obtain crystallographic features of the serrated GBs using Philips CM20 electron microscope operating at 200 kV. The serrated GB plane normal was obtained from the cross product of at least two beam directions to which the GB plane is parallel. In this narrowest boundary area situation, the GB is seen as a line. This method has been previously used for other alloy systems [11,13].

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Table 1
The chemical composition of an AISI 316 stainless steel (all in wt%).

C	Si	Mn	P	S	Cr	Ni	Mo
0.067	0.6	1.3	0.04	0.02	16.9	10.8	2.12

3. Results and discussion

3.1. The behavior of GB serration at CSL and random GBs

Fig. 1 shows the GB morphologies in an isothermally heat treated 316 stainless steel. The specimen was solutionized at 1323 K for 1 h in air atmosphere followed by aging at 1033 K for 60 min. It is observed that some GBs were considerably serrated while some other GBs were not serrated even though heat treatment condition was same. Therefore, in order to verify the reason why GB serration was partially occurred under the same heat treatment condition, the present authors paid attention to investigate the GB serration behaviors at coincidence site lattice (CSL) and random GBs. In general, since the misorientation across an individual GB is mostly represented by a Σ value originated from CSL model, GBs can be divided into two groups of CSL and random GBs. The criterion of the Σ value determining boundary type was 29 in the present study [12]. Fig. 2 shows the neighboring GB morphology change with GB characteristics after the heat treatment. As shown in Fig. 2, it is observed that the GB serration is strongly related with the GB characteristics, i.e. in the case of CSL GBs, the initial straight GB remained to be straight while the random GB was considerably serrated after aging treatment at 1033 K for 60 min.

Fig. 3 shows the typical GB serration in an AISI 316 stainless steel. The misorientation angles of the serrated GB and planar GB by using special TEM investigation [11,13] were measured in an attempt to compare the GB energies of the planar and serrated GBs. The result is summarized in Table 2. It is observed that the misorientation angle can be decreased after occurrence of the GB serration. And, also the present authors investigated that density of the CSL points of serrated and planar GB on the basis of the HRTEM

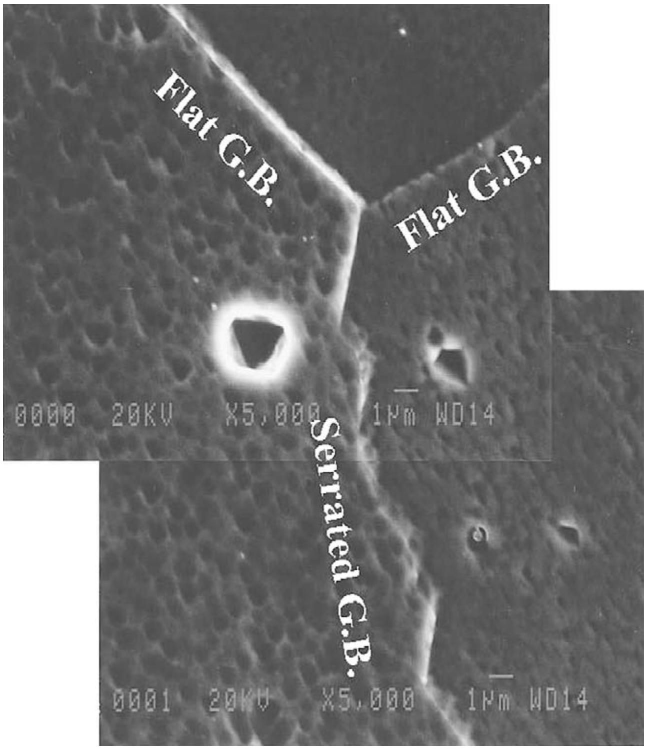

Fig. 1. The grain boundary morphologies of AISI 316 stainless steel solution-treated at 1323 K at 1 h and then aged at 1033 K for 60 min.

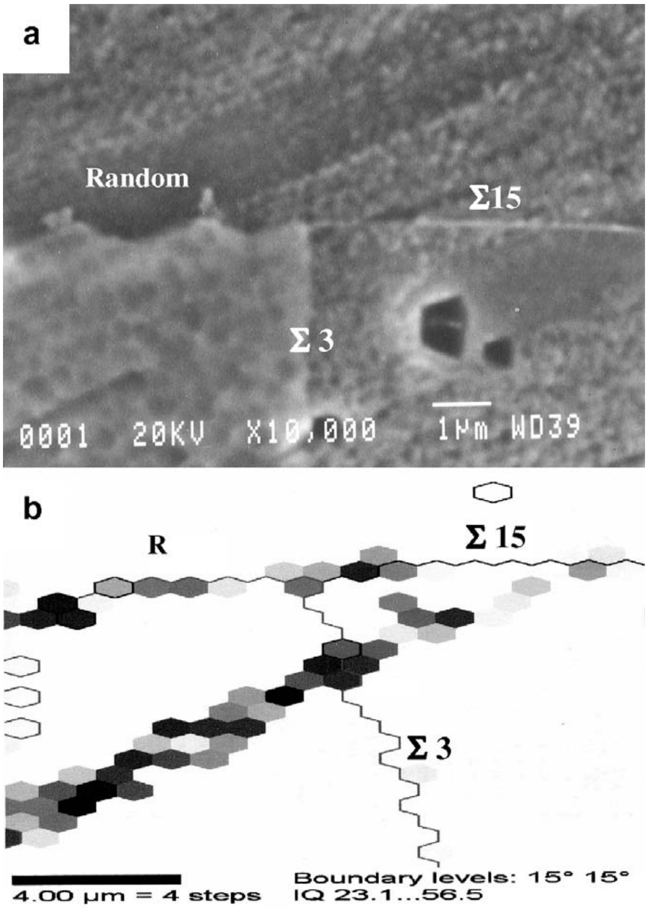

Fig. 2. Grain boundary configuration with grain boundary characteristics, (a) SEM micrograph and (b) OIM of correspondent region of (a).

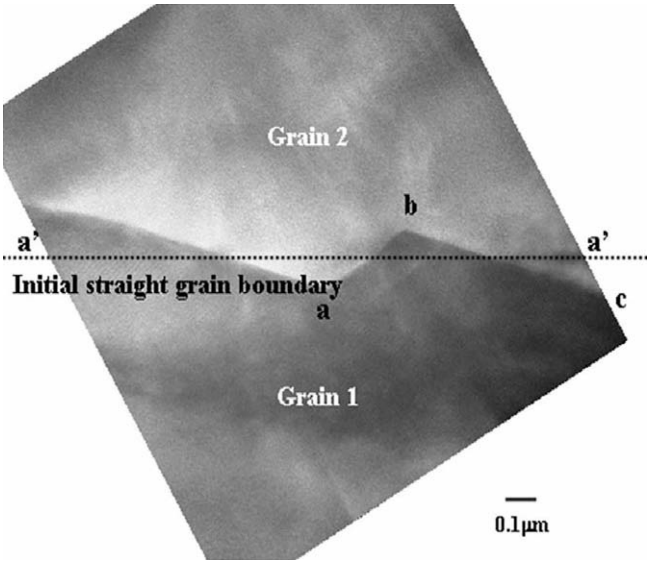

Fig. 3. TEM micrograph showing the typical grain boundary serration. Note that $\overline{ab}$ and $\overline{bc}$ lines correspond to serrated GBs, and $\overline{a'a'}$ is the estimated location of the initial planar grain boundary.

Table 2
Misorientation angles between grain 1 and grain 2, depending on the type of grain boundary in Fig. 3.

Grain boundary type	Misorientation angle between the grain 1 and grain 2
$\overline{ab}$ Serrated segment	23.7°
$\overline{bc}$ Serrated segment	12.1°
$\overline{a'a'}$ Planar segment	34.2°

photograph, respectively [13]. From the result of the previous report, it can be summarized that the density of the CSL points of the serrated GB is much higher than that of the planar GB. In general, the boundary energy is one of the most suitable parameters that represent the character of a GB determined by the crystallographic arrangement of atoms in the boundary. A higher degree of disorder in the atomic arrangement along a GB may result in higher boundary energy. Therefore, for a given alloy system, GB energy may be strongly related with misorientation angle and atomic arrangement between neighboring grains. Hence, it may be suggested that the GB serration is a preferable phenomenon to reduce the boundary energy at intermediate temperature ranges, since the misorientation angle is observed to be decreased and atomic matching is increased after the boundary serration. So, it is expected that since the random GBs, whose misorientation angle is high and atomic matching is low, may have higher GB energy, they may be preferentially serrated to decrease the boundary energy. On the other hands, because the CSL boundaries with lower misorientation angle and higher atomic matching have lower GB energy, they may not have enough driving force for the boundary to be serrated. Therefore, it is suggested that the initial GB energy is driving force for the GB serration which reduces the higher GB energy to the lower one by modification of the misorientation angle and atomic mismatching between the neighboring grains.

3.2. The effect of aging time on the GB serration

Fig. 4 shows the progressive morphological change of random GB under isothermal aging treatment at 1033 K with increasing aging time. As shown in Fig. 4(a), the initial straight GB is observed after the solution treatment. And as aging time increases, it is ob-

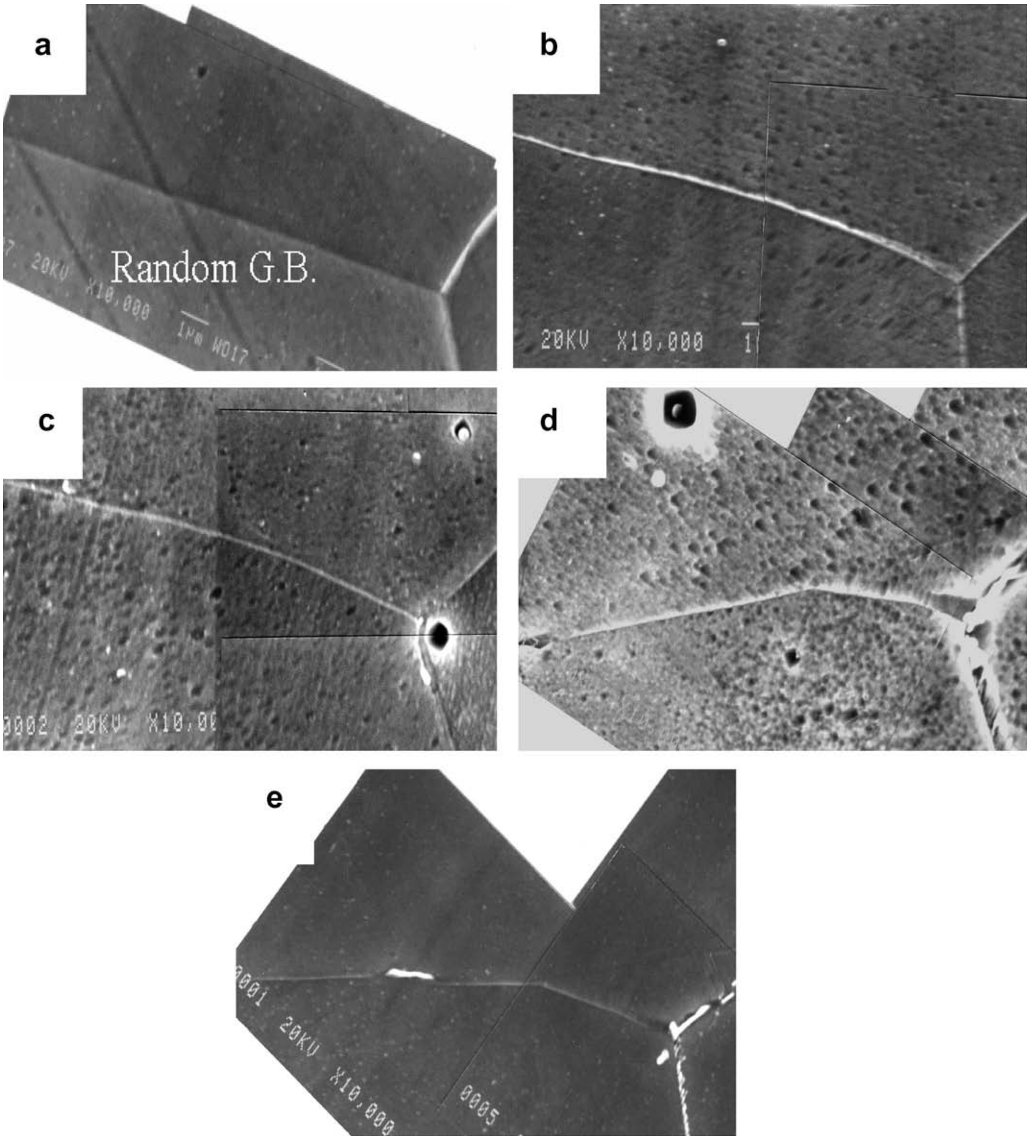

Fig. 4. Behavior of the random grain boundary with isothermal aging treatment at 1033 K for increasing aging time, (a) after solution treatment at 1323 K, (b) aged for 45 min, (c) additionally aged 5 min, (d) additionally aged for 5 min, and (e) additionally aged for 20 min.

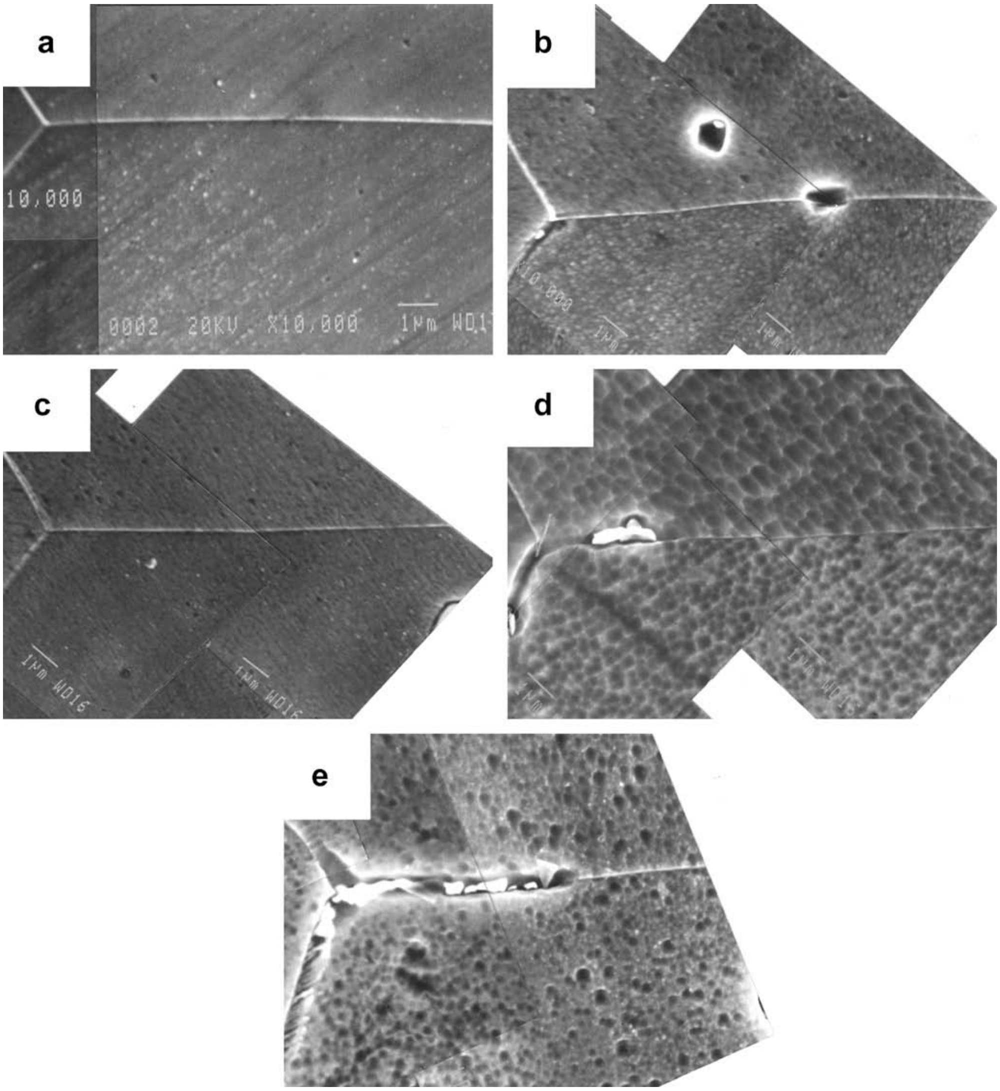

Fig. 5. Behavior of the CSL grain boundary with isothermal aging treatment at 1033 K for increasing aging time, (a) after solution treatment at 1323 K, (b) aged for 45 min, (c) additionally aged for 5 min, (d) additionally aged for 5 min, and (e) additionally aged for 20 min.

served that the straight GB is still maintained during aging treatment as shown in Fig. 4(b) and (c). However, as shown in Fig. 4(d), it is clearly seen that the straight GB is beginning to be serrated without any indication of carbide formation at this GB and then, after the extended aging treatment, planar type carbides form at the serrated GB as shown in Fig. 4(e). This observation is different from the previous investigations [1–5] reporting that the GB serration occurred after formation of the second phases at the boundary. On the other hands, compared with random boundary, the behavior of the $\Sigma15$ CSL boundary is quite different. As shown in Fig. 5(a)–(c), the initial straight GB is also observed after solution treatment and the straight GB is still maintained during aging treatment. However, under the same aging condition, as shown in Fig. 5(d) and (e), it is clearly seen that carbides form at the initial straight GB without any indication of the GB serration. In other words, CSL boundary is not serrated. From these observations, it can be suggested that in the case of the random GB, the initial straight GB is serrated at first and then carbides form along this serrated GB. On the other hands, in the case of CSL boundary, the GB may not be serrated, since the driving force for the GB serration may not be high enough.

4. Conclusions

(1) The GB serration occurred at random GBs with Σ value of 29 higher in an 316 stainless steel during aging treatment. No GB serration at special boundaries with Σ value of 29 lower was found.
(2) It can be inferred from the study on the misorientation that the occurrence of GB serration at random GBs may be attributed to the reduction of the total GB energy.
(3) Random GBs were serrated without any indication of $M_{23}C_6$ carbide formation. After the substantial GB serration, the carbides began to form at the serrated GBs.

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