The Temperature Dependence of Abnormal Grain Growth and Grain Boundary Faceting in 316L Stainless Steel

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When commercial 316L stainless steel specimens are heat-treated in a single phase state at 1 100°C, abnormal grain growth (AGG) occurs and some grain boundaries are observed to be faceted with hill-and-valley structures in transmission electron microscopy. Some segments of these faceted grain boundaries are expected to be singular. When heat-treated at 1 300°C normal grain growth occurs with all grain boundaries smoothly curved. These grain boundaries are expected to be atomically rough. At 1 200°C AGG still occurs but there is no excessively large grain in the specimen heat-treated at 1 100°C. This correlation between the grain boundary structure and grain growth is consistent with those observed previously in pure metals, oxides, and a single phase model alloy. The occurrence of AGG with faceted grain boundaries is attributed to grain boundary movement with boundary steps either produced by two-dimensional nucleation or existing at the junctions with dislocations. As the grain boundaries become rough at 1 300°C normal growth occurs because the grain boundaries migrate continuously with their rate expected to increase linearly with the driving force arising from the size difference. If a specimen heat-treated at 1 100°C is further heat-treated at 1 300°C, the AGG mode appears to switch to normal growth.

KEY WORDS: 316L stainless steel; abnormal grain growth; grain boundary faceting.

1. Introduction

Abnormal grain growth (AGG) has been observed in many pure metals such as Ag,1–3) Cd,4) Cu,5–7) Ni,8,9) Fe,10) Pb,4) Sn.11) It was recently observed by Lee and co-workers9) that when Ni was annealed in a carburizing atmosphere AGG occurred at temperatures below 0.7\(T_m\) and normal growth above 0.7\(T_m\), where \(T_m\) is the melting point. When AGG occurred, all or a fraction of the grain boundaries were found to have faceted hill-and-valley structures. When normal growth occurred, all grain boundaries were defaceted with smoothly curved shapes. Similar variation of the grain growth behavior and grain boundary structure with the annealing temperature was also observed in Ag.3)

A model Ni-base superalloy showed AGG during heat-treatment at 1 200°C (above the solvus temperature of the \(\gamma'\) precipitate) with grain boundaries mostly faceted and normal growth at 1 300°C with all grain boundaries defaceted.12) Similar correlations between grain boundary structure and grain growth behavior were also found in alumina13) and BaTiO\(_3\).14)

It was proposed9,15) that at least some segments of the faceted grain boundaries were likely to be singular corresponding to cusps in the polar plot of the grain boundary energy against the boundary normal. These atomically flat boundaries are expected to move by nucleation of boundary steps. Because of the exponential dependence of the boundary velocity on the driving force arising from grain size difference, a few large grains could grow at accelerating rates, producing AGG behavior. Even with dislocations at grain boundaries produced by slight deformation, AGG was still found to occur in Cu.16) If grain boundaries were defaceted at high temperatures, the grain boundaries were atomically rough and hence their velocity would increase linearly with the driving force. Then normal growth was expected in agreement with the observations. The analogies to the crystal surface roughening transition and changes in the crystal growth mechanism have been pointed out.9)

The purpose of this work is to observe the grain boundary defaceting transition and its effect on grain growth in a commercial alloy in the temperature ranges where the alloy is a single phase. A 316L stainless steel has been selected because its precipitates and their solvus temperatures have been relatively well characterized. This alloy is known to be free of precipitates at temperatures above 1 070°C17,18) and to melt at about 1 400°C. Therefore, the temperatures of 1 100, 1 200, and 1 300°C were selected for heat-treatments to observe possible grain boundary defaceting transition and changes of the grain growth behavior. Previously, Mizera et al.19) observed apparent transitions from normal to abnormal grain growth between 900°C and 1 200°C. But no explanation was given for this transition from normal to abnormal growth with temperature increase. Kashyap and Tangri20) also examined grain growth in 316L stainless steel...
with or without added boron. While apparently ignoring the occurrence of AGG, they observed discontinuous changes in the activation energies for grain growth at about 1200°C.

2. Experimental Procedure

A plate of 316L stainless steel of 6 mm in thickness was obtained from Pohang Iron and Steel Company. Its composition is given in Table 1. The plate was cut into approximately 7×7×6 mm pieces, which were heat-treated without any deformation. The specimens were heat-treated at 1100, 1200, 1300°C in a horizontal tube furnace under a flowing Ar atmosphere. They were rapidly pushed into the center of the preheated furnace and quenched in water after the heat-treatments. The heat-treated specimens were examined under a transmission electron microscope (TEM). The grain boundaries were examined on micrographs using a digitizer connected to a personal computer. The sizes of about 100–300 grains were measured in each specimen.

3. Results and Discussion

The grains in the as-received plate were relatively uniform in size with an average value of about 40 μm, but there were clusters of fine grains in some regions as shown in Fig. 1. The X-ray pole figures did not show any texture in these specimens. The characteristics of grain growth behavior were found to depend on the heat-treatment temperature. During the heat-treatment at 1100°C, AGG occurred as shown in Fig. 2. After heat-treating for 30 min at 1100°C some large grains appeared as shown in Fig. 2(b) and these apparently continued to grow to larger sizes during the heat-treatments for 60 min and 24 h as shown in Figs. 2(c) and 2(d), respectively. The grain size distributions shown in Fig. 3 confirmed the presence of very large grains exceeding 700 μm in size in the specimen heat-treated for 24 h. The large grains appearing after heat-treating for 60 min and 24 h are indicated by arrows in Fig. 3. The specimens in these experiments will be designated by their heat-treatment temperature and time; thus 1100°C/24 h for the specimen heat-treated at 1100°C for 24 h, for example.

Typical grain boundary shapes in the 1100°C/60 min specimen (Fig. 2(c)) are shown in Fig. 4. It shows a faceted grain boundary with hill-and-valley structure meeting two smoothly curved grain boundaries at a triple junction. The orientations of the adjacent grains were checked by electron diffraction to make sure that the faceted boundaries were not incoherent twin boundaries. Out of about 30 grain boundaries examined under TEM, 6 of them showed such a faceted structure. The heights of the hill-and-valley structures ranged from about 0.1 to 0.2 μm and the lengths from about 1 to 3 μm. The smoothly curved grain boundaries are expected to be atomically rough. The occurrence of AGG in these specimens with some faceted grain boundaries is consistent with the previous observations in Ni,9) a Ni-base superalloy,12) Cu,7) alumina,13) and BaTiO3.14) In Ni the fraction of the faceted grain boundaries was observed to decrease with increasing heat-treatment temperature,9) but AGG still occurred at relatively high temperatures where the fraction of the faceted grain boundaries was quite small.

The faceting of a grain boundary between two grains of a certain misorientation angle depends on the equilibrium shape of one of the grains embedded in the other grain with the same misorientation angle. This equilibrium shape in turn is related to the polar plot (the γ-plot) of the grain boundary energy against the grain boundary normal or the inclination angle. A grain boundary with an average normal direction or inclination angle which does not appear in the equilibrium shape will be faceted with a hill-and-valley shape as proposed by Herring.21) In principle, all flat planes of a faceted boundary can be atomically rough corresponding to the curved boundary segments of an equilibrium shape,22,23) but such a case is unlikely, because in analogy to the crystal surfaces,22,24) the equilibrium shapes are likely to consist of flat singular planes with rounded corners and edges. Therefore, as we proposed earlier,9,12,15) at least some segments of the faceted grain boundaries are expected to be singular with atomically flat structures.

Various grain boundary migration mechanisms have been earlier proposed. Based on the TEM observations of grain boundary migration, Gleiter,25–27) Bobcock and Balluffi,28,29) and Rae and Smith30) proposed that the grain boundaries migrated by the movement of grain boundary steps. They furthermore proposed that under high driving

<table>
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Fig. 1. The optical microstructure of as-received 316L stainless steel.
forces, the grain boundaries could migrate by nucleating two-dimensional steps analogously to the crystal growth process. Such step migration mechanism will operate in the singular grain boundaries. We have earlier proposed that when the grain boundaries migrate by such a step mechanism, AGG could occur because the mobility of the larger grains will be higher than the smaller grains.9,12,15) Even when a fraction of the grain boundaries are faceted with singular segments, they can control the overall growth behavior because their mobilities will be lower than those of the rough grain boundaries with abundant thermally activated steps. Even with dislocations impinging upon faceted grain boundaries in slightly deformed Cu,16) AGG was observed. The most definitive evidence for the boundary step growth mechanism for AGG is that in TiO₂-doped BaTiO₃ with faceted grain boundaries,14) only those grains with double twin boundaries grew abnormally during heat-treatment in air. When the grain boundaries became rough by heat-treating in hydrogen, there was no preferential growth in the direction of the twin boundaries.

When the heat-treatment temperature was increased to 1300°C, nearly normal grain growth was observed as shown in Fig. 5. In the 1300°C/5 min specimen shown in Fig. 5(a), there were some clusters of fine grains that were probably present in the as-received specimens. During the subsequent heat-treatment for over 10 min, these disappeared. There were some slightly large grains as indicated in the grain size distributions of Fig. 6, but plots of the size
distributions normalized to the average size did not change significantly during the heat-treatment, indicating that the grain growth was nearly normal. All of about 20 grain boundaries examined under TEM were smoothly curved as shown in Fig. 7. If a faceted grain boundary becomes defaced and hence curved at high temperatures, its atomic structure must be rough. Such a roughening transition has been extensively studied for crystal surfaces within the framework of solid-on-solid model. It has been predicted that when a flat singular surface undergoes roughening transition, it develops a curvature. Such a prediction has been experimentally verified for crystal surfaces. If a surface or a grain boundary segment is curved at all scales the surface or the grain boundary energy changes continuously with the normal direction. This is possible only if the interface is atomically rough with unlimited amount of thermally produced steps. Such an interface will migrate continuously with its velocity increasing linearly with the driving force. Then normal grain growth is expected as predicted by the analysis of Thompson et al. and the simulation of Srolovitz et al. The normal grain growth with atomically rough grain boundaries is consistent with the previous observations in Ni, Ag, a Ni-base superalloy, alumina, and BaTiO3.

In one experimental series, a specimen was first heat-treatment
treated at 1100°C for 60 min to obtain an AGG structure with a few grains as large as 400 μm as shown in Figs. 2(c) and 3. When this specimen was heat-treated again at 1300°C, the fine matrix grains coarsened but the largest grains did not grow abnormally as shown in Fig. 8. The grain size distribution thus appeared to approach that of normal growth, which is expected to occur at 1300°C. This result shows that even the abnormally large grains produced at 1100°C did not continue to grow abnormally when the growth mechanism was changed with the rough grain boundaries at 1300°C. The computer simulations of Grest et al.41 and Rollett et al.42 indeed showed that the grain size advantage alone could not induce AGG. In another step heat-treatment series, the specimens were first heat-treated at 1300°C for different periods to obtain grains of various average sizes by normal growth. When these were heat-treated again at 1100°C AGG occurred if the initial average grain size obtained by normal growth at 1300°C was relatively fine. These results confirmed that the growth behavior could be readily varied by temperature change.

At the intermediate heat-treatment temperature of 1200°C, the grain growth behavior was roughly between those at 1100°C and 1300°C. As shown in Fig. 9, the grain growth appeared to be almost normal, but some large grains appeared after heat-treating for 15, 30, and 60 min as more clearly shown in the grain size distributions of Fig. 10. The observed grain growth at 1200°C should be still classified as AGG, but the characteristics are less distinctive than the AGG at 1100°C shown in Fig. 2. The gradual change from AGG to normal growth with temperature increase is consistent with the step growth mechanism as pointed out earlier.7,9 If the grain boundary roughening transition resembles

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Fig. 8. The optical microstructure (a) and the grain size distributions (b) of the specimens heat-treated at 1100°C for 60 min and again at 1300°C for 15 min.

Fig. 9. The optical microstructures of the specimens annealed at 1200°C for (a) 5 min, (b) 15 min, (c) 30 min, and (d) 60 min.
the surface roughening transition of the infinite order Kosterlitz–Thouless universality class\(^{33-38}\) as proposed by Rottman\(^{43}\), the faceted grain boundaries and AGG in this commercial alloy as previously found in pure metals\(^{2,3}\) and a single phase Ni alloy\(^{12}\). It is also shown that AGG occurs in the single phase alloy without any precipitates pinning the grain boundaries as often suggested for the mechanism of AGG. The observed change of the growth mode from AGG to normal growth and the defaceting of grain boundaries with temperature increase provide qualitative evidences for the boundary step growth mechanism of AGG. By better understanding the normal and abnormal grain growth behavior, the grain sizes and their distributions can be more easily controlled in a commercial alloy like 316L, but in other alloys the defaceting transition temperatures of the grain boundaries may not fall in the temperature range between the solvus temperatures of the precipitates and the melting point.

### 4. Conclusions

The results of this study confirm the correlation between the faceted grain boundaries and AGG in this commercial alloy as previously found in pure metals\(^{2,3}\) and a single phase Ni alloy.\(^{12}\) It is also shown that AGG occurs in the single phase alloy without any precipitates pinning the grain boundaries as often suggested for the mechanism of AGG. The observed change of the growth mode from AGG to normal growth and the defaceting of grain boundaries with temperature increase provide qualitative evidences for the boundary step growth mechanism of AGG. By better understanding the normal and abnormal grain growth behavior, the grain sizes and their distributions can be more easily controlled in a commercial alloy like 316L, but in other alloys the defaceting transition temperatures of the grain boundaries may not fall in the temperature range between the solvus temperatures of the precipitates and the melting point.

### Acknowledgment

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### REFERENCES