Effect of Grain Size on Thermal and Mechanical Stability of Austenite in Metastable Austenitic Stainless Steel

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In order to clarify the grain size dependence of mechanical stability of austenite, deformation-induced martensitic transformation behavior was investigated on uniaxial tensile deformation in a metastable austenitic stainless steel (Fe–16%Cr–10%Ni) with the grain size controlled from 1 to 80 μm. In addition, crystallographic characteristics of deformation-induced martensite were analyzed by means of the EBSD (electron backscattering diffraction) method to discuss the variant selection rule. It was found that mechanical stability of austenite is independent of its grain size, although thermal stability of austenite is remarkably increased by grain refinement. Some special martensite variants tend to be selected in an austenite grain on the deformation-induced martensitic transformation (near single-variant transformation), and this results in the formation of a texture along tensile direction. This suggests that the most advantageous variants are selected in the deformation-induced martensitic transformation to release tensile strain and leads to the grain size independence of mechanical stability of austenite.

KEY WORDS: deformation-induced martensite; austenitic stainless steel; variant selection; grain refinement; stability of austenite.

1. Introduction

The mechanical properties of steels containing an austenite phase, such as austenitic stainless steels and TRIP-assisted low-alloy steels, strongly depend on the stability of austenite phase, because the austenite stability reflects the tendency of deformation-induced martensitic transformation and the work hardening behavior of the steels. In general, the austenite stability is discussed in terms of thermal and mechanical stabilities, and they are evaluated with Ms (Ms: temperature where athermal martensitic transformation starts) and Md30 temperatures (Md30: temperature where 50% of martensite is obtained by true tensile strain of 0.30), respectively. The austenite stability varies depending on the chemical composition, and thus both Ms and Md30 temperatures are expressed as a function of the total amount of alloying elements. On the other hand, grain size is also known to be one of the important factors influencing the thermal stability of austenite. The authors investigated the effect of grain size on athermal martensitic transformation behavior in a metastable austenitic stainless steel (Fe–16%Cr–10%Ni) with an austenite grain size ranging from 0.8 to 80 μm, and showed that the thermal stability of austenite is markedly increased by grain refinement, particularly when the austenite grain size is below 1 μm. We discussed this phenomenon on the basis of strain energetics of martensitic transformation and proposed a “spatial restriction effect”, that is the mechanism on the suppression of martensitic transformation by grain refinement: The elastic strain energy per unit volume generated by the formation of a martensite lath is significantly increased with the refinement of austenite grains, particularly when the grain size is less than 1 μm.

By contrast, the effect of grain size on the mechanical stability has not been sufficiently understood. Shirinivas et al. investigated the deformation-induced martensitic transformation behavior during cold rolling in type 304 and 316 stainless steels with various grain size. As a result, the volume fraction of deformation-induced martensite increases with decreasing grain size in 304 stainless steel, while the martensite formation was insensitive to the grain size in 316 stainless steel. However, the austenite grain size discussed in the Shirinivas’s report was not fine but larger than 50 μm. Considering that the grain size dependence becomes significant on thermal austenite stability when the grain size is less than 1 μm, it is necessary to take a careful look at the grain size dependence of deformation-induced martensitic transformation behavior in the range of ultrafine grain sizes. Additionally, the evaluation on ultrafine-grained austenite is

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also important from an industrial point of view, because the TRIP effect is usually realized by fine-grained retained austenite with a size below 1 μm in commercial multi-phase low-alloy steels. In order to improve the mechanical properties of commercial TRIP-aided steels, the stability of fine retained austenite must be controlled properly.

In this study, the deformation-induced martensitic transformation behavior was investigated on uniaxial tensile deformation for a metastable austenitic stainless steel with different grain sizes of 1 μm or less. Furthermore, crystallographic characteristics of deformation-induced martensite was analyzed by means of the electron back scattering diffraction (EBSD) technique, and then the grain size dependence on the mechanical stability of austenite was discussed in terms of the spatial restriction effect.

2. Experimental Procedures

2.1. Material and Methods

A metastable austenitic stainless steel (Fe–16%Cr–10%Ni) was used in this study. Chemical composition of the steel is shown in Table 1. A 1.5 kg ingot was produced in vacuum with an induction furnace. The ingot was hot-rolled to a 20 mm thickness at 1323 K, and then homogenized at 1523 K for 18 ks. After hot-rolling to a 15 mm thickness at 1323 K, the plate was solution-treated at 1273 K for 1.8 ks, followed by air-cooling. In order to obtain an ultrafine-grained austenite single structure, this material was subjected to the following thermo-mechanical treatment.4,7) At first, the solution-treated material was cold-rolled to a 90% thickness reduction to obtain a fully deformation-induced martensitic structure. The cold-rolled material was then reheated and held at 923 K to 1323 K for 0.6 ks to reverse martensite to austenite, followed by air-cooling. The grain size of austenite can be varied from 1 to 80 μm, which depends on the re-heating temperature.

A portion of the specimens with different austenite grain sizes were subjected to subzero treatment at 77 K for 1.8 ks in liquid nitrogen to investigate the thermal stability of austenite. Tensile tests were carried out at a strain rate of 5.6 × 10^-4 s^-1 for the plate specimens (gage: 3 w × 12.25 × 12.25 mm) at room temperature to investigate the mechanical stability of austenite. Microstructure was observed with an optical microscope (OM), a transmission electron microscope (TEM, JEOL JEM-2010) and a field-emission scanning electron microscope (FE-SEM, Hitachi S-4300). The grain size of austenite was measured by the quadrature method using optical micrographs.8) In the case of TEM images, the grain size of austenite was measured by the quadrature method. The data obtained by EBSD were analyzed by using software for automatic crystal orientation mapping (OIM analysis, TSL). Crystallographic orientation mapping was performed with a step size of 20, 100 or 150 nm, and the data obtained with a confidence index value more than 0.1 were used for the detailed crystallographic analysis.

2.2. Variant Determination

The variants of martensite were determined by a pole figure method based on the crystallographic information of austenite and martensite obtained by EBSD,10 and then the relation between slip planes and deformation-induced martensite variants was discussed. In general, it is known that the martensite formed in metastable austenitic stainless steel has a Kurdjumov-Sachs (K-S) orientation relationship (((111)γ//(011)α', [-101]γ// [-1-11]α')) with original austenite and there are 24 variants derived from cubic systems symmetry.10–12) The plane parallel and the direction parallel relationships between original austenite and the 24 martensite variants (V1–24) are summarized in Table 2. The 24 variants are classified into four variant groups according to the plane parallel relationship, V1–6, V7–12, V13–18, V19–24, and they are denoted as Packet 1, 2, 3 and 4. Figure 1(a) shows (001)α' pole figure of the 24 K-S variants corresponding to Table 2 superimposed on (001)γ standard stereographic projection. The variant of an experimentally observed martensite can be identified by comparing its (001)α' pole figure with Fig. 1(a). In the decision of the variant number of each

Table 1. Chemical composition of the steel used in this study (mass%).

| C  | Si  | Mn  | P   | S   | Cr | Ni | N  | Fe |
|----|-----|-----|-----|-----|----|----|----|----|
| 0.002 | 0.011 | 0.080 | 0.003 | 0.002 | 16.4 | 10.1 | 0.032 | Bal. |

Table 2. Martensite variants in the K-S orientation relationship.

| Variant No. | Parallel plane (Austenite)/(Martensite) | Parallel direction (Austenite)/(Martensite) |
|-------------|----------------------------------------|------------------------------------------|
| V1          | (1 1 1) // (0 1 1)                      | [-1 0 1] /[1 -1 -1]                      |
| V2          | (1 1 1) // (0 1 1)                      | [-1 0 1] /[1 -1 -1]                      |
| V3          | (1 1 1) // (0 1 1)                      | [0 1 -1] /[1 -1 -1]                      |
| V4          | (1 1 1) // (0 1 1)                      | [0 1 -1] /[1 -1 -1]                      |
| V5          | (1 1 1) // (0 1 1)                      | [1 -1 0] /[1 -1 -1]                      |
| V6          | (1 1 1) // (0 1 1)                      | [1 -1 0] /[1 -1 -1]                      |
| V7          | (1 -1 1) // (0 1 1)                     | [1 0 -1] /[1 -1 -1]                      |
| V8          | (1 -1 1) // (0 1 1)                     | [1 0 -1] /[1 -1 -1]                      |
| V9          | (1 -1 1) // (0 1 1)                     | [1 -1 0] /[1 -1 -1]                      |
| V10         | (1 -1 1) // (0 1 1)                     | [1 -1 0] /[1 -1 -1]                      |
| V11         | (1 -1 1) // (0 1 1)                     | [0 1 1] /[1 -1 -1]                       |
| V12         | (1 -1 1) // (0 1 1)                     | [0 1 1] /[1 -1 -1]                       |
| V13         | (-1 1 1) // (0 1 1)                     | [0 -1 1] /[1 -1 -1]                      |
| V14         | (-1 1 1) // (0 1 1)                     | [0 -1 1] /[1 -1 -1]                      |
| V15         | (-1 1 1) // (0 1 1)                     | [-1 0 1] /[1 -1 -1]                      |
| V16         | (-1 1 1) // (0 1 1)                     | [-1 0 1] /[1 -1 -1]                      |
| V17         | (-1 1 1) // (0 1 1)                     | [1 1 0] /[1 -1 -1]                       |
| V18         | (-1 1 1) // (0 1 1)                     | [1 1 0] /[1 -1 -1]                       |
| V19         | (1 1 -1) // (0 1 1)                     | [-1 1 0] /[1 -1 -1]                      |
| V20         | (1 1 -1) // (0 1 1)                     | [-1 1 0] /[1 -1 -1]                      |
| V21         | (1 1 -1) // (0 1 1)                     | [0 -1 1] /[1 -1 -1]                      |
| V22         | (1 1 -1) // (0 1 1)                     | [0 -1 1] /[1 -1 -1]                      |
| V23         | (1 1 -1) // (0 1 1)                     | [1 1 0] /[1 -1 -1]                       |
| V24         | (1 1 -1) // (0 1 1)                     | [1 0 1] /[1 -1 -1]                       |
martensite, the orientation of each original austenite grain is indexed under the condition that the rolling direction of the steel sheet or tensile direction of tensile specimen was located within the standard triangle, which is hatched in the (001)\(\gamma\) standard stereographic projection of Fig. 1(b). In this situation, the close packed planes of Packet 1, 2, 3 and 4, (111)\(\gamma\), (1-11)\(\gamma\), (-111)\(\gamma\), (11-1)\(\gamma\), shown by the colored circles, correspond to the primary, the cross, the critical and the conjugate slip planes in the original austenite, respectively. This variant determination makes it possible to discuss the variant selection rule in connection with the slip planes in prior austenite.

3. Results and Discussions

3.1. Microstructures of Fe–16%Cr–10%Ni Alloy

Figure 2 represents a TEM image of the Fe–16%Cr–10%Ni alloy with the reversion treatment of 923 K–0.6 ks after 90% cold rolling. It was confirmed that an ultrafine-grained austenitic structure with an average grain size of approximately 1 \(\mu\)m is formed. The electron diffraction pattern obtained from the selected area of 10 \(\mu\)m in diameter indicates that crystallographic orientations of the austenite grains are random without forming a special texture. Figure 3 shows the relation between reversion temperature and austenite grain size. With increasing reversion temperature, the austenite grain size gradually increases, and it reaches 85 \(\mu\)m when the reversion temperature is raised to 1 323 K. In this study, the reversion-treated materials at 923 K, 1 073 K and 1 273 K are referred to as ‘1 \(\mu\)m material’, ‘8 \(\mu\)m material’ and ‘60 \(\mu\)m material’, respectively.
3.2. Effect of Grain Size on Thermal Stability of Austenite

Figure 4 shows the changes in the volume fraction of athermally transformed martensite before and after subzero-treatment at 77 K for 1.8 ks as a function of the austenite grain size. Although the volume fraction of martensite is small at room temperature in all materials, subzero-treatment stimulates athermal martensitic transformation. The grain size dependence of thermal stability clearly appears: Approximately 30 vol% of martensite is formed in the subzero-treated materials with the grain size above 20 μm, whereas the martensitic transformation is remarkably suppressed by grain refinement below 20 μm. Particularly in the 1 μm material, the amount of martensite formed by the subzero-treatment is less than 3 vol%. This result demonstrates that the thermal stability of austenite is significantly increased by grain refinement as reported in our previous study.4) Figure 5 shows crystallographic orientation maps of bcc phase (martensite) in 60 μm material (a) and 1 μm material (b) with the subzero-treatment. The white lines in these figures denote original austenite grain boundaries. The inverse pole figure (c) obtained from the area of (a) shows the distribution of orientation along the rolling direction. In the 60 μm material, martensitic transformation partially occurred in each austenite grain and the martensite had various crystal orientations (multi-variant transformation), which resulted in the formation of a typical lath martensitic structure composed of blocks and packets and no texture (see (a) and (c)). On the other hand, it is found in the 1 μm material that martensitic transformation takes place in some austenite grains. It should be noted that the formed martensite has an equiaxed structure and identical crystal orientation as indicated by the arrows. Detailed crystallographic analysis reveals that each martensite tends to have no K-S orientation relationship with surrounding austenite grains. This means that an ultrafine austenite grain transforms to martensite with a single variant (single-variant transformation), leading to the disappearance of block and packet substructures.

In the previous study, the authors proposed the spatial restriction effect which explains the austenite grain size dependence on athermal martensitic transformation.4) It is generally accepted that the formation of martensite lath generates anisotropic strain in surrounding untransformed austenite. In order to disperse the transformation strain, several variants of martensite have to be formed simultaneously within an austenite grain (multi-variant transformation) as shown in Figs. 5(a) and 5(c). This should result in a minimization of total strain energy. However, if an austenite grain is refined to the size comparable to that of a martensite lath or block, several variants of martensite cannot be formed simultaneously within an austenite grain due to the shortage of space. In other words, it is impossible to minimize the strain energy generated by martensitic transformation in an ultrafine austenite grain.

3.3. Effect of Austenite Grain Size on Mechanical Stability of Austenite

Figure 6 shows the nominal stress-strain curves of the 1 μm, 8 μm, and 60 μm materials. The grain refinement results in an increase in yield strength as well as tensile strength. The 0.2% proof stresses of the specimens are expressed by the Hall-Petch relationship as shown in Fig. 7, which agrees well with our previous study.13) It is found that the work hardening rate is accelerated in all specimens, particularly when the strain is increased more than 10%. This indicates that martensitic transformation is induced during the tensile deformation and TRIP effect takes place. Figure 8 shows the changes in volume fraction of martensite during the uniform deformation stage. The volume fraction of deformation-induced martensite is continuously increased from the early stage of plastic deformation and then reaches 80 vol% at the maximum in all materials. The difference in
the transformation rate among three specimens is small. Based on this result, the volume fraction of deformation-induced martensite is plotted as a function of the austenite grain size in Fig. 9. By contrast with the case of athermal martensitic transformation (Fig. 4), the amount of deformation-induced martensite only depends on the deformation strain and is not affected by grain refinement, even by 1 μm. This result proves that the mechanical stability is never increased by grain refinement, unlike the thermal stability.

3.4. Texture Formation by Deformation-induced Martensitic Transformation

Figure 10 represents crystallographic orientation maps of 60 μm material (a) and 1 μm material (b) for bcc phase (martensite) formed by tensile deformation at 30%. The tensile direction is shown by ‘TD’. The orientation indicated by the color corresponds to that along the tensile direction. Inverse pole figures obtained from the image (a) and (b) are also shown as (c) and (d), respectively. The 60 μm material exhibits a lath martensitic structure, similarly to the as-quenched steel. However, the laths tend to have an identical orientation within a grain, and thus, coarse blocks and packets are formed, unlike the subzero-treated material shown in Fig. 5(a). That is, the martensite variant formed in an austenite grain is limited to some special ones. Furthermore, the inverse pole figure (c) reveals that the deformation-induced martensite has a significant transformation texture where the <101>α’ direction tends to be parallel to the tensile direction (The transformation texture was confirmed to exist even in several low magnified inverse pole figures). This means that deformation-induced martensitic transformation takes place not in the multi-variant transformation but in the near single-variant one. On the other hand, in the case of the 1 μm material, each austenite grain transforms to a single-variant martensite, like the subzero-treated material (Fig. 5(b)). It was also confirmed that the transformation texture (d) is similar to that of the 60 μm material (c).

Figure 11 shows the volume fractions of each variant and packet which are analyzed for 4 original austenite grains in the subzero-treated 60 μm material (a) and 18 ones in 30% tensile-deformed 60 μm material (b), respectively. Each color of red, green, blue and yellow correspond to the Packet 1, 2, 3 and 4 as shown in Fig. 1. In the case of athermal martensitic transformation (a), almost all kinds of 24 K-S variants appear and the fraction of each packet (Packet 1, 2, 3 and 4) is comparable. Therefore, the athermal martensite can be regarded as formed by multi-variant transformation. By contrast, in the case of deformation-induced martensitic transformation (b), an obvious variant selection occurs and some limited variants appear. In particular, the fraction of V5 and V6 accounts for as much as approximately 60% of the total

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fraction. As for the packet ratio, the Packet 1 occupied the maximum fraction at 69%, followed by Packet 2 and 4, whereas the martensite of Packet 3 hardly formed. Since the close-packed planes of Packet 1 and 3 correspond to the primary and critical slip planes of the original austenite, respectively, this variant selection should be attributed to slip deformation of the original austenite. Recently, Miyamoto et al. proposed a simple variant selection model for ausformed martensite; martensite variants whose close-packed plane is nearly parallel to the primary or secondary slip planes are selected, and among them the variants whose growth direction (close-packed direction) is nearly parallel to both the primary and secondary slip planes are more preferentially formed. According to their model, the preferential formation of V5 and V6 in Fig. 11(b) can be explained, because the close-packed plane is parallel to primary slip plane and the close-packed direction is also parallel to both primary and conjugate slip planes of the original austenite (see Table 2 and Fig. 1). The above results suggest that advantageous variants are selected in the deformation-induced martensitic transformation to release the unidirectional tensile strain.

3.5. Mechanism of Grain Size Independence of Mechanical Stability of Austenite during Uniaxial Tensile Deformation

Assuming that the martensite variant is preferentially formed to accommodate the unidirectional tensile strain, the reason why the mechanical stability of austenite is independent from grain size can be explained by considering the variant restriction effect, as schematically illustrated in Fig. 12. When a martensite lath forms within an austenite grain, an anisotropic transformation strain corresponding to the volume expansion is generated in the grain. In athermal martensitic transformation, the anisotropic transformation strain can be accommodated by a combinational formation of multi-variants (a). However, when the austenite grain size is quite fine, the martensitic transformation must take place via single-variant transformation due to the spatial restriction effect (b). This makes the accommodation of transformation strain by multi-variant transformation impossible and eventually athermal martensitic transformation hardly occurs. On the other hand, some special variants are selected so that the anisotropic transformation strain can be released by the tensile strain in deformation-induced martensitic transformation. In addition to the slip plane restriction, the accommodation mechanism leads to the variant selection and transformation texture of deformation-induced martensite (c), (d). In that case, the suppression of martensitic transformation by grain refinement becomes invalid because the multi-variant transformation is no longer necessary and single-variant transformation is favored for the martensitic transformation. This leads to the grain size independence of mechanical stability of austenite.

4. Conclusion

The effect of grain size on the mechanical stability of austenite was investigated in a metastable austenitic stainless steel with different grain sizes. The results obtained are summarized as follows:

(1) Martensitic transformation on cooling (athermal martensitic transformation) is markedly suppressed by grain refinement of austenite, particularly when the grain size is below 20 μm. That is, the thermal stability of austenite significantly depends on the grain size. On the other hand, there is no effect of grain size on the deformation-induced martensitic transformation behavior during tensile deformation, which suggests that the mechanical stability of austenite is independent of the grain size.

(2) In athermal martensitic transformation, 24 variants are equally formed to minimize the anisotropic transformation strain (multi-variant transformation) when the grain size is sufficiently large, while only one variant can be selected in an ultrafine grain due to the spatial restriction effect (single-variant transformation). The strain energy generated by martensitic transformation cannot be minimized via single-variant transformation, leading to suppression of martensitic transformation.

(3) The deformation-induced martensite formed in ten-
sile-deformed specimen has a texture where the \( <101>_{\alpha'} \) direction tends to be parallel to the tensile direction. This is because some specific variants are selected so that the anisotropic transformation strain can be released by the tensile strain. In that case, the suppression of martensitic transformation by grain refinement becomes invalid because the multi-variant transformation is no longer necessary and single-variant transformation is favored for the martensitic transformation. This leads to the grain size independence of mechanical stability of austenite.

REFERENCES

1) G. H. Eicherman, Jr. and F. C. Holl: Trans. ASM, 45 (1953), 77.
2) T. Angel: J. Iron Steel Inst., 177 (1954), 165.
3) K. Nohara, Y. Ono and N. Ohashi: Tetsu-to-Hagané, 63 (1977), 772.
4) S. Takaki1, K. Fukunaga, J. Syarif and T. Tsuchiya: Mater. Trans., 45 (2004), 2245.
5) E. Jimenez-Melero, N. H. van Dijk, L. Zhao, J. Sietsma, S. E. Offerman, J. P. Wright and S. van der Zwaag: Scr. Mater., 56 (2007), 421.
6) V. Shrinivas, S. K. Varma and I. E. Murr: Metall. Mater. Trans. A, 26 (1995), 661.
7) K. Tomimura, S. Takaki, S. Tanimoto and Y. Tokunaga: ISIJ Int., 31 (1991), 721.
8) JIS Hand Book Iron and Steel 1, Japan Standard Association, Tokyo, (2006), 638.
9) S. Takaki, Y. Tokunaga and K. Tomimura: Tetsu-to-Hagané, 73 (1987), 8539.
10) S. Morito, H. Tanaka, R. Konishi, T. Furuhara and T. Maki: Acta Mater., 51 (2003), 1789.
11) A. R. Marder and G. Krauss: Trans. ASM, 62 (1969), 957.
12) G. Miyamoto, A. Shibata, T. Maki and T. Furuhara: Acta Mater., 57 (2009), 1120.
13) K. Tomimura, H. Nagamori, S. Takaki and Y. Tokunaga: Jpn. Inst. Met., 55 (1991), 376.
14) G. Miyamoto, N. Iwata, N. Takayama and T. Furuhara: Acta Mater., 60 (2012), 1139.