Pseudo-texture memory in AISI 321 austenitic stainless steel

A A Tiamiyu*, J A Szpunar, A G Odeshi
Department of Mechanical Engineering, University of Saskatchewan, Saskatoon, SK, Canada
E-mail: ahmed.tiamiyu@usask.ca (A.A. Tiamiyu).

Abstract. In this study, a 45 µm-sized AISI 321 austenitic stainless steel was cryo-rolled at -196 °C to 50 % thickness reduction to completely transform the γ-austenite phase to αʹ-martensite. The cryo-rolled sample was subsequently annealed in the temperature range of 923 – 1073 K for 30-28800s (0.5 – 480 mins) to reverse the αʹ-martensite back to γ-austenite and develop ultrafine grain (UFG) structure at an optimum annealing condition. Average austenite grain sizes of 0.22 and 0.31 µm were obtained at an optimum annealing conditions of 923 K for less than 480 minutes and 1023 K for 10 minutes, respectively. High resolution EBSD and XRD texture results shows that ζ-fibre ({110}<uvw>) is the major texture component of austenite grains in the UFG structure and it is stronger in specimen annealed at 923 K than those of higher temperatures (973, 1023 and 1073 K) used in this study. The strong intensity of ζ-fibre in UFG is attributed to pseudo-texture memory in AISI 321 i.e. the ability of the steel to memorize the crystallographic orientation of the deformed austenite, rather than the conventional as-received austenite phase that is random.

1 Introduction

Texture memory effect (TME) is a phenomenon which occurs when the texture of material strengthens or remains unchanged after the phase transformation cycle from one phase to another and back to the initial phase [1,2]. TME has mainly been observed after the α' (bcc) → γ (fcc) → α' (bcc) transformation in BCC metals [2,3] and α (hcp) → β (bcc) → α (hcp) transformation in HCP metals [4,5] during a heating and cooling cycle. On the completion of transformation cycle in 0.1% C-1% Mn steel [2], a BCC metal, {100}<011>, {311}<011> and {332}<113> texture components reappeared with little decrease in intensity. Other alloys in which TME has been reported includes interstitial free (IF) steel [3], Ti–6Al–4V [4] and zirconium [5]. The orientation relationship (OR) between γ and α‘ in steels is the Kurdjumov–Sachs (K–S) relation, {111}γ\|(110)α‘ and <110>γ\ll<111>α‘, or the Nishiyama–Wasserman (N–W) relation, {111}γ\|(110)α‘ and <110>γ\ll<111>α‘. There are about 24 and 12 crystallographic variants in K–S and N–W relations, respectively. In the aforementioned metals that exhibit TME, crystallographic OR is obeyed between parent and product phases. Thus, implying that out of the dozens of the equivalent crystallographic orientations allowed under the OR, only specific ones are selected after the completion of the phase transformation cycle. This phenomenon is termed variant selection [2] and its description can be found elsewhere [6,7]. Although the mechanism behind TME is still not very clear, mechanisms related to transformation strain, role of precipitates, and development of special boundaries during heating have been proposed [1,2].
In the present study, UFG structure was developed in AISI 321 austenitic stainless steel (ASS) to improve its mechanical properties. AISI 321 is a titanium (Ti)-stabilized austenitic stainless steel that is widely used in chemical process plants [8] and fabrication of pressure vessels [9]. To the best of the authors’ knowledge, the development of UFG structure and the accompanying crystallographic texture in AISI 321 austenitic stainless steel has not been well addressed. The objective of this study is to investigate texture evolution during the development of UFG structure in AISI 321 steel. For the first time, the mechanism of texture memory in AISI 321 is addressed.

2 Material and methods

The nominal composition of the hot-rolled AISI 321 austenitic stainless steel plate used in this study is 17.61 Cr, 9.17 Ni, 1.56 Mn, 0.42 Mo, 0.40 Si, 0.36 Ti, 0.30 Cu, 0.15 Co and 0.044 C in weight percent. The average grain diameter of the as-received steel is 45±5µm. As-received sample was solution treated by soaking at 1373 K for 30 minutes and subsequently quenched in water at room temperature. The solution-treated sample was soaked in liquid nitrogen (77 K) for 25 minutes, followed by multi-pass unidirectional rolling. The steel specimen was intermittently re-soaked to avoid temperature rise during the multiple pass rolling. To obtain complete transformation and crushing of α'-martensite, cryo-rolling up to 50% thickness reduction (r = 50%) was conducted on 105 mm x 33 mm x 11 mm plates. The cryo-rolled samples were reversed to austenite (γ) by annealing in the temperature range of 923 – 1073 K for 30-28800s (0.5 – 480 mins). UFG structure developed in specimens annealed at 923 K and 1023 K. For brevity, only the results of specimens annealed at 923 K and 1023 K for 60-28800s (1-480 mins) and 30-1200s (0.5-20 mins), respectively, are presented in section 3. The specimens were electrolytically polished and subsequently characterized by X-ray diffraction (XRD) and electron back-scatter diffraction (EBSD) techniques. EBSD measurements were conducted using an SU 6600 Hitachi field emission scanning electron microscope coupled with an Oxford Instruments Nordlys Nano EBSD detector. The volume fraction of BCC α' was obtained using a Feritscope MP30E. The actual martensite content was determined by multiplying the Feritscope reading by a correction factor of 1.7 [10]. Hardness measurement was conducted using Mitutoyo Micro Vickers hardness testing machine (MVK-H1). All microstructural analysis and measurements in this study were conducted on the rolling plane (RP).

3 Results and discussion

Figure 1a shows a random texture in the γ-austenite phase of the as-received AISI 321 ASS. The as-received AISI 321 sample was slightly deformed (early stage of rolling) to avoid the start of martensitic transformation and the resulting deformed structure is hereafter referred to as the deformed austenite (γd). The EBSD IPF map of the γd grains (Fig. 1b) showed a significant texturing near the ζ-fibre ((110)<uvw>). The strong development of {110}<uvw> could be explained using grain B in Fig. 1b. This grain shows a possible orientation interplay between {110}||ND and {111}||ND i.e. the gradual rotation of {111}||ND grains during deformation to {110}||ND, which is the most stable end-orientation. Schmid factor (SF) map using {111}[1-10] as the active slip system was plotted to establish the correlation between plastic deformation mechanism (slip) and the orientation of γd (Fig. 1c). The SF varies with orientations in the following
order: \{100\}||ND (highest) > \{110\}||ND > \{111\}||ND. This indicates that the ease at which slip will occur on \{111\}[1\overline{1}0] slip system for the grains with \{111\}||ND orientation is lowest and as a matter of fact, these set of grains are near hard orientation. The \{111\}||ND oriented-grains are then “forced” to rotate during deformation to a more stable orientation (\{110\}||ND orientation with higher SF) [11]. Comparing the spread of orientation in the inverse pole figure (IPF) of undeformed

![Figure 1](image.png)

Fig 1. (a) EBSD-IPF map of the as-received sample, (b) IPF and (c) Schmid factor maps of deformed austenite (γd), (d) inverse pole figures of selected undeformed and deformed austenite grains. (e) EBSD-phase color, BCC and FCC IPF color maps of γd and α', (f) \{111\}γ and \{110\}α' pole figures of selected grains in (e), (g) revised sequence of strain-induced martensitic transformation (Color figure online).
\(\gamma\) (grain A) and deformed \(\gamma_d\) (grains B and C) in Fig. 1d shows that the \(\gamma_d\) has undergone plastic deformation by slip. The orientation of grain A converges to a point in the IPF triangle; suggesting a strain free grain. The grains B and C show the widest orientation spread, which is an indication of large plastic deformation by slip. The IPF triangle could also be a useful tool in showing the gradual rotation of \{111\}||ND to \{110\}||ND as in grain B (a spread of blue color towards green) and C (rotation nearly complete as confirmed by the predominant green color). Upon 15% thickness reduction during cryo-rolling, 80% of the \(\gamma\) phase is transformed to \(\alpha'\)-martensite (Fig. 1e). While the BCC IPF map in Fig. 1e shows a significant texturing of \(\alpha'\)-phase around \{100\}||ND, the remaining \(\gamma_d\) is textured around \{110\}||ND. To validate the existing OR between the \(\gamma_d\) and \(\alpha'\) phases, grains i and ii in Fig. 1e are selected and analyzed in Fig. 1f. The comparison of \{110\}\(\alpha'\) and \{111\}\(\gamma\) pole figures for grains i and ii, respectively, in Fig. 1f shows the coincidence of poles (circled). These confirms that K-S orientation relationship exists between the \(\gamma_d\) and \(\alpha'\) and not \(\gamma\) and \(\alpha'\). Hence, the \(\gamma \rightarrow \alpha'\) sequence is revised to \(\gamma \rightarrow \gamma_d \rightarrow \alpha'\) as also expressed in Fig. 1g.

The results of the X-ray measurement that confirms the complete martensitic transformation of the austenite phase in the as-received sample after cryo-rolling at 77 K are presented in Fig. 2a. Although, \(\gamma\)-austenite of the as-received steel was transformed completely to \(\alpha'\)-martensite at 20% thickness reduction, it was increased to 50% to generate finer martensite grains (insets in Fig. 2a) that accumulate enough stored energy. This promotes the development of ultra-fine strain-free austenite grains during subsequent annealing. The sigmoidal isothermal reversion curves and associated hardness profiles at two annealing temperatures are presented in Fig. 2b. Feritscope reading showed that austenitic reversion had barely occurred for samples annealed at 923 K and 1023 K for 60 s. As annealing time increases, specimens showed an increase in austenite fraction and a corresponding decrease in hardness values with annealing time. A notable observation in Fig. 2b is the sharp drop in hardness value of specimens annealed beyond 600s at 1023K. This is suspected to signal the onset of grain growth. Therefore, annealing at 1023 K for 600 s may be the optimum reversion condition to develop UFG structure via the martensitic shear reversion mechanism. Similarly, annealing at 923 K for 28800s may be the optimum reversion condition via the diffusional reversion mechanism since the volume fraction of the unreversed martensite is approximately 6%.

The results of macrotexture analyses show that the UFG structure is significantly textured with strong Brass \{110\}<112>, Goss \{110\}<100> and Copper \{112\}<111> components (Fig. 3a). Specimen annealed at 923 K for 28800 s (8 h) developed stronger texture than the specimen annealed at 1023 K for 600s (10 min). The EBSD maps of a specimen annealed for 180s at 923 K are presented in Fig. 3b-d, which indicate an early stage of reversion. Fairly equiaxed austenite grains with an average size of 0.16 \(\mu\)m randomly nucleated in the \(\alpha'\)-martensite lath boundaries (Fig. 3b). At the annealing time of 300s, the area fraction, morphology and texture of the phases are significantly changed (Fig. 3e-g). The shape of the \(\alpha'\)-martensite grains had changed from lath to near equiaxed after annealing for 300s. It is apparent in Fig. 3e-g that the morphology of the growing austenite grains is influenced by the morphology of their neighboring martensite grains.
Fig 2. (a) X-ray diffraction patterns of the as-received sample and cryo-rolled specimens. (b) Volume fraction of reversed austenite and corresponding hardness profile during reversion. Inset in (a) is the associated EBSD inverse pole figure (IPF-Z) maps of both the FCC-austenitic phase of the as-received and the BCC-martensitic phase of the cryo-rolled specimens.
To expatiate on this, four grains (1, 2, 3 & 4) in Fig. 3e-g are considered. The equiaxed martensite grain 1 has a corresponding equiaxed austenite grain 3 as its neighbour. Similarly, grains 2 and 4 are neighbours and both elongated. This shows that a growing austenite grain boundary may be hindered by the boundaries of their neighbour grains; forcing the austenite grain to take the morphology of the martensite. Elsewhere, the morphology of transformed α’ have been reported to influence the shape of the reversed austenite (γr) [12]. On the crystallographic orientation of grains, few variants are present in both BCC and FCC phases (Fig. 3h and i). All the variants developed during reversion in this study are presented in Table 1. The comparison of {111}γ and {110}α’ pole figures for all grains in Fig. 3j and k confirms the coincidence of poles. Grains 5 (6 {100} oriented-α’ and 6 {110} oriented-γ) are selected and further analyzed as presented in Fig. 3l. One of the poles of {111}γ is parallel to one of the poles of {110}α’. This confirms that K-S orientation relationship exists between the reversed γ-austenite and α’-martensite.

The EBSD maps of specimens annealed for 28800s (Fig. 4a-c) and 600s (Fig. 4d-f) at 923 K and 1023 K, respectively, shows the development of UFG structure with very low and acceptable fraction of α’-martensite. The average austenite grain sizes are 0.22 µm and 0.31 µm in 923 K and 1023 K-annealed specimens, respectively. Although, the orientation of γr grains are close to {110}<uvw> (ζ-fibre), texture is stronger in specimen annealed at 923 K (Fig. 4c) than specimen annealed at 1023 K (Fig. 4f). This agrees with the result of the macrotexture measurement in Fig. 3a. It is worth noting that most of the unreversed α’-martensite and TiC particles are located at the austenite triple junctions. These contribute to the stability of the UFG structure via Zener pinning of grain boundaries [13]. At higher annealing temperature and times, the larger fraction of the TiC particles dissolve and the volume fraction of α’-martensite was near-zero. This leads to abnormal grain growth by the breaking away of high angle boundaries and loss of texture [14]. To develop an UFG structure without abnormal grain growth, it may therefore be impractical to completely reverse the α’ to γ without retaining some negligible fraction of the α’.

While the texture of the γ-phase (parent) in the as-received AISI 321 ASS is random (Fig. 1a), it is apparent that the ζ-fibre texture of the γr (product) in the UFG structure memorizes the texture of the deformed austenite (γd) on the completion of γ (parent) → α’ → γr (product) transformation cycle. Since γd is intermediated between γ (parent) → α’ and conventional texture memory like those observed in BCC and HCP metals require that the texture of the product phase ‘remembers’ that of the initial parent phase, the phenomenon in AISI 321 steel is hereby referred to as Pseudo-texture memory. Hence, the transformation cycle is revised to γ → γd → α’ → γr as shown in Fig. 4g. The mechanism behind the pseudo-texture memory involves microstructural sites such as special grain boundaries. The variant that acted on the first transformation (γd → α’) in the special grain boundary is memorized and the same path becomes more accessible than the others for the α’ → γr reversion during annealing. This is schematically shown in Fig. 4h-l. At the early stage of cryogenic rolling of AISI 321 ASS, the austenite grains will first be deformed (Fig. 4h). As cryogenic rolling proceeds, two α’ grains for instance nucleate along the same γd boundary (Fig. 4i).
Fig 3. (a) Volume fraction of selected texture components. (b-g) EBSD maps of specimen annealed at 923 K (650 °C) for (b-d) 180s and (e-g) 300s: (b and e) phase color maps, (c, d, f, g) IPF color maps. {110} and {111} pole figures of specimens annealed at (h) 180s and (i) 300s. Superimposed {111}γ and {110}α′ pole figures of all grains in specimen annealed for (j) 180s and (k) 300s, (l) {111}γ and {110}α′ pole figures of selected grains in specimen annealed for 180s (Color figure online).
Fig 4. EBSD maps of specimen cryo-rolled and subsequently annealed at (a-c) 923 K (650 °C) for 28800s and (d-f) 1023 K (750 °C) for 600s. (g) sequence of transformation/reversion, (h-l) schematic of the pseudo-texture memory process adapted from Ref. [1] (Color figure online).
Table 1 The \{hkl\}<uvw> of all identified variants in the EBSD pole figures and ODFs.

| Variants | \(\alpha^\prime\)-martensite | \(\gamma\)-austenite | \(\alpha^\prime\)-martensite | \(\gamma\)-austenite | \(\alpha^\prime\)-martensite | \(\gamma\)-austenite |
|----------|-----------------------------|---------------------|-----------------------------|---------------------|-----------------------------|---------------------|
|          | 293 K / 180s                | 293 K / 300s        | 293 K / 28800s              |
| V1       | (001)<101>                  | (001)<201>          | (110)<3-11>                 | (001)<201>          | (001)<101>                  | (110)<101>          |
| V2       | (111)<12-31>                | (110)<110>         | (110)<12-11>                | (113)<2-51>         | (110)<1-10>                 | (001)<101>          |
| V3       | (110)<3331>                 | (113)<3-16>        | (111)<1-21>                 | (112)<0-21>         | (332)<1-22>                 | (111)<1-34>         |
| V4       | (112)<12-33>                | (212)<1-51>        | (113)<1-41>                 | (111)<1-23>         | -                           | (211)<0-32>         |
| V5       | (123)<2-33>                 | -                   | (213)<1-30>                 | (213)<1-32>         | -                           | -                   |
|          | 1023 K / 180s               | 1023 K / 600s      | 1023 K / 900s               |
| V1       | (023)<1-32>                 | (110)<331>         | (001)<011>                  | (011)<0-11>         | (011)<0-11>                 | (011)<0-11>         |
| V2       | (112)<111>                  | (113)<1-10>        | (111)<3-41>                 | (114)<1-10>         | (001)<102>                  | (001)<3-10>         |
| V3       | (012)<2-41>                 | (012)<110>         | (213)<0-41>                 | (213)<0-41>         | (111)<1-21>                 | (223)<3-41>         |
| V4       | (014)<1-71>                 | (015)<4-31>        | -                           | -                   | (210)<2-41>                 | (210)<1-34>         |
| V5       | -                           | -                   | -                           | -                   | (213)<0-21>                 | (213)<0-21>         |

These two \(\alpha^\prime\) grains obey the K-S orientation relationship with \(\gamma\) and exhibiting different variants of the 24 possible K-S conditions. As the percentage thickness reduction increases, both grains should grow into the neighbor \(\gamma\) grain and then merge to establish a ‘special grain boundary’ in the new \(\alpha^\prime\) grain structure (Fig. 4j). When the new \(\alpha^\prime\) grain structure is annealed, the special grain boundary will be highly favored for the nucleation of \(\gamma\) grains that possess the same orientation as the \(\gamma\) since it is the orientation that fulfills the K-S condition (Fig. 4k), as indicated in the experimental results presented in Fig. 3b-d, h-l. The available special grain boundaries in the specimen therefore act as crystallographic orientation memory sites during austenitic reversion. This process is more pronounced at lower annealing temperature (923 K) where TiC precipitates are more stable than at higher annealing temperature (1023 K). This explains why the intensity of \(\zeta\)-fibre is stronger at lower temperature (Figs. 3a and 4c) and weaker at higher annealing temperature (Figs. 3a and 4f). TME in Mn-IF steel has been attributed to the formation of TiC precipitates [1]. It can therefore be inferred that TiC precipitates played a significant role in the TME exhibited by AISI 321 steel. The schematic of pseudo-texture memory and stages of martensitic transformation and austenitic reversion during the development of UFG structure is shown in Fig. 5.
Fig. 5 Schematic of texture memory and stages of martensitic transformation and austenitic reversion. Shape and size of grains and particles are exaggerated to show the sequence of UFG development (Color figure online).

4 Conclusion
The strong ζ-fibre ({110}<uvw>) observed in the austenite phase of the UFG structure can be traced to AISI 321 ASS’s ability to memorize the orientation of the deformed austenite.

Acknowledgment
The authors wish to acknowledge the Natural Sciences and Engineering Research Council of Canada (NSERC) and Vanier Canada Graduate Scholarship for the financial support of this study. The support of ACUREN Group Inc. for the use of Fischer Feritscope MP30E is well appreciated.

References
[1] Hutchinson B and Kestens L A I 2008 Appl. Texture Anal., Rollett A D (New Jersey: John Wiley 81 Sons Inc) p 281.
[2] Tomida T, Wakita M, Yasuyama M, Sugaya S, Tomota Y and Vogel S C 2013 Acta Mater. 61 2828.
[3] Yoshinaga N, Inoue H, Kawasaki K, Kestens L and De Cooman B C 2007 Mater. Trans. 48 2036.
[4] Beladi H, Chao Q and Rohrer G S 2014 Acta Mater. 80 478.
[5] Romero J, Preuss M and Fonseca J Q 2009 Acta Mater. 57 5501.
[6] Nakada N, Tsuchiyama T, Takaki S and Hashizume S, 2007 ISIJ Int. 47 1527.
[7] Humbert M, Petit B, Bolle B and Gey N 2007 Mater. Sci. Eng. A 454–455 508.
[8] Wang J, Lin Y, Yan J, Zen D, Zhang Q, Huang R and Fan H 2012 Surf. Coatings Technol. 206 3399.

[9] Llewellyn D T and Hudd R C 1998 STEELS-Metallurgy and Applications (Oxford, UK: Reed Educational and Professional Publishing Ltd) p 337.

[10] Talonen J, Aspegren P and Hänninen H 2004 Mater. Sci. Technol. 20 1506.

[11] Nesterenko V F, Meyers M A, LaSalvia J C, Bondar M P, Chen Y J and Lukyanov Y L 1997 Mater. Sci. Eng. A 229 23.

[12] Tomimura K, Takaki S and Tokunaga Y 1991 ISIJ Int. 31 1431.

[13] Zhang X F, Terasaki H and Komizo Y 2001 Philos. Mag. Lett. 91 491.

[14] Rajasekhara S and Ferreira P J 2011 Acta Mater. 59 738.