

Amorphization under fracture surface in hydrogen-charged and low-temperature tensile-tested austenitic stainless steel

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Abstract

The microstructure just below the fracture surface in hydrogen-charged stable austenitic SUS 316L stainless steel, which was subjected to a low strain rate tensile test at -70 °C, was studied by a combination of the focused-ion-beam method and transmission electron microscopy. An amorphous region with a chemical composition almost identical to that of the polycrystalline region was found under the lath-like structure on the fracture surface, although no deterioration of tensile properties by hydrogen appeared. In the amorphous region, band-like regions with wavy contrasts were observed, which were often accompanied by cracks at the boundaries. The presence of the amorphous region with band-like regions implies that amorphization occurred due to high-density vacancies accompanied by agglomerations of excess vacancies in the hydrogen-charged SUS 316L stainless steel that was tensile-tested at low temperatures.

1. Introduction

Stable austenitic (γ) stainless steels such as SUS 316 (SUS: notation based on a Japanese industrial standard similar to AISI, *i.e.*, Type 316) exhibit high hydrogen embrittlement resistance and are expected to be used in hydrogen environments [1, 2]. However, in extreme environments with high hydrogen densities and/or at low temperatures, hydrogen embrittlement tendencies have been reported even for these stainless steels with excellent hydrogen embrittlement resistance, such as a reduction in the relative area maximized at around -70 °C and a decrease in relative elongation with high concentrations of surface hydrogen up to more than 100 ppm [3-6]. Such effects of hydrogen appear not only with respect to elongation and reduction of area but also on the fracture surface [4, 6]. Quasi-cleavage has been found to occur locally on the tensile fracture surface of stable γ stainless steel exposed to hydrogen, whereas dimple fracture surfaces were observed in general without hydrogen [4, 6]. Specifically, hydrogen embrittlement in stable γ stainless steel is characterized by a fracture surface with local quasi-cleavage. Quasi-cleavage is generally accompanied by a so-called lath structure due to the presence of deformation bands and cell structures that originate from a high density of dislocations [7]. Lath-like microstructures on brittle intragranular fracture surfaces were observed in fractographies performed for tensile fracture surfaces of hydrogen-embrittled γ stainless steel SUS 304 and SUS 316 [3, 4]. However, little research has been done on the crystallographic and morphological features immediately under these lath-like regions on the tensile fracture surface of γ stainless steels.

Direct observations of the deformation microstructures immediately under the fracture surfaces are indispensable in order to elucidate the origin of these characteristic fracture surfaces. Direct observations can be performed by transmission electron microscopy (TEM) combined with selective slicing of the exact areas immediately under the fracture surfaces by a focused ion beam (FIB). TEM observations combined with FIB were carried out on pipeline steel compact-tension-

tested in high-pressure hydrogen gas environments of 5-100 MPa. The results revealed the presence of deformation microstructures with slip band intersections immediately under the quasi-cleavage fracture surfaces [8, 9]. Similar observations of deformation microstructures immediately under the fatigue fracture surface of hydrogen-charged 304 and 316 stainless steels showed evident differences in microstructures related to the mechanisms by which hydrogen modifies deformation processes associated with different surface morphologies [10]. The deformation microstructures immediately under the fracture surface of γ stainless steel pre-charged uniformly with hydrogen and subjected to tensile testing, however, remain unclear.

In this paper, we report TEM observations combined with FIB concerning the deformation microstructures immediately under the quasi-cleavage region on the fracture surface of hydrogen-charged stable γ SUS 316L stainless steel subjected to a low strain rate tensile test at -70 °C. An amorphous region with a chemical composition almost identical to that of the polycrystalline region present further down was found under the lath-like structure on the fracture surface, although no deterioration of tensile properties by hydrogen appeared. In the amorphous region, band-like regions with wavy contrasts were observed, which were often accompanied by cracks at the boundaries. The presence of the amorphous region with band-like regions implies that amorphization occurred due to high-density vacancies accompanied by agglomerations of excess vacancies in the hydrogen-charged SUS 316L stainless steel that was tensile-tested at low temperatures.

2. Experimental

Tensile tests were conducted at a temperature of -70 °C, at which SUS 316L is most susceptible to hydrogen embrittlement, to maximize the quasi-cleavage region on the fracture surface. The chemical composition of the commercially available SUS 316L used in this study is

shown in Table 1. A cold-rolled and annealed thin sheet of SUS 316L with a thickness of 1.2 mm was further cold-rolled to a thickness of 0.45 mm, followed by heating at 1050 °C for 30 seconds and air cooling. The grain size after these treatments was adjusted to 30 μm. Test pieces 12.5 mm wide and 60 mm long were cut out from the cold-rolled thin sheet and charged with hydrogen in a high-pressure hydrogen (purity: 99.99999%) atmosphere of 95 MPa at 300 °C for 72 hours. A hydrogen charge of up to 90 mass ppm was confirmed by gas analysis of the specimens. Immediately after hydrogen charging, specimens were subjected to a low strain rate tensile test in air using a Shimadzu AG-100kNX tensile tester at -70 °C at a strain rate of $8.3 \times 10^{-4} \text{ s}^{-1}$, with a distance between gauge points of 50 mm. In order to observe the morphological features immediately under the fracture surface, TEM samples were carefully fabricated by FIB in the direction perpendicular to the fracture surface, after deposition of a thick tungsten protective layer on the surface. A Hitachi FB-2000A FIB system (acceleration voltage: 30 kV) equipped with a FIB microscope was used to fabricate the samples. FIB microscopy observations of the fracture surface were also done during FIB fabrication in order to investigate the presence of voids and cracks, in addition to the macroscopic deformation microstructures immediately under the fracture surface. Note that the FIB microscopy observations were carried out only in the initial stage of FIB processing at low magnifications to avoid damaging the TEM samples. A JEM-3000F (acceleration voltage: 300kV) TEM equipped with a double-tilt sample holder and energy dispersion X-ray spectroscopy (EDS) were used for morphological and crystallographical observations as well as for chemical analysis. Imaging plates were also used to record real-space images and electron diffraction patterns.

3. Results

The stress-elongation curves of SUS 316L with and without hydrogen pre-charging

obtained from the tensile tests at $-70\text{ }^{\circ}\text{C}$ at a strain rate of $8.3 \times 10^{-4}\text{ s}^{-1}$ are shown in Fig. 1(a). The tensile strengths were 741 MPa and 735 MPa, and the elongations were approximately 63 % and 55 %, respectively, for hydrogen-charged and non-charged samples in the -70°C tensile tests, indicating that no significant differences in tensile properties were observed with or without hydrogen charging.

Quasi-cleavage fracture surfaces with lath-like structures were found on the fracture surface of the tensile-tested samples. Figures 1(b) and 1(c) present FIB microscopy images obtained from the fracture surface of a tensile-tested sample and an enlarged view of the framed area in Fig. 1(b), respectively. The fracture surface was predominantly dimple ductile fracture (e.g., the area framed by D in Fig. 1(b)), but characteristic quasi-cleavage fracture surfaces with lath-like structures (e.g., the areas framed by QC in Fig. 1(b)) were locally present. The dimple ductile fracture surface did not display any significant differences with or without hydrogen charging, which can also be inferred from the fact that the tensile properties were not significantly different. No quasi-cleavage features other than lath-like ones were found during the observations. Because the area ratio of the quasi-cleavage fracture surfaces was less than 5%, FIB was used to fabricate TEM samples from a very small area of these characteristic surfaces. FIB fabrication of the samples for cross-sectional TEM observation of the microstructures immediately under the quasi-cleavage fracture surfaces was performed in the area indicated by the light blue frame in Fig. 1(c). Figure 1(d) shows a FIB microscopy image obtained during FIB fabrication taken from the direction perpendicular to that of the fabrication (i.e., the direction almost parallel to the fracture surface). Note that the dark band-like contrast region (indicated by arrow W) of up to $1\text{ }\mu\text{m}$ between the fracture surface and the vacuum region is the tungsten layer that was deposited to protect the fracture surface during FIB fabrication. Two types of regions with different contrasts were present immediately under the quasi-cleavage surface; one was a dark contrast region near the fracture

surface, and the other was a slightly bright contrast region about 4 μm from the surface. Some cracks (arrow A) and voids (arrow B) in the dark contrast region can also be clearly observed. Note that the vertical bright streaks are processing marks induced by FIB. It should be noted that the FIB microscopy observations were performed at low magnifications in order to avoid damaging the fracture surface by the high-density ion flux, which often occurs during high-magnification observations. Detailed observation of the lath-like features needs to be done with a more advanced FIB system using a scanning electron microscope in future work.

Ring-like electron diffraction patterns, so-called halo patterns, were revealed in the dark contrast region immediately under the fracture surface, suggesting that this region might be crystallographically distinct from the polycrystalline region that was present further down from the surface. Figure 2(a) presents a TEM micrograph obtained from the FIB sample immediately under the quasi-cleavage region shown in Fig. 1(c). There are two types of areas in the TEM image; a bright contrast area (Area I) further away from the fracture surface, and a dark contrast area (Area II) immediately under the fracture surface, similar to the FIB microscopy image observed during FIB fabrication. The presence of the deposited tungsten layer (indicated by arrow W) of up to 1 μm in thickness between the fracture surface and the vacuum region indicates that the fracture surface of the tensile test piece was protected after FIB fabrication. It should be noted that the large hole adjacent to Area I (indicated by arrow H) was an extrinsic defect induced during FIB fabrication, as a result of different milling rates between Areas I and II. More concretely, because the milling rate of Area II was much slower than that of Area I, the latter area was thinned quickly. In order to prevent further damage to Area I, Area II was not sufficiently thinned.

Figure 2(b) and 2(c) show electron diffraction patterns taken in Area I inside the circle denoted as A and in Area II inside the circle denoted as B, respectively. Each electron diffraction is typical of the pattern observed in each area. As shown in Fig. 2(b), the diffraction pattern

obtained from Area I exhibits polycrystalline diffraction spots resulting from the presence of grains with various crystallographic orientations. The corresponding bright-field (BF) images in Fig. 2(d) and 2(e) taken under different diffraction conditions, namely, with transmitted and diffracted electrons (BF1) and only transmitted electrons (BF2), indicate that Area I was characterized by the presence of a polycrystalline texture with grain sizes on the order of hundreds of nanometers stacked along the depth direction from the fracture surface. On the other hand, the electron diffraction pattern obtained from Area II in Fig. 2(c) shows obvious halo rings due to the presence of an amorphous region, the features of which will be described in detail later. In addition, some cracks (arrow C) similar to those in Fig. 1(d) can be observed along the dark stripe contrasts in Area II in Fig. 2(a). The differences in these diffraction patterns clearly indicate that the local crystal structure in Area II was evidently distinct from that in Area I. Chemical analysis was performed at A and B in Fig. 2(a) by energy dispersive spectroscopy (EDS) in order to investigate chemical inhomogeneities. Figure 2(f) shows EDS profiles measured at A and B in an energy range of 4.0 to 10.0 keV, in which there are K-edge peaks of the main components of this material, i.e., Fe, Cr, and Ni. Surprisingly, the EDS profiles obtained from A and B are almost identical, indicating that both regions had nearly the same composition, even though both were obviously distinct crystallographically.

The electron diffraction patterns obtained from Area II show the presence of an amorphous region characterized by halo rings. Figure 3(a) presents the whole image with emphasis on the contrast in the electron diffraction pattern obtained from a wide area of Area II in Fig. 2(a), with the intensity profile measured along the dotted line in this pattern shown in Fig. 3 (b). Halo rings with significant peak broadening can be clearly observed in the electron diffraction pattern. It should be noted that there are no diffraction spots indicating the presence of crystalline regions in the electron diffraction pattern. The diffraction pattern is similar to those observed in amorphous

materials with a stable face centered cubic (FCC) structure such as Zr-Cu alloys [11]. These experimental results suggest the presence of an amorphous region immediately under the lath-like structures on the quasi-cleavage fracture surface in the tested material. It should be emphasized that the amorphous region in Area II was intrinsic and not due to damage caused by FIB fabrication because this area clearly retained the protective W layer as mentioned above; in general, regions damaged by FIB can be easily removed during further FIB milling.

Careful TEM observations also revealed the presence of band-like regions of different material densities in the amorphous region of this material. The morphological features of these band-like regions are well displayed as contrast differences in bright-field images. Figure 3(c) shows a bright-field image taken in the area indicated by 'i' in Area II in Fig. 2(a). Band-like bright contrast regions with wavy patterns are clearly observed in the direction along the fracture surface in the matrix with dark contrast. Despite the distinct contrast between the band-like regions and the matrix, the diffraction patterns obtained from only these regions are almost identical, with halo rings similar to those in Fig. 3(a). The EDS results indicated that there was no clear difference in chemical composition between the band-like regions and the matrix. Figure 3(d) exhibits the bright-field (left panel) and dark-field (right panel) images taken using part of the halo ring indicated by the circle in Fig. 3(a) in another region. A notable feature here is that the band-like regions with bright contrast in the bright-field image also exhibit bright contrast even in the dark-field image taken using a part of the halo ring. This result revealed that the bright contrast in both the bright- and dark-field images was not due to the diffraction contrast originating from scattered electrons, but to the absorption contrast associated with the amount of electrons transmitted through the sample. Because the sample thickness was deemed to be relatively uniform on a micrometer order during FIB fabrication, the band-like regions with bright contrast presumably had a lower material density than the matrix region. In addition, the band-like regions consist of

stacks of the fine-wavy non-uniform contrast (indicated by an arrow) as shown in Fig. 3(c), indicating the presence of inhomogeneities in the density of materials in the band-like region. Note that because of severe dynamical diffraction effects in the thick amorphous region, the fine-wavy contrasts are obscured in Fig. 3(d). These results imply that the band-like regions may have originated from high-density vacancies accompanied by agglomerations of excess vacancies in the amorphous region, and that the fine wavy contrasts may have resulted from a density effect of the substructures in the amorphous phase. A careful comparison of Fig. 2(a) and Fig. 3(c) based on locations and contrast shape reveals that the boundaries between the band-like regions and the amorphous matrix presumably correspond to the dark-stripe contrasts in Fig. 2(a), as indicated by the dotted lines in the frame denoted as 'i'.

Cracks were found in the band-like regions of the amorphous matrix in this material. Figure 3(e) is a bright-field image obtained from the area in the square denoted as 'ii' in Fig. 2(a), which is different from the one in Fig. 3(c). The crack (indicated by a gray arrow) that appears along the boundary between the band-like region and the matrix can be clearly observed in the bright-field image. It should be mentioned that cracks can be identified because of the morphological feature that they appear between the band-like regions and the matrix. Similar cracks in the vicinity of the boundaries between the band-like regions and the amorphous matrix can also be observed in Fig. 2(a), which are accompanied by linear and zigzagging forms. Because stress concentration is likely to occur in a region with such an extreme density change, cracks could be preferentially induced at the boundaries. Note that a bubble-like pattern can be seen at the end of the cracks, although the origin of this particular shape remains unclear in the present study. The presence of many such cracks implies the occurrence of a high density of cracks immediately under the quasi-cleavage. The band-like regions in the amorphous matrix would, therefore, play an important role in quasi-cleavage fracture through the cracks induced during the tensile test.

4. Discussion

The foregoing results clearly demonstrate the presence of an amorphous region accompanied by a band-like region, which occurred at relatively equal intervals in an amorphous matrix immediately under the lath-like structure on the fracture surface of SUS 316L that was tensile-tested at $-70\text{ }^{\circ}\text{C}$ after hydrogen charging. It should be emphasized that similar lath-like structures were not found on the fracture surface of SUS 316L that was tensile-tested at low temperatures without hydrogen charging or tensile-tested at room temperature with hydrogen charging. Moreover, polycrystalline morphologies similar to those in Area I without the presence of an amorphous region were found in a FIB sample fabricated from immediately under dimples on the low-temperature tensile fracture surface of a sample without hydrogen charging (not shown here). These results indicate that hydrogen charged to the material relatively uniformly plays a key role in amorphization in the low-temperature tensile process.

In the previous studies, amorphization in alloys and intermetallic compounds was found as the result of the introduction of vacancies by high-energy electron or ion irradiation [12,13] and heavy plastic deformation by mechanical alloying [14,15]. The presence of amorphized regions immediately under the fracture surface was also reported in tensile-tested hydrogen-charged low-carbon ferritic steels, suggesting that vacancies in the cellular structures in a high-density dislocation region were responsible for such amorphization [16]. In fact, the promotion of vacancy generation by hydrogen has been reported in many metallic materials [17], including γ -stabilized stainless steels [6, 18-23]. For example, an increase in the amount of vacancies due to the introduction of strain in SUS304 was found by positron lifetime measurement, indicating the generation of excessive vacancies in the heavily plastically deformed region immediately under the fracture surface. In addition, a calculation of hydrogen-vacancy interaction in the temperature

range between 400 and 1100K in Fe-H binary alloys suggested an excessive increase in vacancy concentration with decreasing temperature [23]. Such excessive vacancies promoted by hydrogen are believed to contribute to amorphization and void formation related to lattice instabilities in the fracture process [24]. According to the hydrogen-enhanced strain-induced vacancy (HESIV) theory, hydrogen promotes vacancy generation and coalescence associated with plastic deformation and induces ductile fracture [6-7, 18]. In fact, it has been discussed that the amorphized region immediately under the fracture surface in tensile-tested hydrogen-charged ferritic steel can originate from the formation of high-density vacancies associated with hydrogen [16]. Therefore, high-density vacancies introduced as a result of the interaction between hydrogen and vacancies presumably induced amorphization in the highly plastically deformed regions of stainless steel charged with hydrogen in this study.

5. Conclusions

TEM studies were conducted on FIB samples fabricated from immediately under the quasi-cleavage on the fracture surface in hydrogen-charged stable γ SUS 316L stainless steel, which was uniformly hydrogen-charged to 90 wt. ppm and tensile-fractured after elongation of 63 % in a low strain-rate test at -70°C . Although no deterioration of tensile properties appeared, an amorphous region with a thickness of up to $4\ \mu\text{m}$ and with a chemical composition almost the same as that of the polycrystalline region was found immediately under the lath-like structure at the quasi-cleavage. Band-like regions with a wavy pattern were also clearly observed in the amorphous region, along with the presence of cracks at the boundaries between these band-like regions and the amorphous matrix. Although the hydrogen-charged SUS 316L stainless steel exhibited no hydrogen embrittlement in tensile properties at low temperatures, the HESIV mechanism presumably plays a crucial role in a hydrogen effect that appears as amorphization.

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Figure captions

Table 1 (Color Online)

Chemical composition of SUS 316L used in this study.

Fig. 1 (Color Online)

(a) Stress-elongation curves of SUS 316L with and without hydrogen pre-charging. (b) FIB microscopy image of the fracture surface in hydrogen-charged SUS 316L, (c) enlarged image of the area in the square in (b), and (d) FIB microscopy image taken during FIB fabrication from the framed area in (c). The ion incident direction in (d) is substantially perpendicular to the fabrication direction (namely, parallel to the fracture surface).

Fig. 2 (Color Online)

(a) TEM image obtained from immediately under the lath-like structure. Areas I and II exhibit a bright contrast and a dark contrast, respectively. (b) and (c) Selected area electron diffraction patterns obtained from the areas in circles A and B, respectively. Circles A and B are located in Areas I and II near the boundary, respectively. (d) and (e) bright-field images taken with transmitted and diffracted electrons (BF1) and only transmitted electrons (BF2) from the polycrystalline region under the amorphous area, respectively. (f) EDS profiles measured in circles A and B, respectively, in (a).

Fig. 3 (Color Online)

(a) Whole selected area electron diffraction pattern obtained from a wide area in Area II shown in Fig. 2 (a). Note that the contrast is slightly enhanced compared with that in Fig. 2(c). (b) Electron intensity profile measured along the dashed red line in (a). (c) Bright-field image taken in the area in the square denoted as 't' in Fig. 2(a). (d) The left and right panels are a bright-field image and

a dark-field image taken using a part of the halo (indicated by the circle in (a)). (e) Bright-field image taken in the area in the square denoted as 'ii' in Fig. 2(a).





