Tensile Properties of 22Cr-12Ni Austenitic Stainless Steel Thick Plates and Bars at Cryogenic Temperatures

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Abstract. The tensile properties of XM-19 austenitic stainless steels with thick plates (30 mm thickness), extra-thick plates (100 mm thickness), and rectangular bars (square of 14.3 mm) were examined at cryogenic temperatures. Influence of thermomechanical treatments on the steels was demonstrated. Even though their 0.2 % proof stresses were over 1200 MPa at 4.2 K, there were variations in strength depending on location. Namely, the 1/2t specimen (the position of the half in the thickness) of extra-thick plate showed lower 0.2% proof stress, elongation, and reduction of area at 4.2 K than the 1/4t (the position of the quarter in the thickness) and the thick plates. The effect of grain size on the 0.2% proof stress was clearly described by the Hall-Petch relationship for the materials.

1. Introduction
Austenitic steels are the structural materials of choice for high-field superconducting magnets. Especially at 4.2 K, nitrogen strengthened austenitic stainless type 316LN is commonly used for its high strength and thermodynamic stability. In addition, cryogenic steels called "Japanese Cryogenic Steels" have been developed for the superconducting magnets of the Fusion Experimental Reactor in 1980’s [1,2]. In recent years, there has been some interests in investigating and developing stronger and tougher weldable alloys for cryogenic structural components, particularly for magnet systems such as fusion demonstration reactor (DEMO) [3], where 0.2% proof stress of the structural material should be over 1200 MPa at 4.2 K.

A proper balance between 0.2% proof stress and fracture toughness at 4.2 K is derived from the grain coarsening. ASME standard XM-19 (22Cr-12Ni-5Mn-2Mo-0.2V-0.2Nb-0.3N, in mass%) stainless steel is strengthened by nitrogen solid-solution and grain refinement with (Nb,V) precipitates and shows the advantages of high strength and excellent weldability [4]. However, thickness dependence of strength
and toughness should be taken into account [4]. Limited data are available for the mechanical properties of extra-thick plates of XM-19. In this study, tensile properties of extra-thick plates (100 mm thickness), thick plates (30 mm thickness) and grooved rolled bars for XM-19 steels were evaluated at cryogenic temperatures.

2. Experimental procedure

2.1. Materials

The chemical compositions of the test steels are listed in table 1. The 180 kg steel ingots with φ210～φ250 × 525 mm were hot-forged into rectangular blocks in a square of 150 mm and hot-rolled. The 100-mm-extra-thick plates (ETPs) solution-treated at 1373 K, 1423 K or 1473 K for 21.6 ks and 30-mm-thick plates (TPs) solution-treated at 1373 K for 21.6 ks were provided. The test pieces were taken from the positions of the half (1/2t) and the quarter (1/4t) in the thickness and parallel to the transverse direction (TD). A 30-mm-thick plate in a square of 30 mm was cold-groove-rolled to a rectangular bar in a square of 14.3 mm. The rectangular bars (RBs) were annealed at 1073, 1173, 1273, 1373 or 1473 K for 3.6 ks followed by water quenched. The test pieces were taken from the bar parallel to the rolling direction (RD).

| Steels | Concentration (mass%) |
|--------|-----------------------|
|        | C  | Si  | Mn  | P   | S   | Ni  | Cr  | Mo  | V   | Nb  | N   |
| ETP    | 0.031 | 0.390 | 4.53 | 0.015 | 0.002 | 12.26 | 21.95 | 2.11 | 0.20 | 0.200 | 0.330 |
| TP     | 0.029 | 0.410 | 4.56 | 0.016 | 0.002 | 12.31 | 21.91 | 2.11 | 0.21 | 0.190 | 0.329 |
| RB     | 0.015 | 0.430 | 5.47 | 0.016 | 0.002 | 13.32 | 21.82 | 1.82 | 0.11 | 0.100 | 0.291 |

2.2. Tensile tests

Round bar tensile test specimens with 31.25 mm in gauge length and a nominal diameter of 6.25 mm (the length of reduced section is 37.5 mm) were cut from the materials. The tensile test was carried out at 293 K (in air), 77 K (immersed in liquid nitrogen) and 4.2 K (immersed in liquid helium) with the initial strain rate of 2.38×10^{-4} s^{-1}. Tensile properties were evaluated as the average of three tests data for each condition in the ETPs and TPs, and of two in the RBs. The geometries on the specimen surface after the tensile tests were evaluated by laser microscopy to characterize the local necking deformation. Fracture surfaces were analyzed by scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDS).

3. Results and discussion

3.1. Tensile properties

Figure 1 shows the tensile properties of the TP and ETPs such as 0.2% proof stress, ultimate tensile strength, elongation, and reduction of area. As the test temperature decreased from 293 K to 4.2 K, their strength increased but elongation decreased. The ETPs showed lower ductility at 4.2 K than the TP. Especially, the ETP 1/2t solution-treated at 1373 K showed a poor elongation (figure 1(b)). Their fracture surfaces of the ETPs specimens were flat consisted of facets. Nb segregation was detected on the fracture surface by EDS as shown in figure 2. In the ETPs, the diffusion of Nb during hot-rolling and solution treatment may be insufficient owing to the central segregation in the cast. Thus, Nb compound precipitates may cause the early fracture as described in below.

Figure 3 summarized the tensile properties of the RBs with the annealed at between 1073 K and 1473 K. The dependence of their tensile properties on the test temperatures exhibited almost the same trend to that of ETPs and TP. Both the ultimate tensile strength and 0.2% proof stress were higher as the decrease of annealing temperature, although the elongation and reduction of area were almost the same at 4.2 K for recrystallized materials. The RBs annealed at 1073 and 1173 K showed quite poor ductility.
Figure 1. Tensile properties of the plates with the test temperatures: (a) TP, (b) ETP annealed at 1373 K, (c) ETP annealed at 1423 K, and (d) ETP annealed at 1473 K.
3.2. Deformation behavior

Figure 4 shows the true stress-true strain curves and work hardening rates at 4.2 K for the EPTs and RB annealed at 1373 K. The EPT 1/2t sample exhibited the early fracture, while the EPT 1/4t and RB exhibited serrations, and higher strains and work hardening rates than the EPT 1/2t, respectively. Figure 5 shows their diameter profiles of gauge length after tensile fracture. The EPTs showed a localized deformation, in which the work hardening in the necked part was not enough. Therefore, the further deformation was localized in the first necked part or caused in the early failure. The RBs, on the other hand, showed a higher work hardening rate, which resulted in a multiple necking and higher elongation.
Figure 4. True stress - true strain curves and work hardening rates at 4.2 K for ETPs (1/2t and 1/4t) and RB annealed at 1373 K.

Figure 5. Diameter profiles of gauge length after tensile fracture for ETPs (1/2t and 1/4t) and RB annealed at 1373 K. Arrows indicate fracture points and necking parts, respectively. 0 position is fitted to an end of gauge length.

3.3. Microstructure dependence on 0.2% proof stress
Table 2 represents the average diameter of austenite grains for the materials. While the RBs annealed at 1273, 1373 and 1473 K were recrystallized austenite, the RBs annealed at 1073 and 1173 K were partially recrystallized austenite with σ phase precipitation which was determined by EDS chemical composition analysis.

The effect of grain size on the 0.2 % proof stress of the recrystallized RBs is clearly described by the Hall-Petch relationship as shown in figure 6. The Hall-Petch equation is given as follows:
\[ \sigma_y = \sigma_0 + k_y \cdot d^{1/2} \]

where \( \sigma_y \) is the yield stress, \( \sigma_0 \) is the corresponding stress for large single crystals, \( k_y \) is the Hall-Petch coefficient, and \( d \) is the average grain size. The Hall-Petch coefficient at 4.2 K is higher than that at 293 K, because the nitrogen strengthening of austenitic stainless steels is remarkable at lower temperature. Furthermore, lower content of N in the RB resulted in the lower 0.2 % proof stress than the ETPs and TP at 4.2 K. The 0.2 % proof stresses of the partially recrystallized RBs at 4.2 K and 77 K were lower than the expected values from the Hall-Petch equations indicated in figure 6, respectively. The dispersion of movable dislocations in the recovered austenitic grains may lower the 0.2 % proof stress. The Hall-Petch relationship was also applicable to the ETPs and TP, in which the trend was the same as the previous work [4]. Movable dislocations dispersed in the matrix during hot-rolling as well as rather inhomogeneous grain structure with Nb segregation for the ETP 1/2t may provide lower 0.2 % proof stress than for the ETP 1/4t and TP at 4.2 K. Therefore, grain refinement is very effective to increase the strength for the XM-19 at cryogenic temperature. From the viewpoint of stronger and tougher weldable alloys for cryogenic structural components, the evaluated materials in the present study exhibited the balance between 0.2% proof stress and fracture toughness as shown in figure 7 [2,5].

| Materials | Diameter (μm) |
|-----------|---------------|
|           | 1073 K | 1173 K | 1273 K | 1373 K | 1423 K | 1473 K |
| ETP, 1/2t | -      | -      | 26.0   | 41.2   | 50.3   |
| ETP, 1/4t | -      | -      | 24.3   | 45.8   | 59.0   |
| TP        | -      | -      | 20.0   | -      | -      |
| RB        | 1.5    | 3.3    | 8.9    | 42.5   | -      | 147.5  |

**Figure 6.** Temperature dependence on Hall-Petch relationship for austenitic stainless steels.
Figure 7. Relationship between fracture toughness and 0.2% proof stress for high nitrogen austenitic stainless steels. JAERI (Japan Atomic Energy Research Institute) -Box is a development target of strength and toughness for magnet systems at 4.2 K (0.2% proof stress, $\sigma_{0.2} > 1200$ MPa, fracture toughness, $K_{IC} > 200$ MPa√m). NIST (National Institute of Standards and Technology) trend exhibits a trade-off relationship between strength and toughness.

4. Conclusions
The tensile properties of XM-19 austenitic steels in the thick plates (30 mm thickness), extra-thick plates (100 mm thickness) and rectangular bars (square of 14.3 mm) were evaluated at cryogenic temperatures. Their 0.2% proof stresses at 4.2 K were over 1200 MPa and exhibited the temperature dependence by Hall-Petch relationship. The extra-thick plates showed a poor elongation at 4.2 K, especially for solution-treated at lower temperature. It revealed localized deformation and lower work hardening rate.

5. References
[1] Nakajima H, Yoshida K and Shimamoto S 1990 ISIJ International 30 567–578
[2] Umezawa O 2021 Materials Performance and Characterization 10(2) 3–15
[3] Tobita K, Asakura N, Hiwatari R, Someya Y, Utoh H, Katayama K, Nishimura N, Sakamoto Y, Honma Y, Kudo H, Miyoshi Y, Nakamura M, Tokunaga S, Aoki A and the Joint Special Design Team for Fusion DEMO 2017 Fusion Science and Technology 72(3) 537–545
[4] McRae D M, Balachandran S and Walsh R P 2017 Materials Science and Engineering 279 012001
[5] Ono Y, Umezawa O, Nishimura A, Kumagai S and Yuri T, unpublished data

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