Effect of Heat Treatment on Microstructure and Mechanical Properties of High-Strength Steel for in Hot Forging Products

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Abstract: High-strength steel is widely used in hot forging products for application to the oil and gas industry because it has good mechanical properties under severe environment. In order to apply to the extreme environment industry requiring high temperature and high pressure, heat treatments such as austenitizing, quenching and tempering are required. The microstructure of high-strength steel after heat treatment has various microstructures such as Granular Bainite (GB), Acicular Ferrite (AF), Bainitic Ferrite (BF), and Martensite (M) depending on the heat treatment conditions and cooling rate. Especially in large forged products, the difference in microstructure occurs due to the difference in the forging ratio depending on the location and the temperature gradient according to the thickness during post-heat treatment. Therefore, this study attempted to quantitatively analyze various phases of F70 high-strength steel according to the austenitizing temperature and hot forging ratio using the existing EBSD analysis method. In addition, the correlation between microstructure and mechanical properties was investigated through various phase analysis and fracture behavior of high-strength steel. We found that various microstructures of strength steel depend on the austenitizing temperature and hot forging ratio, and influence the mechanical properties and fracture behavior.

Keywords: high-strength steel; hot forging; heat treatment; microstructure analysis; phase analysis

1. Introduction

F70 High-strength steel is widely used in hot forging products for extreme environment applications such as oil pipeline systems, offshore plants, and gas industries owing to its good formability, economical, and high strength even in high-pressure environments achieved by post-heat treatments. Low-alloy steel generally increases the carbon content to achieve high strength, whereas the carbon content of F70 high-strength steel is limited to a maximum of 0.26 wt.% according to ASTM A694 [1]. Instead of solid solution strength by adding carbon, high-strength steel adds a small amount of grain refinement elements such as Nb, Ti, V in combination with C and N forming nitrides and/or carbides resulting in the grain refinement strengthening by the grain boundary pinning effect [2–5].

In addition, forged products made of high-strength steel require post-heat treatment such as austenitizing, quenching and tempering to ensure high strength and high toughness. In the case of high-strength steel having a ferrite-pearlite structure, it is transformed into austenite under post-heat treatment process and has various microstructures depending on
the cooling rate. Polygonal ferrite (PF) formed during cooling in the austenite-ferrite two-phase region has low strength but excellent toughness, while the structure formed at low-temperature transformation by rapid cooling depends on the heat treatment temperature and cooling rate such as Granular Bainite (GB), Acicular Ferrite (AF), Bainitic Ferrite (BF), and Martensite (M) as the faster cooling rate, the higher strength of the structure formed [6–8]. Such high-strength low-temperature transformation structure secures high strength and high toughness by a subsequent tempering process. Therefore, it is important to investigate the correlation between microstructure and mechanical properties of high-strength steel with complex microstructure.

However, due to the complex microstructure, it is difficult to visualize images through OM and SEM. Recently, a quantitative analysis method for various phases of low-carbon steel has been reported using EBSD analysis [8–10]. This study attempted to quantitatively analyze the phases of F70 high-strength steel by applying the existing EBSD analysis method. In addition, as the size of the forged product manufactured by hot forging becomes larger and the shape becomes more complex, the difference in microstructure is unlike with to the difference in the forging ratio depending on the location and the temperature gradient according to the thickness during post-heat treatment [11–14]. As a result, in large forged products such as flanges, inhomogeneity of the microstructure and different level of stress concentration depending on shape, location and thickness may occur deteriorating the quality of the product. In order to solve this problem in large forged products, it is very important to investigate the correlation between microstructure and mechanical properties through quantitative analysis on high-strength steels with various microstructures. Major factors affecting the microstructure and mechanical properties of high-strength steel include composition of alloying elements and heat treatment and hot forging conditions, and the effects of these factors on the microstructure and mechanical properties have been studied [15–20]. In addition, in the case of high-strength steel, various microstructure factors do not change independently, but rather counter interact each other in complex ways [8,21,22].

Recently, a quantitative analysis method for various phases of low carbon steel through the grain orientation spread (GOS) map and grain boundaries map of EBSD analysis has been reported [8,23,24]. However, studies on quantitative analysis of microstructures according to various hot forging ratios and post-heat treatment of high-strength steel are still limited. Therefore, this study attempted to quantitatively analyze various phases of high-tensile steel according to the austenitizing temperature and hot forging ratio using the existing EBSD analysis method. In addition, the microstructure and mechanical properties of F70 high-strength steel were evaluated according to the austenitizing temperature and hot forging ratio. The correlation between microstructure and mechanical properties was investigated through various phase analysis and fracture behavior of high-strength steel.

2. Materials and Methods

The chemical composition of the F70 high-strength steel used in this study is shown in Table 1. A plate of the F70 high-strength steel with 120mm thickness was hot forged using a Hydraulic 2000 ton press (F-HP-001, KALTEK) at 1200 °C with different reductions in thickness by 20%, 40%, and 60%, respectively.

Table 1. Chemical composition (mass %) of the present steel sheet.

| Element | C  | Si  | Mn  | P   | S   | Cr  | Ni  | As  | B  |
|---------|----|-----|-----|-----|-----|-----|-----|-----|----|
| Content | 0.1605 | 0.246 | 1.266 | 0.0147 | 0.0045 | 0.191 | 0.014 | 0.004 | 0.0002 |
| Ca      | 0.0002 | 0.027 | 0.082 | 0.0037 | 0.0019 | 0.002 | 0.0016 | 0.054 | 0.026 |
| Cu      | 0.0002 | 0.027 | 0.082 | 0.0037 | 0.0019 | 0.002 | 0.0016 | 0.054 | 0.026 |
| Mo      | 0.0002 | 0.027 | 0.082 | 0.0037 | 0.0019 | 0.002 | 0.0016 | 0.054 | 0.026 |
| N       | 0.0002 | 0.027 | 0.082 | 0.0037 | 0.0019 | 0.002 | 0.0016 | 0.054 | 0.026 |
| Nb      | 0.0002 | 0.027 | 0.082 | 0.0037 | 0.0019 | 0.002 | 0.0016 | 0.054 | 0.026 |
| Sn      | 0.0002 | 0.027 | 0.082 | 0.0037 | 0.0019 | 0.002 | 0.0016 | 0.054 | 0.026 |
| Ti      | 0.0002 | 0.027 | 0.082 | 0.0037 | 0.0019 | 0.002 | 0.0016 | 0.054 | 0.026 |
| V       | 0.0002 | 0.027 | 0.082 | 0.0037 | 0.0019 | 0.002 | 0.0016 | 0.054 | 0.026 |
| Al      | 0.0002 | 0.027 | 0.082 | 0.0037 | 0.0019 | 0.002 | 0.0016 | 0.054 | 0.026 |
| Fe      | 0.0002 | 0.027 | 0.082 | 0.0037 | 0.0019 | 0.002 | 0.0016 | 0.054 | 0.026 |

After hot forging, in order to investigate the microstructure and mechanical properties analysis according to the heat treatment conditions, the hot forged specimens were
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austenitized at the various temperature of 850 °C, 900 °C and 950 °C for 2 h followed by water quenching and then tempered at 600 °C for 3 h. The specimens for microstructure and mechanical analysis were obtained from the middle of a block of high-strength steel. Schematic diagram of hot forging and heat treatment process for F70 high-strength steel was shown in Figure 1. Specimen information according to the experimental conditions used in this study was shown in Table 2.

![Figure 1. Schematic diagram of heat treatment process for F70 high-strength steels in this study.](image)

Table 2. Specimen information according to the nine different test.

| Specimen  | Hot Forging Ratio (%) | Austenitizing Temperature (°C) | Austenitizing Time (h) | Tempering Temperature (°C) | Tempering Time (h) |
|-----------|-----------------------|-------------------------------|------------------------|-----------------------------|-------------------|
| HF20_850  | 20                    | 850                           | 2                      | 600                         | 3                 |
| HF20_900  |                       | 900                           |                        |                             |                   |
| HF20_950  |                       | 950                           |                        |                             |                   |
| HF40_850  | 40                    | 850                           | 2                      | 600                         | 3                 |
| HF40_900  |                       | 900                           |                        |                             |                   |
| HF40_950  |                       | 950                           |                        |                             |                   |
| HF60_850  | 60                    | 850                           | 2                      | 600                         | 3                 |
| HF60_900  |                       | 900                           |                        |                             |                   |
| HF60_950  |                       | 950                           |                        |                             |                   |

In order to evaluate the microstructure, the test specimens were physically polishing using a 360, 600, 1200 and 2000 grit SiC paper and micro-polished using a 3 and 1 µm diamond paste. Then, specimen surfaces were etched with 4% nital solution. The microstructure of specimen were observed using optical microscopy (OM) and FE-SEM HITACHI S-4800 (HITACHI, Tokyo, Japan).

Electron backscattering diffraction (EBSD) used a JEOL FE-SEM 7200F (JEOL, 7200F, Tyoko, Japan) with OIM software (EDAX, OIM v7.2.1 Orientation Imaging Microscopy, Mahwah, NJ, United States). The EBSD analysis were performed at a voltage of 20 kV and step size was 0.5 µm (×1000). For EBSD analysis, the specimen was micro-polished using 1/4 µm diamond paste and colloidal silica in the final polishing step. GOS map for different experimental conditions were obtained to measure for quantitative analysis of microstructure and average grain size. In addition, grain boundary maps were obtained to discuss the relationship between microstructure and mechanical properties.

Vickers hardness test was performed with a load of 4.903 N and a load time of 10 s. The hardness values was measured at least nine time and the average value was calculated excluding the maximum and minimum values.

Tensile test was performed one time for each condition using the round type tensile specimens (gauge length 25 mm, diameter 6 mm) in ASTM-E8 [25]. The tensile tests were conducted using a 100kN fully automatic tensile machine (MTS E45, Eden Prairie, MN,
USA) at a cross head speed of 1.5 mm/min. The yield strength was calculated at the 0.2% offset stress in the specimens showing continuous yielding behavior. The tensile strength was determined using the maximum load point, and the elongation was calculated by the displacement of the crosshead.

Charpy impact tests were conducted one time for each condition using an instrumented Charpy test machine (Zwick/Roell, PSW750+TZE, Ulm, Germany) according to ASTM E23 [26] at temperatures from 60 °C to −100 °C. Charpy impact tests were performed on standard sized Charpy specimen with 10 mm (width), 10 mm (height) and 55 mm (length) and 45° V-notch of 2 mm depth. Using this result, the Charpy impact absorbed energy curve was drawn and the ductile brittle transition temperature (DBTT) was calculated using Hyperbolic tangent function: (1)

\[ E = A + B \cdot \tanh[C(T - T_0)] \]  

where \( E \) is the absorbed energy (J), \( A, B, C \) are constants, \( T_0 \) is DBTT (°C), \( T \) is temperature (°C). In addition, in order to analyze the crack propagation energy of the 0 °C instrumented Charpy impact test and fracture surface analysis were performed. Instrumented data was analyzed for load–displacement through instrumented Charpy impact test. The Charpy impact energy was measured by considering the crack initiation energy (\( E_i \)) and the crack propagation energy (\( E_p \)). The crack initiation point was estimated by the compliance changing rate method [27,28]. The microstructure analysis of the crack propagation was performed by phase analysis through EBSD. Microstructure specimens was prepared by coating for 3 h at 85–90 °C using a Ni plating solution (84% distilled water + EN-8100NP(M) 10% + EN-8100NP(A) 6%). The fracture surface of the Charpy impact test specimen was observed with a FE-SEM HITACHI S-4800 (HITACHI, Tokyo, Japan).

3. Results
3.1. Microstructure Analysis of Hot Forged F70 High-Strength Steels

Figures 2 and 3 show the microstructure of F70 high-strength steels after different austenitizing temperatures of hot forging F70 high-strength steels. In general, the change of microstructure due to tempering affected carbide precipitation or softening matrix, but it was reported not to directly affect the type and fraction of microstructure [11,29–31]. Therefore, in this study, the focus was on phase analysis of microstructure changes according to the austenitizing temperature greatly influencing the type and fraction of the microstructure. The microstructure according to the austenitizing temperature after 20% hot forging are shown in Figures 2a–c and 3a–c. At an austenitizing temperature of 850 °C, a complex microstructure was observed mainly due to the mixture of Polygonal Ferrite (PF), Granular Bainite (GB), and Acicular ferrite (AF). PF is a structure formed from cooling in austenite-ferrite two-phase region. It is formed at the highest temperature and low cooling rate, and has fine grains and a polygonal shape [8]. GB has a secondary phase inside the grain in the form of island, and it is formed at the slowest cooling rate among the bainite structure (relatively coarse grain), while AF is generated in the austenite deformation band at a cooling rate faster than GB resulting in fine grain and irregular grain orientation [8]. When the austenitizing temperature increased to 900 °C (Figures 2b and 3b), a mixture of PF, GB, AF and Bainitic Ferrite (BF) were observed, and at 950 °C (Figures 2c and 3c), PF, GB and AF seemed to be disappeared showing BF and Martensite (M). The BF was generated at a faster cooling rate than the AF structure leading to coarse grains with lath-shaped secondary phases inside [8]. The M was formed under the fastest cooling rate with large grains and lath structured packet inside the grain [8]. At an austenitizing temperature of 850–900 °C, the PF was formed indicating the austenite transformation temperature was below the \( A_{γ₃} \) temperature. On the other hand, at the higher austenitizing temperature of 950 °C, the PF disappeared indicating all the structure became fully austenite at 950 °C. In general, it was known that the \( A_{γ₃} \) temperature of low-carbon steel ranges from 850 to 880 °C depending on the amount of carbon and the composition of alloying elements [32,33]. Unlike studied in the literature, the material used in this study was not
fully austenite at 900 °C. The microstructures formed at a high cooling rate were observed as the austenitizing temperature increased. As a result, it is presumed that the higher the austenitizing temperature, the faster the cooling rate is due to the influence on the cooling rate. Figures 2d–f and 3d–f show microstructure after heat treatment of 40% hot forging. Similar to the 20% hot forging, a mixture of PF, GB, and AF was observed at an austenitizing temperature of 850 °C (Figures 2d and 3d), and PF, GB, AF, BF were observed at an austenitizing temperature of 900 °C (Figures 2e and 3e). At an austenitizing temperature of 950 °C, a complex microstructure of M and BF was observed (Figures 2f and 3f). After 60% hot forging, PF, GB, and AF were observed at an austenitizing temperature of 850 °C (Figures 2g and 3g). At an austenitizing temperature of 900 °C (Figures 2h and 3h), unlike hot forging 20% and 40%, BF and M were observed. At an austenitizing temperature of 950 °C, the most M was observed along with BF (Figures 2i and 3i). It was found that the austenite single-phase region was formed at a temperature lower for 60% hot forging than 20% and 40% hot forging. This is because the A_r3 temperature decreases when the hot forging ratio is high by plastic deformation. Through microstructure analysis, in the case of hot forging 20% and 40%, there was a change in microstructure due to an increase in austenitizing temperature, but the difference in hot forging ratio was not significant. However, in 60% of hot forging, it was confirmed that the difference in microstructure according to the hot forging ratio and austenitizing temperature occurred compared to 20% and 40% of hot forging. EBSD analysis was carried out for clear classification and quantitative phase analysis of various microstructures of F70 high-strength steel.

Figure 2. Optical microstructure of F70 high-strength steels after heat treatment: (a) HF20_850, (b) HF20_900, (c) HF20_950, (d) HF40_850, (e) HF40_900, (f) HF40_950, (g) HF60_850, (h) HF60_900 and (i) HF60_950.
Figure 3. The microstructure of F70 high-strength steels after heat treatment using the scanning electron microscopy (SEM): (a) HF20_850, (b) HF20_900, (c) HF20_950, (d) HF40_850, (e) HF40_900, (f) HF40_950, (g) HF60_850, (h) HF60_900 and (i) HF60_950.

Figure 4 shows an EBSD analysis for quantitative measurement of various phases and grain size according to the austenitizing temperature after hot forging. Quantitative analysis of different phases was performed using the Grain Orientation Spread (GOS) map and Grain boundaries map. The GOS analysis was performed by calculating the average misorientation between each point in one grain using a grain orientation spread map. The GOS analysis uses a misorientation setting value of 5° or less and grain boundary map with 15° angle was matched to the characteristics of each microstructure such as the shape of the structure, the grain orientation, the low angle grain boundaries within the grain, and the dislocation density as the quantitative phase fraction was measured [8]. The PF was classified into a structure with a misorientation value of about 2–3° or less, a low dislocation density inside the grain, and did not have any secondary phase [8]. The GB and BF have higher misorientation values (approximately 3–5° or less) than PF and have coarse grains. Among them, it was a GF structure if dislocation density inside the grain was relatively low and the secondary phase inside the grain was in the form of an island. On the other hand, if the dislocation density was relatively high while the second phase inside the grain was in the lath structure, it was classified as a BF [8,9]. The M structure had lath structure similar to the BF structure, but it is classified as a structure composed of various grain orientations with a HAGBs of lath structure inside the grain boundaries [34]. In the case of AF, after identifying PF, GB, BF, and M, the remaining was classified as AF in which the areas had fine grains with relatively high dislocation density compared to PF [8]. Table 3 numerically summarizes the fraction and grain size of each microstructure according to the austenitizing temperature after hot forging under all conditions.
Figure 4. Phase analysis of F70 high-strength steels after heat treatment using the EBSD analysis: (a) HF20_850, (b) HF20_900, (c) HF20_950, (d) HF40_850, (e) HF40_900, (f) HF40_950, (g) HF60_850, (h) HF60_900 and (i) HF60_950.

Table 3. Microstructure area fraction (%) and average grain size of F70 high-strength steel.

| Scheme     | Microstructure Area Fraction (%) | Average Grain Size (µm) |
|------------|----------------------------------|-------------------------|
|            | Polygonal Ferrite | Granular Bainite | Acicular Ferrite | Bainitic Ferrite | Martensite |              |
| HF20_850   | 24.3       | 61.4           | 14.3            | -               | -          | 8.5 ± 5.2    |
| HF20_900   | 16.3       | 52.2           | 20.6            | 10.9            | -          | 10.8 ± 8.3   |
| HF20_950   | -          | -              | -               | 47.8            | 52.2       | 6.8 ± 5.0    |
| HF40_850   | 26.5       | 66.6           | 6.9             | -               | -          | 10.7 ± 7.7   |
| HF40_900   | 17.1       | 50.3           | 10.0            | 22.6            | -          | 11.2 ± 7.3   |
| HF40_950   | -          | 2.3            | -               | 45.8            | 51.8       | 9.5 ± 6.6    |
| HF60_850   | 39.3       | 49.9           | 10.8            | -               | -          | 10.7 ± 6.6   |
| HF60_900   | 2.2        | 32.4           | 16.8            | 43.3            | 5.3        | 11.5 ± 9.0   |
| HF60_950   | -          | -              | -               | 30.1            | 69.9       | 5.2 ± 3.7    |

The fraction of microstructure according to the austenitizing temperature after 20% hot forging ratio was measured at an austenitizing temperature of 850 °C (Figure 4a) as 61.4% of the GB and 24.3% and 14.3% of the PF and AF, respectively. At an austenitizing temperature of 900 °C (Figure 4b), the fractions of GB and PF decreased to be 52.2% and 16.3%, respectively. The fraction of AF increased to 20.6%, and BF was measured to be 10.9%. At an austenitizing temperature of 950 °C (Figure 4c), the PF, GB and AF disappeared, and the fraction of M was 52.2% and that of BF was 47.8%. It was considered that all the structures became fully austenite above 950 °C. In addition, the fraction of M and BF increased as the heat treatment temperature increased. The fraction of microstructure
after 40% hot forging ratio was measured as GB 66.6%, PF 26.5%, and AF 6.9% at an austenitizing temperature 850 °C, similar to that after 20% hot forging ratio (Figure 4d). At an austenitizing temperature of 900 °C (Figure 4e), the fraction of GB and PF decreased while the fraction of AF and BF increased (50.3%, 17.1%, 10.0% and 22.6%, respectively). At an austenitizing temperature of 950 °C (Figure 4f), microstructures showed M (51.8%) and BF (45.8%).

The fraction of microstructure after 60% hot forging showed that PF increased to be 39.3 (as compared to 20%, 40% hot forging) while GB and AF was measured to be 49.9% and 10.8%, respectively, at an austenitizing temperature of 850 °C (Figure 4g). The increase in the fraction of PF was resulted from an increase in the amount of deformation in the austenite region as the hot forging ratio increased promoting transformation of austenite-ferrite two phase region [35,36].

At an austenitizing temperature of 900 °C (Figure 4h), the fraction of PF significantly decreased to 2.2% while that of BF increased significantly to 43.3% as compared 20% and 40% of hot forging. In addition, the fraction of GB and AF 32.4% and 16.8%, respectively, while M was measured 5.3%, which did not appear in hot forging 20% and 40%. In the case of 60% hot forging, the austenite transformation temperature (A_r3) decreases due to a high plastic deformation leading to decreases the temperature of austenite single phase region. At an austenitizing temperature of 950 °C (Figure 4i), the fraction of M was the highest at 69.9% as compared to 20% and 40% for hot forging, while BF was observed at 30.1%. Usually, deformation and defects are increased with increasing plastic deformation, resulting in decreasing nucleation temperature during the phase transformation. Consequently, it is attributed to the fraction of BF and M increased with hot forging ratio increase at an austenitizing temperature 900 °C and 950 °C [36,37].

The grain size of F70 high-strength steel was measured using a grain size map through EBSD analysis. The average grain size was to be 8.5–10.7 µm due to relatively high fractions of fine PF at an austenitizing temperature of 850 °C. At an austenitizing temperature of 900 °C, the average grain size was to be 10.8–11.5 µm due to the high fractions of relatively coarse grained GB and BF. At an austenitizing temperature of 950 °C, the finest grain size of 5.2–9.5 µm was observed by the packet of HAGBs inside. However, the grain size showed a high standard deviation due to the large difference in size of mixed phases.

Figure 5 shows EBSD grain boundary map of the microstructure of F70 high-strength steels after heat treatment. In the grain boundary map, Low angle grain boundaries (LAGBs) with 2° < θ < 5° and high angle grain boundaries (HAGBs) with θ > 15° are shown as green lines and black lines, respectively. In the PF, all grain boundaries consisted of a HAGBs, and no other grain boundary was inside. In the AF, a LAGBs was inside with a HAGBs similar to that of PF. In GB and BF, grains with a coarse HAGBs and LAGBs were observed in the form of island and lath structure, respectively. In the M, both grain boundaries and packet had HAGBs, and a LAGBs were inside the packet. After all hot forging, the fraction of LAGBs was the highest and the fraction of HAGBs was the lowest at an austenitizing temperature of 900 °C and the fraction of HAGBs was the highest and the lowest fraction of LAGBs at an austenitizing temperature of 950 °C. At an austenitizing temperature of 900 °C, grain were coarse and the fraction of GB and BF with LAGBs was high while the fraction of M with HAGBs packets was high at an austenitizing temperature of 950 °C. In addition, the fraction of GB was higher at an austenitizing temperature of 850 °C than an austenitizing temperature 900 °C, while the fraction of PF with HAGBs was higher resulting in the higher fraction of HAGBs at an austenitizing temperature of 850 °C.

3.2. Vickers Hardness and Tensile Property of Hot Forged F70 High-Strength Steels

Figure 6 shows Vickers hardness test results of the F70 high-strength steels according to the austenitizing temperature after hot forging. In case of 20% a hot forging, the hardness value was measured to be 233 Hv at an austenitizing temperature of 850 °C. As an austenitizing temperature increased to 900 °C and 950 °C, the hardness value increased to 243 Hv and 291 Hv, respectively. Here, as the austenitizing temperature increased, the fractions of PF and GB with low dislocation density and low strength decreased while the fractions of the BF and M with relatively high dislocation density and high-strength
increased [11]. In particular, the hardness value increased significantly at an austenitizing temperature 950 °C due to formation of the hard M. In case of 40% and 60% hot forging, the hardness value increased as the austenitizing temperature increased, similar to the results of the 20% hot forging.

Figure 5. Grain Boundary (GB) map of the F70 high-strength steels after heat treatment: (a) HF20_850, (b) HF20_900, (c) HF20_950, (d) HF40_850, (e) HF40_900, (f) HF40_950, (g) HF60_850, (h) HF60_900 and (i) HF60_950.

Figure 6. Vickers hardness values of F70 high-strength steels after heat treatment.
The Vickers hardness value of 20% and 40% hot forging at an austenitizing temperature of 850 °C was 233 Hv and 231 Hv, respectively, while 60% hot forging showed a decrease in hardness value of 222 Hv. Such low hardness for 60% hot forging may result from an increase fraction of the most soft PF structure where the more ferrite in the austenite-ferrite two-phase region remained as the more hot forging ratio applied. In other words, as the hot forging ratio increases, a lot of ferrite remains in the austenite-ferrite two-phase region, so the fraction of PF with low strength increases. In addition, the hardness value increased as the hot forging ratio increased at an austenitizing temperature of 900 °C to 950 °C. Such increased hardness may be due to the increased fraction of hard BF and M from higher the nucleation rate and the lower the Ar3 temperature.

Figure 7 shows the results of tensile properties for F70 high-strength steels according to the austenitizing temperature and hot forging ratio. In Figure 7a, after 20% hot forging at an austenitizing temperature of 850 °C, the yield strength and the tensile strength (YS/TS) were 575 MPa and 690 MPa, respectively. As an austenitizing temperature increased to 900 °C and 950 °C, the YS increased to 623 MPa and 685 MPa, as well as the TS increased to 739 MPa and 776 MPa, respectively. Similar to the Vickers hardness test result, as the austenitizing temperature increases, the increase in YS and TS is due to an increase in the fraction of the M and BF with relatively high strength and high dislocation density. However, the elongation decreased from 25.0% to 23.9% and 18.8% as the austenitizing temperature increased from 850 °C to 950 °C. The elongation showed the best properties at an austenitizing temperature of 850 °C with the highest fraction of PF structure with good ductility. In addition, as the austenitizing temperature increased, the fraction of PF structure decreased and the elongation decreased. In the case of 40% and 60% hot forging ratio in Figure 7b,c, as the austenitizing temperature increased, the YS and TS increased but the elongation decreased. As the austenitizing temperature increases, the PF and GB fractions decrease while the BF and M fractions increase. As shown in the microstructure analysis results (Figure 4), this is due to a decrease in fractions of PF and GB and an increase in fractions of BF and M as the increase in austenitizing temperature.

As the hot forging ratio increased, both YS and TS decreased while the elongation increased at an austenitizing temperature of 850 °C, whereas the YS and TS increased at an austenitizing temperature of 900 °C and 950 °C. Here, as increasing the hot forging ratio, the fraction of PF with low strength increased at an austenitizing temperature of 850 °C, while the high-strength BF and M significantly increased at an austenitizing temperature 900 °C and 950 °C. Table 4 summarizes the mechanical properties such as Vickers hardness and tensile properties tested in this study.

![Figure 7](image-url)

**Figure 7.** Tensile properties of F70 high-strength steels after heat treatment: (a) 20% hot forging, (b) 40% hot forging and (c) 60% hot forging.
Table 4. Mechanical properties of the specimens used in this study.

| Specimen | Average Vickers Hardness (Hv) | Yield Strength (MPa) | Tensile Strength (MPa) | Elongation (%) |
|----------|-------------------------------|----------------------|-----------------------|----------------|
| HF20_850 | 233.4 ± 5.1                   | 575                  | 690                   | 25.0           |
| HF20_900 | 242.2 ± 2.8                   | 623                  | 739                   | 23.9           |
| HF20_950 | 289.4 ± 1.0                   | 685                  | 776                   | 18.9           |
| HF40_850 | 231.6 ± 3.4                   | 573                  | 701                   | 25.6           |
| HF40_900 | 242.6 ± 7.4                   | 624                  | 736                   | 23.1           |
| HF40_950 | 291.2 ± 5.4                   | 678                  | 772                   | 21.4           |
| HF60_850 | 222.8 ± 5.2                   | 518                  | 663                   | 26.3           |
| HF60_900 | 265.4 ± 3.6                   | 640                  | 754                   | 23.9           |
| HF60_950 | 299.4 ± 7.0                   | 739                  | 821                   | 19.2           |

3.3. Impact Toughness of Hot Forged F70 High-Strength Steels

Figure 8 shows Charpy impact test results of the F70 high-strength steels according to the austenitizing temperature and hot forging ratio. In order to evaluate the absorbed energy and ductile brittle transition behavior, charpy impact test were conducted from 60 °C to −100 °C. In case of 20% hot forging (Figure 8a), the upper shelf energy (USE) was 202 J and DBTT was −10 °C at an austenitizing temperature of 850 °C. As the test temperature decreases, the absorbed energy decreased indicating typical characteristic of BCC materials ductile brittle transition behavior. In all hot forging conditions, the impact characteristics at an austenitizing temperature of 850 °C showed the best performance with the lowest DBTT temperature (Figure 8a–c) because all grain boundaries formed HAGBs at 850 °C with the lowest dislocation density and high fraction of PF with excellent toughness [8]. As the austenitizing temperature increased, the absorbed energy decreased and the DBTT temperature increased. The fraction of PF structure decreased as the austenitizing temperature increased, and the fraction of the BF and M with high dislocation density, lath structure and low toughness increased. In addition, the impact characteristics at an austenitizing temperature of 950 °C were higher than that of 900 °C under all hot forging conditions possibly owing to the difference in crack resistance and crack propagation path microstructure. The instrumented data analysis and fracture surface analysis was performed to analyze the absorbed energy and crack propagation according to the type and fraction of microstructures.

![Figure 8](image-url)

**Figure 8.** Charpy impact property of F70 high-strength steels after heat treatment: (a) 20% hot forging, (b) 40% hot forging and (c) 60% hot forging.

Figure 9 shows the load–displacement curve obtained by the instrumented Charpy impact test at 0 °C according to different austenitizing temperature after 60% hot forging. In Figure 9a–c, the crack initiation energy of austenitizing temperature at 850 °C, 900 °C, and 950 °C was 59.88 J, 51.07 J, and 57.60 J, respectively. Similarly, the crack propagation energy showed a big difference depending on the austenitizing temperatures (99.39 J, 14.61 J, and 53.97 J, respectively). It was evident that the total absorbed energy ($E_i + E_p$)
was determined by the crack propagation energy, and such difference in crack propagation energy was dependent on the difference in the crack propagation path according to the microstructure. Subsequent fracture surface analysis was performed to analyze the crack propagation path according to the microstructure, described as follows.

Figure 9. Load–displacement curves obtained by the instrumented Charpy impact test at 0 °C of F70 high-strength steels after heat treatment: (a) HF60_850, (b) HF60_900 and (c) HF60_950.

Figure 10 shows the microstructure of the cross-sectional area of the fractured Charpy impact specimens at 0 °C after 60% hot forging. Figure 10 a–c shows at low magnification (×500) using an optical microscope to observe the overall crack propagation mode, and Figure 10 d–f indicated EBSD maps to analyze the correlation between the crack propagation and the microstructure (phase) at higher magnification (×1000). At an austenitizing temperature 850 °C (Figure 10a), the crack propagation path was observed to be short and bent frequently. Especially, the length of the main crack was shortened due to frequent cracking by PF and AF during crack propagation. In general, the crack propagation path caused refraction in HAGBs of 15° or more, and since PF and AF had relatively fine grain and the grain boundaries of HAGBs (Figure 5), the crack propagation path was short and often bent increasing the crack propagation energy. At an austenitizing temperature 900 °C, it was observed that the crack propagated in a straight line for a long time without bending (Figure 10b). The main crack propagated in a straight line between GB and BF during crack propagation (Figure 10e). GB and BF had coarse grains and LAGBs inside the grains (Figure 5), so the effective grain size increased. Effective grains were HAGBs, and the smaller the effective grain size were, the shorter the propagation path and the more often bent [4,38]. Since GB has coarse grain and consisted of island structured LAGBs, the effective grain size was also coarse, and the crack propagation path propagated linearly for a long time during crack propagation reducing the crack propagation energy. In addition, BF also has coarse effective grain size and consists of lath structured LAGBs inside, so that the effective grain size increases in which the crack propagation path propagates linearly, to decrease the crack propagation energy. At an austenitizing temperature of 950 °C (Figure 10c), the crack propagation path was observed as a mixture of a part that propagated in a straight line and the length of the crack was short and bent frequently. The crack propagation path propagated in a straight line in BF, but it was observed that the crack propagation path was relatively short and bent frequently in M (Figure 10f). The effective grain size decreases more in case of M compared to GB and BF due to packets with HAGBs inside the grain. A complex and fine effective grains of M shorten and bent frequently the crack propagation path increasing the crack propagation energy. Therefore, the crack propagation energy was highest at an austenitizing temperature 850 °C with relatively fine grains and high PF and AF fractions made of HAGBs. In addition, it showed the lowest crack propagation energy at an austenitizing temperature 900 °C where the effective grain size was coarse and the fraction of GB and BF was high with LAGBs. Meanwhile, the effective grain size was fine and the fraction of M with HAGBs inside the grain was high at
an austenitizing temperature of 950 °C where the crack propagation energy was between 850 °C and 900 °C.

Figure 10. Microstructure of the cross-sectional area of the fractured Charpy impact specimens at 0 °C after 60% hot forging ratio: (a,d) HF60_850, (b,e) HF60_900 and (c,f) HF60_950.

Figure 11 shows the result of fracture analysis after Charpy impact test at 0 °C according to different austenitizing temperature of 60% hot forging. In the fracture surface under all conditions, ductile fracture and brittle fracture were mixed, and large and small dimples were observed, as shown Figure 11a–c. At an austenitizing temperature of 850 °C (Figure 11a), the crack propagation path was short and often bent by PF and AF, and many dimples of small size were observed. At an austenitizing temperature of 900 °C (Figure 11b), the crack propagation path was propagated by GB and BF, and brittle fracture surfaces were observed a lot. At an austenitizing temperature of 950 °C (Figure 11c), the crack propagation path was short and often bent due to M with a high fraction of HAGBs, so that many dimples were observed with a size smaller than austenitizing temperature of 900 °C. In addition, the brittle fracture was easily propagated by the BF. Therefore, the correlation between the microstructure (phase fraction and effective grain) and impact toughness according to the heat treatment temperature was investigated through impact test results and fracture analysis.

Figure 11. The fracture surface of CVN specimen at 0 °C: (a) HF60_850, (b) HF60_900 and (c) HF60_950.
4. Conclusions

In this study, we investigate the microstructure and mechanical properties according to the austenitizing temperature and hot forging ratio of F70 high-strength steel, and analyzed the correlation between the microstructure and mechanical properties.

- After hot forging and austenitizing, the microstructure of F70 high-strength steel exhibited complex microstructure with various phase (PF, GB, AF, BF and M). As an austenitizing temperature increased from 850 °C to 950 °C, the fractions of PF and GB decreased while the fractions of M and BF increased. In the PF, all grain boundaries consisted HAGBs, and no other grain boundary was inside the grain. In the AF, a LAGBs was inside with a HAGBs similar to that of PF. In GB and BF, grains with a coarse HAGBs and LAGBs were observed in the form of island and lath structure, respectively. In the M, both grain boundaries and packet had HAGBs, and a LAGBs were inside the packet.

- As the austenitizing temperature increased from 850 °C to 950 °C, the yield strength and tensile strength increased but the elongation decreased. As the austenitizing temperature increases, the PF and GB fractions decrease while the BF and M fractions increase. According to microstructure analysis, this is due to a decrease in fractions of PF and GB and an increase in fractions of BF and M with increasing austenitizing temperature.

- In all hot forging conditions, the impact characteristics at an austenitizing temperature of 850 °C showed the best performance with the lowest DBTT temperature because all grain boundaries formed HAGBs at 850 °C with the lowest dislocation density and high fraction of PF with excellent toughness. As the austenitizing temperature increased from 850 °C to 950 °C, the absorbed energy decreased and the DBTT temperature increased. The fraction of PF structure decreased as the austenitizing temperature increased, and the fraction of the BF and M with high dislocation density, lath structure and low toughness increased.

- With instrumented Charpy impact tests, the total absorbed energy was determined by the crack propagation energy, and difference in crack propagation energy was dependent on the difference in the crack propagation path according to the microstructure. The crack propagation energy was highest at an austenitizing temperature of 850 °C with relatively fine grains and high PF and AF fractions made of HAGBs. In addition, it showed the lowest crack propagation energy at an austenitizing temperature of 900 °C where the effective grain size was coarse and the fraction of GB and BF was high with LAGBs. Meanwhile, the effective grain size was fine and the fraction of M with HAGBs inside the grain was high at an austenitizing temperature of 950 °C where the crack propagation energy was between 850 °C and 900 °C.

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