Microstructural Evolution and Mechanical Properties of FCAW Joints in 9% Ni Steel Prepared with Two Types of Ni-Based Weld Metals

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Abstract
The microstructural and mechanical evaluation of 9% Ni steel with flux-cored arc welding (FCAW) was performed with two different Ni-based weld metals: Inconel 625 and Hastelloy 609. The weld metals showed microstructural changes depending on the temperature gradient and crystal growth rate for each region during the cooling after welding. A cellular/planar growth was exhibited at the bottom of the weld metal, which was rapidly cooled in contact with the cold base metal. Columnar dendrites were exhibited in the central region that cooled relatively slowly, and precipitates were observed in the interdendritic region. The weld joints between the base metal and weld metal have a compositional transition region due to dilution. The transition region comprised a martensite layer and a γ-phase cellular/planar layer, according to the compositional distribution. In the low-temperature toughness test, the absorbed impact energies were 89 and 55 J for Inconel 625 and Hastelloy 609, respectively. When Inconel 625 is used as the weld metal compared to Hastelloy 609, the high content of the γ-stabilizer and martensite start temperature decreasing elements leads to the formation of a thicker γ-phase layer and thinner martensite layer in the transition region. In addition, the high content of these elements suppresses the martensite transformation and maintains the stability of the weld joint interface even at low temperatures, resulting in the higher absorbed impact energy. The microstructure of weld joints and its influences on mechanical properties help to improve the practical application of 9% Ni steel FCAW.

Keywords 9% Ni steel · Flux-cored arc welding · Microstructure evolution · Transition area · Low-temperature toughness

1 Introduction

In 2020, the International Maritime Organization (IMO) imposed a new upper limit of 0.5% sulfur content in the shipping fuel (reduced from 3.5%) [1]. The new regulation aims to reduce the harmful sulfur oxide emissions from ships and prompts the shipping industry to adopt environmentally friendly fuels. Liquefied natural gas (LNG), a well-established low-sulfur alternative to petroleum, is drawing attention as a next-generation clean fuel because it significantly reduces the emission of air pollutants such as sulfur oxide, nitrogen oxide, carbon dioxide, and soot [2]. The LNG-fueled ships require cryogenic fuel tanks suitable for the long-term storage of LNG at temperatures below its liquefaction temperature of −162 °C. One of the most widely used materials for the construction of such cryogenic storage tanks is 9% Ni steel, owing to its good tensile strength and excellent impact toughness, which makes it highly suitable for cryogenic applications [3, 4]. Ni-based superalloys such as Inconel® and Hastelloy® are used as welding materials for this steel and mainly comprise an austenite phase, resulting in good crack resistance and excellent low-temperature fracture toughness [4, 5]. These properties make them highly suitable for welding 9% Ni steel.

The conventional welding methods such as shielded metal arc welding (SMAW) and submerged arc welding (SAW) have been widely used for 9% Ni steel and Ni-based weld metal welding [4–8]. Among them, SMAW is a welding
method generating an arc between a welding rod and a base material and is widely applied to the manufacturing of liquefied natural gas (LNG) tanks. However, since the welding speed is relatively slow and it is normally performed manually, the quality of the welded part varies according to the skill of the operator. Flux-cored arc welding (FCAW) is a semi-automatic or automatic welding process that provides a continuous supply of flux-cored wire (FCW). FCAW can be welded with uniform mechanical properties compared to SMAW and has a higher deposition rate and productivity; therefore, it is an alternative to SMAW. Recently Kim et al. investigated the effects of the FCAW and SMAW welding processes on the fracture behavior of 9% Ni steel, indicating the applicability of FCAW [9]. However, considering the types of weld metal, welding conditions, and research topics, the number and diversity of studies on FCAW joints of 9% Ni steel are still inadequate.

On the other hand, the Ni-based weld metal part was reported to be a weak part in low-temperature fatigue due to micro-voids or precipitate–dislocation interactions appearing near the precipitate [10, 11]. For low-temperature applications, it is necessary to clearly understand the microstructure of the weld metal part. Also, in the weld joint between dissimilar metals (Fe-based base material and Ni-based superalloys), a dilution area is created where the material microstructure and properties change. Microstructure of dilution area, size of the area, and mismatching of mechanical properties affect low-temperature impact properties. Mu et al. mentioned fluctuations in the microstructure at the fusion boundary and reported that the low-temperature impact toughness of the region where the widened fusion boundary was formed was relatively reduced [12]. As such, the dilution phenomenon plays a crucial role in determining the toughness of the cryogenic storage tanks made by 9% Ni steel [12, 13].

It is important to understand the weld-joint properties of 9% Ni steel with Ni-based superalloys, and their increasing use in manufacturing cryogenic LNG storage tanks requires a more in-depth examination of the mechanical and structural properties of the Ni-based weld joints to ensure an improved low-temperature toughness. Nevertheless, in-depth study has rarely been conducted on the compositional change and low-temperature toughness properties of the FCAW joint and its transition region. The microstructure and mechanical properties of the FCAW joint remain to be explored.

In this study, the Ni-based superalloys, Inconel® and Hastelloy®, were used as welding metals for 9% Ni steel. The microstructure of the weld metal and transition areas was systematically examined depending on the type of Ni-based weld metal used for the FCAW of the 9% Ni steel base metal. In addition, the effects of microstructure and composition of the transition region on the low-temperature toughness were studied. For this, a metallographic analysis of the microstructure was performed in each of the joint interfaces formed by single beads of Hastelloy 609 and Inconel 625 weld metal, which were welded under identical conditions.

### 2 Experimental methods

The chemical composition of the base metal and weld metals was investigated using the inductively coupled plasma (ICP, Perkin Elmer, Optima 7300DV, USA) analysis. The composition of the 9% Ni steel (Nippon steel & Sumitomo metal corporation) and Ni-based weld metals is listed in Tables 1 and 2, respectively. FCW with a diameter of 1.2 mm was the weld metal. The welding was carried out in a single pass using FCAW for each of the weld metals. The welding conditions were summarized in Table 3. The same welding conditions were used for all the specimens, whereas different weld metal samples were used each time. The microstructure of the weld joint was analyzed using optical microscopy (OM, Hirox, MXB-5000REZ, Japan) and field emission-scanning electron microscopy (FE-SEM, Hitachi, SU8230, Japan) equipped with an electron backscatter diffraction (EBSD, Oxford, Aztec HKL Nordlys Nano, UK) equipment. Compositional analysis of the weld joint was performed through

| Weld metal | Ni   | Cr   | Mo   | Nb+Ta | Fe   | W    | Si   | Mn   | Ti   | C    | Cu   | P    | S    |
|------------|------|------|------|-------|------|------|------|------|------|------|------|------|------|
| Hastelloy 609 (609 weld metal) | Bal. | 15.700 | 16.200 | 6.600 | 3.300 | 0.270 | 0.270 | 0.010 | 0.012 | 0.002 |
| Inconel 625 (625 weld metal)   | Bal. | 21.600 | 8.600  | 3.570 | 1.800 | 0.370 | 0.280 | 0.110 | 0.030 | 0.030 | 0.010 | 0.003 |

- **Table 1** Chemical composition of 9% Ni steel (wt. %)
- **Table 2** Chemical composition of weld metals (wt. %)
point and line measurements using energy-dispersive spectrometry (EDS, Oxford, Ultim Max 100, UK) [14]. For the crystal structure analysis, X-ray diffraction (XRD, PANalytical, EMPYREAN, UK) was performed within a 2θ scan range of 20–100 ° with Cu-Kα radiation (λ = 1.54056 Å). The scan speed was 3 °/min scaling with a step size of 0.02 °. The beam size was 7 mm 1/2 degree. A multi-layer FCAW process was performed to evaluate the low-temperature impact toughness of 9% Ni steel with different weld metals. The multi-layer welded sample had a V-type notch with a 2 mm depth in the weld metal region. Charpy impact tests were performed for the weld metal area in a cryogenic environment at −196 °C.

### 3 Results

Figure 1 shows the XRD pattern of the base metal, weld metals, and weld-joint fabricated using FCAW of 9% Ni steel with Hastelloy 609 and Inconel 625. The base metal shows the diffraction peaks of martensite and austenite-γ-phases (Fig. 1a) [15–17]. However, the weld metal and the weld joint of the base metal and weld metal mainly show diffraction peaks of the austenite-γ-phases. The austenite peaks for Hastelloy 609 and its joint interface (Fig. 1c, e) appear at a lower scattering angle compared to those of the sample welded with Inconel 625 (Fig. 1b, d). The most likely reason for the peak shift is the difference in the solid-solution content of the minor element.

Figure 2 shows the cross-sectional OM and SEM images of the joints welded with Hastelloy 609 and Inconel 625, respectively. With Fig. 2a, d, it is noted that the two weld joints are composed with clearly distinguishable weld metal, joint interface, and the heat-affected zone (HAZ) of the base metal. The center of the Hastelloy 609 bead comprises columnar dendrites elongated upward, while the bottom of the bead exhibits dendrite-to-cellular transition morphologies (Fig. 2b, c). Precipitates of various sizes and shapes are located at the interdendritic regions. Similar precipitates were also observed at the bottom of the bead. In addition, the number of precipitates decreased toward the bottom of the bead. EDS was performed to investigate the compositional distribution in the weld metal, and the results are presented in Table 4. The Mo content in the interdendritic regions is higher than that in the dendritic regions, indicating that Mo tends to be rejected to an interdendritic residual liquid. Moreover, the interdendritic precipitates also have high contents of Mo, indicating that high Mo concentrations promote the formation of precipitates. The precipitates located at the interdendritic regions are inferred to be in the ρ phase based on the compositional analysis of Cieslak et al. [18] and Qiu et al. [17]. These results imply that the solidification of the molten Hastelloy 609 starts in the γ-phase, while the ρ phases start to form at later stages. To understand the solidification mechanism, the isothermal state diagram and schematic diagram of Ni-Cr-Mo are shown in Fig. 3. In the ternary phase diagram of Fig. 3a [18], the composition of Hastelloy 609 is indicated by a blue dot, and the composition change according to solidification is indicated by an arrow. The tail of the arrow indicates the composition of the dendrite core crystallized in the melt, and the head of the arrow indicates the final solidification composition of the interdendritic region. During the welding process, the weld metal will be melted (Fig. 3b) and crystallized as it cooled. As can be seen from the phase diagram, depending on the equivalent composition of the alloy, the melt will be in the γ-single-phase region at the start of solidification. Thus, γ-phases form as the primary phase, and depending

| Table 3 | The welding conditions for 9% Ni steel and Ni-based weld metals |
|---------|------------------------------------------------------------|
| Welding parameters | | |
| Welding machine | CMT/KUKA |
| Base metal | 9% Ni steel |
| Weld metal (wire) | Ni-based alloy (Hastelloy 609, Inconel 625) |
| Shield gas | Ar-18% CO₂ |
| Welding current [A] | 200 |
| Welding speed [cm/min] | 20 |

Fig. 1 XRD results of the weld joints from the welding of 9% Ni steel with Hastelloy 609 and Inconel 625 weld metals; (a) 9% Ni steel, (b) Inconel 625, (c) Hastelloy 609, (d) weld joint interface with Inconel 625 and base metal, (e) weld joint interface with Hastelloy 609 and base metal
on the equilibrium composition, excess elements will diffuse into the surrounding residual melt (Fig. 3c). As a result, the composition of the residual liquid phase will change along the arrow in Fig. 3a, forming a Mo-rich liquid phase. Therefore, the residual liquid phase will be placed in the γ+ρ two-phase region indicated by the arrow head, and upon further cooling, it will solidify into the γ-interdendritic region and ρ precipitates (Fig. 3d) [5, 18, 19]. As can be seen from the phase diagram, the low Cr content of Hastelloy 609 is far from the triple point of γ, ρ, and σ, so that the ρ phase is more stable than σ [17, 18]. On the other hand, the μ phase, which results from the long-term phase transformation of the ρ phase, is not present as heat was not applied for a sufficiently long time during FCAW [18].

The SEM images of the Inconel 625 bead reveal columnar-dendritic growth from most areas of the center of the bead till the bottom (Fig. 2e). Dendrite-to-cellular transition morphologies appear in some areas near the weld joint interface, as shown in Fig. 2f. Moreover, similar to Hastelloy 609, precipitates are observed in the interdendritic regions. The compositional analysis reveals that the interdendritic regions are relatively rich in Mo and Nb (Table 4). These results suggest the liquid segregation of Mo and Nb during the weld joint solidification [6, 20]. The compositional analysis of the interdendritic precipitates reveals that the Nb-enriched phase is NbC. It has been established that the Inconel 625 alloy solidifies to the γ-phase, M6C, and Nb-rich phase (NbC, Laves) from its liquid state through the following process: L → L+ γ → L+ γ + NbC → L+ γ + NbC +M6C → L+ γ + NbC +M6C +Laves → γ + NbC +M6C + Laves [20–22]. The secondary phase (NbC) has a high Nb content and can be distinguished from the Laves phase. It was difficult to identify the Laves phase and M6C even through magnified observations in this study. It was envisaged that a more detailed sample analysis in the future might be able to overcome this limitation.

Table 4 The results of EDS point analysis (wt. %)

| Analysis region | Phase                  | Ni   | Cr  | Fe  | Mo  | Nb  | W  |
|-----------------|------------------------|------|-----|-----|-----|-----|----|
| Hastelloy 609   | Interdendrite (Mo-rich) | 48.28| 15.21| 18.52| 15.64| 2.36|    |
|                 | Dendrite γ-phase       | 53.35| 13.97| 20.41| 10.08| 2.19|    |
|                 | Precipitate ρ-phase    | 26.45| 13.49| 11.72| 43.01| 5.33|    |
| Inconel 625     | Interdendrite (Mo, Nb-rich) | 59.99| 21.70| 8.75 | 7.12 | 2.44|    |
|                 | Dendrite γ-phase       | 61.46| 21.42| 9.51 | 6.22 | 1.39|    |
|                 | precipitate NbC        | 10.34| 7.87 | 2.30 | 4.77 | 74.73|    |

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The SEM images of the joint interface welded with Hastelloy 609, together with the EDS line analysis and EBSD images of the interface area, are presented in Fig. 4. In this case, elements with less than 1 wt. % content are excluded from the analysis since such low values are considered to be below the accuracy limit of the EDS measurement. Compositional analysis was performed along a straight line over the joint interface, as shown in Fig. 4a. The results from the corresponding compositional analysis, shown in Fig. 4b, reveal a transition area in the Hastelloy 609 sample near the joint interface, where the elemental composition changes linearly in the direction from the base metal toward the weld metal. This transition area has a thickness of approximately 20 μm, where the Fe content decreases rapidly, while the Ni, Cr, Mo, and W contents increase. An area farther away from the interface (region 3 in Fig. 4a) exhibits 20 wt. % Fe, indicating a considerably wide dilution area. The transition area consists of a uniform martensite layer (region 1 in Fig. 4c, d) and a γ-phase-planar growth area (region 2 in Fig. 4c, d) [12].

The SEM image of the Inconel 625 joint interface (Fig. 5) reveals a similar structure to that of the Hastelloy 609 joint interface (Fig. 5a). The transition area near the interface is 33 μm thick. Within the transition area, the Fe content decreases when scanning in the direction from the base metal toward the weld metal, while the Ni, Cr, Mo, and Nb contents increase simultaneously (Fig. 5b). A high Nb content is detected in the interdendritic precipitates, confirming the presence of an NbC phase. In Fig. 5c, d, two layers can be distinguished within the transition: the martensite layer (region 1 in Fig. 5c, d) and the γ-phase-cellular growth area (region 2 in Fig. 5c, d).

To assess the toughness of the weld metals after welding, multilayer welding specimens, prepared from the same base metal and weld metal pairs, were subjected to low-temperature impact tests. The absorbed impact energies were average values of 55 J and 89 J for Hastelloy 609 and Inconel 625, respectively, as shown in Fig. 6. The error bars indicate the range of the minimum and maximum values of the measured impact absorption energy. These values greatly exceed the impact absorption energy standards stipulated by the classification regulation (standard of Charpy impact energy: at −196 °C, 27 J in the transverse direction and 41 J in the longitudinal direction). For comparison, the previously reported impact absorption energy according to the welding technique and weld metal for 9% Ni steel is presented in Table 5 [9, 12, 23, 24]. Different impact absorption energies were shown depending on the weld metal used and welding conditions. Even using the FCAW method of this study, it was confirmed that it has low-temperature impact.
Fig. 4 Joint interface welded with Hastelloy 609: (a) SEM image, (b) EDS line analysis, (c) magnified SEM image, (d) EBSD image

Fig. 5 Joint interface welded with Inconel 625: (a) SEM image, (b) EDS line analysis, (c) magnified SEM image, (d) EBSD image
toughness similar to that of commonly used SMAW welds. This shows the potential of FCAW as an alternative technology to SMAW.

### 4 Discussion

The microstructural analysis of the weld metal showed that the type of the dendritic growth varied depending on the position within the weld metal. The columnar dendrite structures in the central part of the weld bead exhibited well-defined secondary arms. At the bottom of the bead, the secondary dendritic arms nearly disappeared, and the morphology transitioned from dendritic to cellular. In this study, the temperature gradient (G) and the crystal growth rate (R) are the two main parameters that govern the microstructure of the weld joint during solidification. The G is defined as the tangent of the melt pool temperature profile with distance, and R is the moving speed of the solid–liquid interface. These solidification shapes according to G and R are shown in Fig. 7 [25, 26]. GR is the grain size during solidification, and the G/R ratio is a factor that determines the preferred shape during solidification. With the decrease of G and the increase of R, the microstructure transitions from planar through cellular, cellular-dendritic, and columnar-dendritic, to equiaxed dendritic [27]. The bottom of the weld bead that is in contact with the cold base metal cools more rapidly during the welding process, while the center of the bead cools slower. That is, at the bead bottom, the high heat extraction due to rapid cooling causes high G/R values, and as a result the planar or cellular growth predominantly occurs at the bottom of the bead. In contrast, at the center of the bead, there is less heat extraction compared to the bead bottom. Thus, it cools slowly, resulting in low G and high R. With low G/R, a columnar dendrite growth with distinctively developed secondary arms is formed in the center part of the weld bead [28]. This microstructural transition was observed during the melt pool solidification process of several different materials. Akbari et al. performed control and monitoring of the melt pool in laser deposition of stainless steel 316L material [25]. The first deposition layer, which

| Impact absorption energy (J) | Weld metal | Welding method | Notch location | Reference |
|-----------------------------|------------|----------------|----------------|-----------|
| 41 in longitudinal direction | Inconel 625 | FCAW | Weld metal | Present study |
| 27 in transverse direction  | Hastelloy 609 | FCAW | Weld metal | Present study |
| 89                          | DW-N70S    | FCAW | Fusion line | [12]      |
| 55                          | Inconel 625 | FCAW | Weld metal | [9]       |
| 43–54                       | ENiMo13-T  | FCAW | Weld metal | [23]      |
| 73                          | Inconel 625 | SMAW | Weld metal | [24]      |
| 95                          | ENiCrMo-6  | SMAW | Weld metal | [9]       |

![Fig. 6 Results of Charpy impact test performed for the weld metal areas in a cryogenic environment at -196 °C](image)

![Fig. 7 Solidification morphology according to the temperature gradient and crystal growth rate [25, 26]](image)
was rapidly cooled, showed fine cellular microstructure, and thereafter, it showed a gradual change to columnar and dendrite at the top layer with less heat extraction. In addition, structural transition from equiaxed to planar was reported in gas tungsten arc welding (GTAW) of Al alloy [29], and Mu et al. confirmed the same dendrite formation in Ni-based weld metal center part of FCAW joint [11].

During welding, a dilution area is formed between the 9% Ni steel and Ni-based weld metal, where both metals are mixed. The transition area of the joint interface where a compositional change occurs due to the dilution process consists of the martensite layer (region 1) and the γ-phase-planar/cellular growth area (region 2). This microstructure of the transition area can be attributed to the cooling rate and the elemental composition of the area [12, 13]. Since Ni is a γ-phase stabilizing element, the Ni content in the base metal (9% Ni steel) helps to stabilize the γ-phase at low temperatures [30]. In the case of Hastelloy 609, the γ-phase in region 2 with a Ni content of 20–45 wt. % was preserved despite the Fe dilution. Additionally, planar growth morphology appeared in the same region due to the large temperature gradient and low growth rate induced by the rapid cooling from the cold base metal. In region 1, high Fe dilution of 60–90 wt. % and low Ni content of 10–20 wt. % were observed. Thereby, the martensite in the corresponding area became a relatively stable phase. Moreover, rapid cooling accelerates the martensite formation; hence, the presence of a fine martensite microstructure indicates rapid cooling. In the case of Inconel 625, the joint interface was also structured in two layers (region 1 and region 2). The factors that governed the growth morphology of the layers were the rapid cooling and the composition. The martensite layer was formed in areas with a high Fe and low Ni content, while the γ-phase-cellular morphology appeared in areas with a high Ni and low Fe content. Similarly, compositional variation through line analysis at the interface of a 9% Ni steel FCAW joint has been reported [12, 13]. Weld metal and welding conditions different from this study showed differences in the transition region structure and microstructure shape, but the two-phase formation of martensite and γ was also reported.

In addition, as a result of the dilution phenomenon, the lattice parameter of the γ-phase increases due to the solid solution of Mo and Nb atoms (with larger atomic radii than Ni and Fe) in the γ-phase during welding (for reference, the atomic radii of Ni, Fe, Mo, and Nb are 0.124, 0.126, 0.139, and 0.146 nm, respectively) [31, 32]. This lattice expansion generates a low-angle shift of the γ-peak in the XRD spectrum. The EDS data, summarized in Table 4, indicate that the Hastelloy 609 sample has a Mo content of approximately 10–16 wt. %, while the Inconel 625 sample has Mo and Nb contents in the range of 7–10 wt. %. Therefore, the γ-phase peaks of the Hastelloy 609 bead region and its joint interface appear at a lower diffraction angle because of the higher Mo solid-solution content. Moreover, the intensity of the γ-phase peak is higher at the weld joint interface compared to the peak intensity at the weld metal area, indicating an intense texture growth. The most likely reason for the strong γ-phase peak is the aligned dendrite-to-cellular transition morphology that developed near the joint interface (or the bottom area of the weld metal bead) due to heat transfer [17]. There is a deviation in the preferential growth direction for the joint interface areas by Hastelloy 609 and Inconel 625. The low volume fraction of the NbC and ρ phases prevents their reliable identification in the XRD pattern.

There are microstructural changes that occur due to the cooling rate variations, by the position within the Hastelloy 609 and Inconel 625 beads, during the welding process. In both cases, aligned columnar dendrites were formed in the center of the bead, while the morphology changed from dendrite-cellular to cellular/planar growth toward the bottom of the bead. Moreover, precipitates were formed in the inter-dendritic regions of both samples. The transition area of the weld joint was structured with two layers (martensite layer and γ-phase cellular/planar layer) depending on the actual Fe and Ni content resulting from the dilution in the transition area. However, the microstructure of the transition area differs for the two weld metals. The use of Inconel 625 resulted in a transition area of 33 μm, compared to a 20-μm-thick transition area for Hastelloy 609, with an 8-μm-thick martensite layer. In contrast, the martensite layer for Hastelloy 609 was 11 μm thick. Elements such as Cr, Mo, and Ni lower the onset temperature for martensite transformation (martensite start temperature, Ms) in steel [12, 13, 33]. As Ms is lowered by the increase of the corresponding elements, the γ-phase is relatively stable up to a lower temperature. This implies an increase in the γ-phase volume fraction and the inhibition of martensite formation [34]. The high content of those elements in the transition area of Inconel 625 can explain the thick γ-phase layer and thin martensite layer observed in the transition. In this regard, Dupont and Kusko used an electroslag welding process to weld Inconel 625 (Ni-based alloy) and 309L stainless steel (Fe-based alloy) weld metal, respectively, and mentioned the martensite layer thickness difference according to the composition gradient of the interface [13]. Compared to the Fe-based weld metal, Inconel 625, which has a higher content of Ni, Cr, and Mo, showed a thin martensite layer in the sample, supporting the results of this study. Table 6 presents the results of studies that mention the compositional transition area along with the thickness of each layer in the transition area of this study [12, 13]. In common, it was found that a thin martensite layer was formed compared to the γ-phase layer when weld metal with high Ms-decreasing element (γ-stabilizer) content was used.

This microstructure and the elemental composition of the transition area can influence the low-temperature fracture
toughness. At low temperatures, the difference in the thermal expansion coefficients between the Ni-based weld metal and the Fe-based base metal can cause cold shrinkage and mechanical stress at the joint interface. Such stresses can induce a transformation from austenite γ-phase to martensite in the joint transition area. In turn, the stress from the martensite transformation can augment the deformation in the adjacent HAZ, and the residual deformation can lead to crack propagation [12, 34–37]. Furthermore, the two materials commonly exhibited precipitation formation in the interdendritic regions upon microstructural analysis (the ρ phase for 609 weld metal and NbC for 625 weld metal). Studies have reported that even a small number of interdendritic precipitations can induce intergranular crack propagation at low temperatures, thus promoting brittle failure of the sample [6]. The intergranular crack propagation mode can be explained in three ways as follows. First, small precipitates are formed, and the crack bypasses them. On the other hand, the second and third appear mainly when large precipitates are formed. The crushing mode means that the precipitates are broken during crack propagation, and the interface mode represents crack propagation along the interface between the γ-matrix and the precipitates. The connection between the γ-matrix and the precipitate weakens due to the difference in the contraction rate between the γ-phase and the precipitate, which deteriorates the low-temperature fracture toughness. This crack propagation mechanism diagram is illustrated in Fig. 8. However, the impact absorption energies of Hastelloy 609 and Inconel 625 were higher than the value suggested in the classification regulations. This is because Hastelloy 609 and Inconel 625 are Ni-based austenitic metals and comprise a γ-phase that does not show brittle fracture at low temperatures. The excellent low-temperature toughness of the γ-phase has already been reported in many studies [38, 39]. At this time, the impact absorption energy was found to have a higher value than when Inconel 625 was used as a weld metal. The high content of the γ-stabilizing and Ms-decreasing elements present in Inconel 625 prevents martensite transformation. The γ-phase stabilization process (martensite inhibition process) enhances the stability of the joint interface at low temperatures. In addition, in Inconel 625, the initially thin martensite layer and thick

| Thickness | Compositional transition area | Martensite layer | γ-phase layer | Reference |
|-----------|-------------------------------|-----------------|--------------|-----------|
| Hastelloy 609 | 20 | 11 | 9 | Present study |
| Inconel 625 | 33 | 8 | 25 | Present study |
| DW-N70S | 47 | 8–14 | 33–39 | [12] |
| Inconel 625 | 14 | 1–3 | 11–13 | [13] |
| 309L stainless steel | 52 | 30–37 | 15–22 | [13] |

Fig. 8 Schematic diagram of intergranular crack propagation in a cryogenic environment
\( \gamma \)-phase layer that help to maintain the residual deformation at a low level increase its toughness. Consequently, a high low-temperature impact absorption energy of 89 J and an outstanding low-temperature toughness were observed when Inconel 625 was used.

To summarize, depending on the type of weld metal utilized, the difference in the dilution area extent and microstructure can be generated even under equal welding conditions. The thick martensite layer in the transition area along with the above-mentioned precipitate can negatively affect the fracture toughness. On the other hand, the following three factors contribute to the improvement of low-temperature toughness.

1. Microstructure with \( \gamma \)-phase as the main phase
2. High content of Ms-decreasing elements (\( \gamma \)-stabilizer)
3. Transition area consisting of a thin martensite layer and a thick \( \gamma \)-phase layer

The combined effect of these factors results in the final low-temperature toughness characteristics. In this study, the microstructure of the transition region of the joint interface as well as the weld metal was studied in detail according to the weld metal, and the difference in low-temperature toughness was explained through this. This study will be helpful in the practical application of FCAW, and further discussion on the microstructure and mechanical properties according to more diverse weld metal types and FCAW welding conditions is required in the future.

5 Conclusions

The mechanical properties of the FCAW weld joints between 9\% Ni steel and Ni-based weld metals (Hastelloy 609 and Inconel 625) were investigated using the microstructure analysis. The findings of this study can be summarized in the following points:

- At the heterojunction between the 9\% Ni steel (Fe-based base metal) and Ni-based weld metals, a compositional transition area was created due to a dilution process at the joint interface. The transition areas for Hastelloy 609 and Inconel 625 were 20 \( \mu \)m and 33 \( \mu \)m, respectively. Depending on the distribution of Fe and Ni in the transition area, two clearly distinguished layers were identified: the martensite layer, where the Fe content is relatively high, and the \( \gamma \)-phase-cellular/planar layer, where the Ni content is high. The microstructure and compositional distribution at the welded part play an important role in determining the low-temperature fracture toughness of the material. First, the observed precipitates in the interdendritic regions facilitate the intergranular crack propagation, thus adversely affecting the material toughness. In contrast, the high content of the \( \gamma \)-stabilizing and Ms-decreasing elements impedes martensite transformation, resulting in a relatively thick and stable \( \gamma \)-phase layer. Thus, excellent toughness can be achieved by hindering additional martensite transformation and maintaining stability at the joint interface. These microstructures were found in the transition area with the application of Inconel 625, which is considered to have a higher toughness compared to Hastelloy 609.

- In the Hastelloy 609 weld joint, an 11-\( \mu \)m martensite layer was observed, amounting to nearly half the thickness of the transition area. In contrast, despite having a thicker transition zone, the thickness of the martensite layer in the Inconel 625 weld joint was approximately 1/4 of the total thickness of the transition zone. In the Inconel 625 transition area, the high content of elements such as Cr, Mo, and Ni, which reduce the martensite transformation starting temperature of steel, inhibited the formation of martensite and increased the austenite volume fraction (thin martensite layer and thick \( \gamma \)-phase layer).

- The variations in temperature gradients and crystal growth rates result from different cooling rates in various positions inside the bead. In the bead center, the relatively low-temperature gradient and high growth rate result in directional columnar dendrite structures. Toward the bottom of the weld bead, where the temperature gradient is higher and the growth rate is lower, a transition from dendrite-cellular structures to cellular/planar morphologies was observed. Additionally, precipitates of the \( \rho \) phase and NbC were found in the interdendritic regions of the Hastelloy 609 and Inconel 625 bead, respectively.

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Declarations

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