

Effect of Nb Content on Microstructure, Defects, and Mechanical Properties of NiCrFe-7 Weld Metal Postprint

Authors: Mo Wenlin, Zhang Xu, Shanping Lu, Li Dianzhong, Li Yiyi

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Abstract

Using methods of phase diagram calculation, filler metal design, filler metal preparation, field welding, and metallographic examination of weld metal, the effect of Nb content on the microstructure, defects, and mechanical properties of NiCrFe-7 weld metal was investigated. The results show that with increasing Nb content in the weld metal, intragranular MX (M=Nb, Ti, X=C, N) precipitates increase, the initial precipitation temperature of grain boundary M₂₃C₆ (M=Cr, Fe) decreases, grain boundary M₂₃C₆ precipitates decrease, and M₂₃C₆ transforms from a multi-row continuous distribution to a single-row discrete distribution; the number and size of high-temperature ductility-dip-cracking (DDC) cracks in the weld metal decrease; the strength, ductility, and bending properties of the weld metal are significantly improved.

Full Text

Preamble

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Effect of Nb Content on Microstructure, Welding Defects and Mechanical Properties of NiCrFe-7 Weld Metal

MO Wenlin^{1, 2}), ZHANG Xu¹), LU Shanping^{1, 2}), LI Dianzhong¹), LI Yiyi¹)

¹) Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016

²) Key Laboratory of Nuclear Materials and Safety Assessment, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016

Correspondent: LU Shanping, professor, Tel: (024)23971429, E-mail: sh-plu@imr.ac.cn

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Abstract

Ni-based filler metal is one of the most important filler metals in building the key components of nuclear power plants; however, ductility-dip-cracking (DDC) and inclusion defects form easily in the weldment and need to be repaired afterward. The precipitation of M₂₃C₆ (M=Cr, Fe) at grain boundaries promotes the nucleation and propagation of DDC. Adding Ti can form Ti(C, N) and reduce M₂₃C₆ precipitate at grain boundaries, which reduces DDC in the weld metal. However, increasing Ti content in filler metal causes inclusion defects. Nb replacing part of Ti in Ni-based filler metal is proposed in this work. The reduction of Ti in filler metal is to reduce the sensitivity of inclusion defects in the weld metal. Nb can form MX (M=Nb, Ti, X=C, N) precipitates to reduce the M₂₃C₆ and DDC in weld metal. The effect of Nb on the size, number, and location of MX and M₂₃C₆ in Ni-based weldment has been investigated systematically in this work. Phase diagram calculations show that Nb is an element forming high temperature MX precipitate, and its affinity with oxygen is poor and not easy to form oxide. According to the phase diagram calculations, five different filler metals are designed and made with 0, 0.4%, 0.7%, 0.85%, and 1.1% Nb content. The results show that the intragranular precipitates are distributed along sub-grain boundaries. The intragranular precipitate for the Nb-free weld metal is Ti(C, N), whereas the intragranular precipitate in the Nb-bearing weld metals is MX. For the increased Nb in weld metals, more MX is produced, and more C is fixed within the grain. As the Nb content increased in weld metals, the initial precipitation temperature of M₂₃C₆ decreases, the intergranular M₂₃C₆ precipitate decreases, and M₂₃C₆ turns discretized at grain boundaries. As Nb content increases in weld metals, the total crack length of DDC decreases. When the Nb content is over 0.85%, little DDC is found in the weld metals. The addition of Nb can improve the tensile strength, plasticity, and bending property of the weld metals.

KEY WORDS NiCrFe-7 weld metal, ductility-dip-cracking, Nb, M₂₃C₆, MX, mechanical property

NiCrFe-7 nickel-based welding consumables are primarily used in the fabrication of key components such as nuclear reactors and steam generators in nuclear island main equipment. The performance of nickel-based welding consumables

is unstable during field welding, and two types of welding defects commonly appear in the weld metal: ductility-dip-cracking (DDC) and inclusion defects.

DDC belongs to a category of welding hot cracks that form at high temperatures after complete solidification. It exhibits intergranular characteristics and lacks intergranular liquid films, making it difficult to detect completely using conventional non-destructive testing methods. DDC often becomes the origin of other cracks, posing significant potential hazards.

Extensive research on DDC has been conducted both domestically and internationally. To test and observe DDC susceptibility in nickel-based welding consumables, Nissley and Lippold [1] developed a strain-to-fracture (STF) DDC testing method based on Gleeble, which effectively matches the effects of temperature and strain on DDC. Researchers [2,3] analyzed DDC cracking behavior at local positions through in-situ SEM observations. Wu et al. [4] developed a DDC susceptibility testing method based on high-temperature tensile testing, using the reduction of area after fracture to measure material DDC susceptibility, and proposed multiple DDC formation mechanisms: (1) Impurity element segregation-induced grain boundary embrittlement mechanism—segregation of impurity elements such as S and P at grain boundaries causes grain boundary embrittlement, reducing grain boundary strength and leading to DDC formation [5–8]. (2) Grain boundary migration mechanism—grain boundaries sweep through the matrix to form migrated grain boundaries (MGBs), which straighten the grain boundaries. Long, straight grain boundaries offer less resistance to grain boundary sliding, making DDC more likely to occur on these boundaries. MX precipitates ($M=Ti, Nb$, $X=C, N$) can hinder grain boundary migration and reduce DDC susceptibility [9–16]. (3) M₂₃C₆ ($M=Cr, Fe$) precipitation-induced crack formation mechanism—M₂₃C₆ precipitates at grain boundaries with a coherent relationship to one side of the matrix and a lattice parameter approximately three times that of the matrix, causing coherency strain that easily induces crack nucleation and promotes DDC [17,18].

The DDC susceptibility of nickel-based weld metal is related to the elements and precipitates in the material. Alloy elements affect the type, quantity, and distribution of precipitates, thereby influencing DDC susceptibility. There are two main types of precipitates in nickel-based weld metal. One is MX precipitates, which can reduce DDC susceptibility in weld metal [9–16]. MX precipitates form during solidification and can pin grain boundaries, hinder grain boundary migration, form tortuous grain boundaries, and reduce DDC susceptibility. The other is M₂₃C₆. Researchers [17,18] believe that a large mismatch between M₂₃C₆ and the matrix leads to stress concentration at carbide ends, promoting crack nucleation and causing DDC—the M₂₃C₆ precipitation-induced crack formation mechanism. Our previous research [19–21] showed that M₂₃C₆ promotes DDC crack nucleation and propagation. We prepared various nickel-based welding consumables with different Ti contents to investigate the effect of Ti content on weld metal microstructure, defects, and properties. The results revealed that as Ti content increased, high-temperature Ti(C, N) precipitates increased in the

weld metal, grain boundary C segregation decreased, grain boundary M23C6 precipitates decreased, and both the quantity and size of DDC in the weld decreased, while the strength and ductility of the weld metal simultaneously increased. However, with increasing Ti content, inclusion defects were detected in the weld by penetrant testing. This occurs because Ti also serves as a deoxidizer during welding. As Ti content in the welding material increases, more oxides form in the weld pool, reducing pool fluidity and making it difficult for inclusions to float to the pool surface. Inclusions then aggregate and grow in the pool, remaining in the weld to form inclusion defects [19].

Therefore, adjusting only the Ti content in the material cannot simultaneously satisfy both low DDC susceptibility and low inclusion defect sensitivity. It is necessary to consider adding other MX-forming elements to replace part of the Ti, ensuring no inclusion defects during welding while reducing DDC susceptibility. Nb is one of the MC precipitate-forming elements, and its affinity for oxygen is weak, so it does not serve as a deoxidizer in nickel-based weld metal [22,23]. This work proposes adding Nb to increase high-temperature MX precipitates, fix more C within grains, reduce grain boundary C segregation, decrease grain boundary M23C6 precipitation, and thereby reduce DDC susceptibility.

1. Experimental Methods

Thermo-Calc thermodynamic calculation software was used to calculate the thermodynamic equilibrium phase diagrams of NiCrFe-7 to assist in alloy composition optimization design. The database used was the TTNi8 database developed by ThermoTech, and the alloy elements included in the calculations were Ni, Cr, Fe, C, Si, Al, Mn, N, Ti, and Nb. [Figure 1: see original paper] shows the two-dimensional vertical cross-section phase diagrams of NiCrFe-7 alloy calculated using Thermo-Calc software. The results indicate that adding Nb can form high-temperature TiN and NbC phases, fixing some C. Therefore, Nb replacing Ti can also control the precipitation of medium-temperature M23C6. Reducing Ti content in welding materials can improve weld pool fluidity [24], making it easier for inclusions to float out and reducing weld inclusion defect sensitivity [19]. [Figure 2: see original paper] presents the calculated phase fractions for weld metals with different Nb contents. When Nb content is below 0.4%, no NbC phase forms. When Nb content exceeds 0.7%, NbC forms, and the initial precipitation temperature of M23C6 decreases with increasing Nb content. M23C6 formation depends on grain boundary C content and formation temperature. Lower precipitation temperature makes C diffusion to grain boundaries more difficult, directly affecting the quantity and size of M23C6 formed. This effect is similar to that of Ti content on the initial precipitation temperature of M23C6 [20].

Based on the phase diagram calculations, five welding materials were designed as shown in . The target Nb contents were 0, 0.4%, 0.7%, 0.85%, and 1.1%. Since the initial precipitation temperature of M23C6 no longer decreases when Nb content exceeds 0.85%, and because segregation during welding can cause

solidification and liquation cracks when Nb content exceeds 1.2% [25], no additional Nb was added to the welding materials.

The welding wire manufacturing process included four steps: alloy melting, ingot forging, rod rolling, and wire drawing. A VIM-25 semi-continuous vacuum induction melting furnace was used to prepare 25 kg alloy ingots of the five compositions. After melting, the ingots were cut with a grinding wheel, and surface defects such as heavy skin, porosity, and slag inclusions were removed on a lathe to prepare for forging. The ingots were heated to 1150 °C in a GSM-28 high-temperature box resistance furnace, held for 2 hours, and then forged into 30 mm × 30 mm square bars using a 750 kg air hammer. The square bars were heated to 1150–1170 °C, held for 1 hour, and then rolled into 8 mm diameter rods on a hot rolling mill. Wire drawing consisted of two major steps: coarse drawing and fine drawing. Before coarse drawing, the rods were annealed at 1080 °C in a well furnace, coated with lime and dried, then coarsely drawn to 5 mm diameter. For fine drawing, the rods first underwent electrolytic pickling and online hydrogen annealing at 1080 °C, followed by drawing. After multiple fine drawing passes, the wire was drawn to 0.9 mm diameter, then spooled and packaged. Finally, the wire was inspected for appearance and physical properties according to GB15620-2008 standards, including diameter deviation, cast, and helix. Welding was performed after all appearance properties and chemical compositions met the requirements.

Welding experiments used Q235 steel as the base material with dimensions of 500 mm × 125 mm × 25 mm and a V-groove with a 20° opening angle. Since this study focused only on the properties of the deposited weld metal, nickel-based buttering was applied at the interface between the weld metal and Q235 material to prevent alloy element migration during welding, as shown in [Figure 3: see original paper]. After buttering, multi-pass multi-layer welding was performed using tungsten inert gas welding. A total of 16 layers and 55 passes were welded with a welding current of 140–160 A, voltage of 10.5–10.8 V, travel speed of 165 mm/min, duty cycle of 50%, gas flow rate of 20 L/min, direct current electrode negative polarity, wire feed speed of 2000 mm/min, interpass temperature below 100 °C, and pure Ar shielding gas.

The weld metal cross-section was dissected using electrical discharge machining. Chemical composition analysis of the weld metal was performed using a 6300 inductively coupled plasma (ICP) emission spectrometer. Since different locations in the multi-pass weld metal experience different thermal cycles, leading to variations in the quantity and size of intragranular and grain boundary precipitates [26], the observation location for precipitates in this study was within 5 mm of the geometric center of the weld metal, as indicated by the rectangle in [Figure 3: see original paper]. The weld metal was mechanically ground and polished, then electrolytically etched for 20–30 s at 10 V DC using 10 g H₂C₂O₄ + 100 mL H₂O. DDC length was statistically analyzed using an Observer.Z1m optical microscope (OM), and grain boundary and intragranular precipitates were observed using an INSPECT F50 scanning electron microscope (SEM). Energy

dispersive spectroscopy (EDS) on the SEM was used to analyze precipitate composition. Transmission electron microscopy (TEM) samples were prepared by electrolytic twin-jet thinning using a 10% perchloric acid ethanol solution (volume fraction), and grain boundary precipitates were observed using a Tecnai F20 TEM. Tensile and bend test specimens were sampled and tested according to GB/T 2652 and GB/T 2653. Tensile tests were conducted using an AG-X250 kN tensile testing machine, and bend tests were performed using an AG-I 500 kN bend testing machine.

2.1 Effect of Nb Content on Weld Metal Microstructure

[Figure 4: see original paper]a shows the macroscopic morphology of the 0Nb weld metal. The grains on both sides of the weld grow diagonally perpendicular to the groove, while grains in the center grow vertically, which is related to heat dissipation during welding as grains grow opposite to the heat dissipation direction. Grains grow epitaxially between weld layers ([Figure 4: see original paper]b). Different grains exhibit anisotropic dendritic growth characteristics, and no obvious differences in macrostructure were observed among weld metals with different Nb contents.

[Figure 5: see original paper] shows the morphology and EDS analysis of intragranular precipitates in weld metals with different Nb contents. As seen in [Figure 5: see original paper]a-d, intragranular precipitates are mainly distributed along interdendritic regions. Combined with [Figure 1: see original paper] and [Figure 2: see original paper], the intragranular precipitates in Nb-free weld metal are Ti(C, N), while those in Nb-bearing weld metals are (Nb, Ti)(C, N). Statistical analysis of intragranular precipitate quantity in weld metals with different Nb contents is shown in [Figure 6: see original paper]. The results indicate that the number of intragranular precipitates increases with increasing Nb content in the weld metal.

During weld pool solidification, TiN precipitates first ([Figure 1: see original paper]). Due to the large mismatch between TiN and the austenite matrix, it cannot serve as a heterogeneous nucleation core for austenite and is rejected to interdendritic regions during solidification [27]. Simultaneously, Ti and Nb segregate to interdendritic regions during solidification [20], making the Ti and Nb contents higher in interdendritic regions than in dendrite cores. In the late solidification stage, TiN transforms to Ti(C, N) in Nb-free weld metal [19,28], while in Nb-bearing weld metals, TiN transforms to (Nb, Ti)(C, N) and NbC also precipitates separately in interdendritic regions [20]. As Nb content in the weld metal increases, the Nb content in interdendritic regions increases, the initial precipitation temperature of NbC increases ([Figure 2: see original paper]), making NbC precipitation easier and TiN transformation to (Nb, Ti)(C, N) more favorable. Therefore, the number of intragranular MX precipitates increases with increasing Nb content in the weld metal.

[Figure 7: see original paper] shows SEM and TEM images of grain boundary

precipitates in weld metals with different Nb contents. Selected area electron diffraction identified the grain boundary precipitates as M23C6 ([Figure 7: see original paper]f). M23C6 is a carbide with a grain boundary precipitation tendency [28] that has a cube-on-cube coherent relationship with one side of the austenite matrix ([Figure 7: see original paper]f) and a lattice parameter approximately three times that of the matrix [29]. As Nb content increases, grain boundary M23C6 precipitates decrease in quantity and transform from multi-row continuous distribution to single-row discrete distribution ([Figure 7: see original paper]a-e).

In NiCrFe-7 weld metal, there are two main types of precipitates: MX precipitates within grains and M23C6 precipitates at grain boundaries. These two types of precipitates compete for C in the matrix. MX precipitates have a higher precipitation temperature than M23C6 precipitates ([Figure 1: see original paper] and [Figure 2: see original paper]), so MX precipitates form first during weld pool solidification, consuming C from the matrix. During heating, M23C6 may precipitate when the temperature is below 1050 °C [30] ([Figure 2: see original paper]). As Nb content in the weld metal increases, more MX precipitates form ([Figure 6: see original paper]), increasing C consumption within grains and reducing the amount of C diffusing to grain boundaries, thereby decreasing grain boundary M23C6 precipitation. Phase diagram calculations ([Figure 2: see original paper]) show that Nb addition can precipitate NbC and lower the initial precipitation temperature of M23C6. M23C6 formation depends on grain boundary C content and formation temperature. Lower precipitation temperature makes C diffusion to grain boundaries more difficult, reducing the driving force for M23C6 precipitation and growth [31] and slowing M23C6 precipitation at grain boundaries, resulting in smaller quantity and size. Adding Nb can fix C and reduce grain boundary M23C6. When Nb content increases from 0 to 0.85%, the initial precipitation temperature of M23C6 gradually decreases to approximately 730 °C ([Figure 2: see original paper]). When Nb content exceeds 0.85%, adding more Nb has no significant effect on further lowering the initial precipitation temperature of M23C6 ([Figure 2: see original paper]), and only small amounts of M23C6 precipitate ([Figure 7: see original paper]d and e).

2.2 Effect of Nb Content on DDC in Weld Metal

[Figure 8: see original paper] shows the morphology of DDC in the 0Nb weld metal. DDC exhibits intergranular characteristics, tends to form on large-angle, long, straight grain boundaries at 45°-90° to the stress direction [14], and has small dimensions that cannot be completely detected by conventional non-destructive testing methods. DDC often becomes a crack source for other cracks [19], posing significant potential hazards for nuclear power products.

Studies [17,21] have shown that grain boundary M23C6 precipitates promote DDC nucleation and propagation, and reducing grain boundary M23C6 content helps decrease DDC formation. As Nb content in the weld metal increases,

M₂₃C₆ transforms from multi-row continuous distribution to single-row discrete distribution. When carbides are continuously distributed at grain boundaries, they promote intergranular crack nucleation and propagation [14,32], facilitating DDC formation. Therefore, Nb addition can precipitate MX phases within grains to fix C, reducing grain boundary M₂₃C₆ precipitation and DDC formation. Additionally, MX precipitates can hinder grain boundary sliding at high temperatures, which also helps reduce DDC [13,14,16,33,34]. Statistical results show that both the quantity and size of DDC in the weld metal decrease with increasing Nb content ([Figure 9: see original paper]). When Nb content exceeds 0.85%, excellent control of DDC crack formation can be achieved.

2.3 Effect of Nb Content on Mechanical Properties of Weld Metal

[Figure 10: see original paper] shows the room-temperature tensile curves for weld metals with different Nb contents. The results demonstrate that both strength and ductility increase with increasing Nb content. The strengths of 0Nb, 0.4Nb, 0.7Nb, 0.85Nb, and 1.1Nb weld metals are 520, 559, 594, 615, and 653 MPa, respectively, while the ductility values are 19.5%, 24.8%, 32.0%, 32.5%, and 35.2%, respectively.

The tensile fracture surfaces exhibit three characteristic regions, as shown in [Figure 11: see original paper]a. The first is a rough flat area without fracture features ([Figure 11: see original paper]a1), caused by DDC with intergranular characteristics [20]. The second is cracks without fracture features around them ([Figure 11: see original paper]a2), also caused by intergranular DDC [20]. The third is dimpled transgranular fracture ([Figure 11: see original paper]a3). DDC is the main factor affecting weld metal mechanical properties. As Nb content increases, DDC decreases in the weld metal, the area of featureless fracture surfaces decreases, and the area of dimpled fracture increases. The area around cracks also transforms from featureless surfaces to shallow, wide dimpled fracture. With increasing Nb content, DDC decreases in the weld metal, and both strength and ductility increase.

[Figure 12: see original paper] shows the surface morphologies of bend test specimens from weld metals with different Nb contents. The 0Nb weld metal bend specimen surface produced numerous cracks. As Nb content increased, surface cracks became fewer. Based on tensile property analysis, the surface cracks in bend specimens are also caused by DDC in the weld metal. As Nb content increases, both the quantity and size of DDC decrease in the weld metal, resulting in fewer surface cracks and improved bending performance.

Conclusions

1. Intragranular precipitates are mainly distributed along interdendritic regions. In Nb-free weld metal, intragranular precipitates are Ti(C, N),

while in Nb-bearing weld metals they are (Nb, Ti)(C, N). The number of intragranular precipitates increases with increasing Nb content.

2. M23C6 precipitates along grain boundaries. Nb addition lowers the initial precipitation temperature of M23C6, slows M23C6 precipitation, and reduces its quantity and size. As Nb content increases, M23C6 transforms from multi-row continuous distribution to single-row discrete distribution.
3. Both the quantity and size of DDC in the weld metal decrease with increasing Nb content. When Nb content exceeds 0.85%, excellent control of DDC crack formation can be achieved.
4. As Nb content increases, the area of featureless fracture surfaces on weld metal tensile fracture surfaces decreases while the area of dimpled fracture increases. The area around cracks also transforms from featureless surfaces to shallow, wide dimpled fracture. Both strength and ductility of the weld metal increase. The 0Nb weld metal bend specimen surface produced numerous cracks, which decreased with increasing Nb content.

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