Effect of Nb and Mo on the Microstructure, Mechanical Properties and Ductility-Dip Cracking of Ni–Cr–Fe Weld Metals

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Abstract A series of Ni–Cr–Fe welding wires with different Nb and Mo contents were designed to investigate the effect of Nb and Mo on the microstructure, mechanical properties and the ductility-dip cracking susceptibility of the weld metals by optical microscopy (OM), scanning electron microscopy, X-ray diffraction as well as the tensile and impact tests. Results showed that large Laves phases formed and distributed along the interdendritic regions with high Nb or Mo addition. The Cr-carbide ($M_{23}C_6$) was suppressed to precipitate at the grain boundaries with high Nb addition. Tensile testing indicates that the ultimate strength of weld metals increases with Nb or Mo addition. However, the voids formed easily around the large Laves phases in the interdendritic area during tensile testing for the weld metal with high Mo content. It is found that the tensile fractographs of high Mo weld metals show a typical feature of interdendritic fracture. The high Nb or Mo addition, which leads to the formation of large Laves phases, exposes a great weakening effect on the impact toughness of weld metals. In addition, the ductility-dip cracking was not found by OM in the selected cross sections of weld metals with different Nb additions. High Nb addition can eliminate the ductility-dip cracking from the Ni–Cr–Fe weld metals effectively.

KEY WORDS: Nb; Mo; Laves phase; Microstructure; Mechanical properties

1 Introduction

Currently, the filler metal 52M (ERNiCrFe-7A) has been widely applied to the manufacture of key components in the energy industry. This Ni-based alloy has a good

performance in resisting intergranular stress corrosion cracking (IGSCC) and primary water stress corrosion cracking (PWSCC) because of the high chromium content [1]. As a filler metal, alloy 52M is mainly used for welding Inconel 600 and Inconel 690 nowadays [2]. The alloy is also used to weld dissimilar materials with several stainless steels, low alloy steels and Ni-based alloy [3, 4]. For the development and application of this Ni–Cr–Fe filler metal, it is necessary to pay attention to the ductility-dip cracking (DDC) of 52M, which is often formed in the welding [5–7].

Ductility-dip cracking is a solid-state cracking phenomenon, which often occurs along grain boundary in austenitic alloys, superalloys or copper alloys at elevated temperature [7]. Due to the small size of the crack, DDC is usually hard to be detected by the conventional nondestructive examination [8]. A number of factors have been reported to contribute to the DDC initiation and propagation, such as welding processes, the grain boundary

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orientation relative to the applied strain, weld metal chemical composition (interstitial element content and impure element segregation in the grain boundaries), grain boundaries sliding and dynamic recrystallization [9–15]. Due to the close relationship between chemical composition and the microstructure, effect of chemical composition on DDC susceptibility of weld metals has been widely investigated by researchers [6, 16–18]. Nb and Mo are the alloying elements commonly chosen to improve austenitic weldment quality. According to the studies of Lippold *et al.* [7, 19], alloy 52M provides higher DDC resistance than the alloy 52 (ERNiCrFe-7) because of the addition of Nb. Further, the alloy 52MSS (ERNiCrFe-13), a new nickel-based alloy material with 30 wt% Cr, has been reported an outstanding DDC resistance with adding about 2.5 wt% Nb and 4.0 wt% Mo [20–22].

In the previous researches [18, 23], the effect of Ti and Nb on the DDC resistance of weld metals has been investigated. The results indicate that Ti or Nb addition could greatly improve the DDC resistance of Ni–Cr–Fe weld metals and reduce the number of DDC. However, there is still a little of DDC existing in Ni–Cr–Fe alloy with low Ti (0.3–0.9 wt%) or Nb (0–1.1 wt%) addition. In order to completely eliminate the DDC from the Ni–Cr–Fe welds, the chemical composition of weld metals need to be optimized further. Nb has a high carbon affinity, which promotes the precipitation of Nb-rich carbide and reduces the precipitation of Cr-carbide at the grain boundaries. Ramirez and Lippold [11, 16] suggested that the eutectic-like precipitates formed at the end of solidification can pin the grain boundaries to form tortuous boundaries, which make grain boundaries slide more difficult and thus improve the DDC resistance of weldments. On the other hand, the intergranular precipitate Cr-carbide formed during the welding can result in the decrease of the DDC resistance of weld metals [8]. In addition, Mo is an alloying element that can improve the materials' plastic deformation resistance at high temperature [24]. According to the previous analysis, high Nb and Mo addition might eliminate DDC effectively. However, high Nb or Mo addition may cause Nb-rich or Mo-rich precipitates, which may have other influences on the mechanical properties of weld metals. Thus, the object of this study is to investigate effect of Nb and Mo on the microstructure and mechanical properties of alloy 52M. The work will be helpful to the development of the modified 52M.

2 Materials and Methods

Six $\Phi 1.2$ mm Ni-based welding wires with different Nb and Mo contents have been fabricated in this investigation. Weld metal fabrication experiments were carried out on

carbon steel Q235 with approximate dimension of 500 mm $\times$ 125 mm $\times$ 25 mm. A butt joint with a single V groove at 22.5° on both sides was generated. The buttering with filler metal 52 was applied to the three carbon steel sides to depress the influence of element migration, especially C, Ni, Cr and Fe, as shown in Fig. 1a. After buttering, butt welding was performed by cold-wire feed multiple semiautomatic gas tungsten arc welding (GTAW) with six kinds of the welding wires. Argon was supplied as the shielding gas in the process of welding. The welding parameters are given in Table 1. To evaluate the DDC sensitivity of each filler metal and avoid distortion, the plates were all welded under strong restraint condition, as shown in Fig. 2. Table 2 shows the chemical compositions of six weld metals.

Six as-welded samples were prepared for microstructure observations after a common mechanical grinding and polishing process, followed by electrolytic etching using a reagent of 10 g Cr_2O_3 + 100 ml H_2O under a potential 10 V DC for 20–30 s. The microstructures were observed using Zeiss optical metallurgical microscopy and JSM-6301F field emission scanning electron microscopy. X-ray energy dispersive spectroscopy (XEDS) was applied to analyze the composition. The second phases of each weld metal were extracted using a 7 ml HCl + 93 ml methanol reagent and the application of a potential of 1.5 V DC. X-ray diffraction (XRD) was used to phase analysis of second phases.

Fig. 1 Schematic diagrams of the weld joint and test specimen: **a** sampling location, **b** impact specimen, **c** tensile specimen

Table 1 Welding parameters

Current (A)	Voltage (V)	Wire feed speed (mm/min)	Travel speed (mm/min)	Gas flow rate (L/min)	Polarity	Duty cycle
160–240	10.5–11.0	900–1200	85–110	20	DC/SP	50%

Fig. 2 Welding plates under strong restraint conditions

Tensile tests and impact tests were applied to evaluate the mechanical properties of deposited metals. The tensile specimens, impact specimens and the sampling locations are shown in Fig. 1. Tensile testing was performed on an MTS E45.105 universal tensile testing machine at room temperature according to the standard GB/T228.1-2010, and INSTRON 5582 universal tensile testing machine was used to measure the tensile properties at 350 °C, which follows the standard GB/T 4338-2006. Impact tests were carried out at room temperature following the standard GB/T 229-2007. In the case of data discretization, three tensile specimens and three impact specimens were fabricated from each weld metal for mechanical properties measurement.

Table 2 Chemical compositions of the weld metals (wt%)

Alloy	Nb	Mo	C	Ni	Cr	Fe	Si	Al	Ti	Mn	S	P
WM-A(2.5Nb/0Mo)	2.38	<0.005	0.023	Bal.	29.98	9.06	0.12	0.20	0.35	0.76	0.0029	0.002
WM-B(2Mo)	2.39	1.9	0.019	Bal.	29.84	9.14	0.12	0.18	0.29	0.79	0.0026	0.002
WM-C(4Mo)	2.37	4.09	0.022	Bal.	29.65	8.99	0.12	0.19	0.31	0.77	0.0031	0.003
WM-D(6Mo)	2.35	6.08	0.024	Bal.	29.80	9.18	0.13	0.19	0.31	0.80	0.0034	0.002
WM-E(1.8Nb)	1.72	<0.005	0.022	Bal.	29.90	9.77	0.12	0.16	0.31	0.77	0.0028	0.002
WM-F(4Nb)	3.82	<0.005	0.024	Bal.	29.78	9.12	0.12	0.16	0.31	0.75	0.0045	0.002

3 Results and Discussion

3.1 Microstructure

Six weld metals are labeled as WM-A(2.5Nb/0Mo), WM-B(2Mo), WM-C(4Mo), WM-D(6Mo), WM-E(1.8Nb) and WM-F(4Nb). Figure 3 shows the optical micrographs of the six weld metals. Similar microstructure characteristics are observed in the weld metals containing different Nb and Mo contents. It is noted that columnar dendrites are the dominant features in the microstructure of all weld metals. The remarkable difference in the six weld metals is that the grain boundary becomes un conspicuous under the same electronic etching condition when the content of Mo or Nb is over 4 wt%, as shown in Fig. 3c, d, f. On the contrary, the interdendritic becomes more and more obvious with Nb or Mo addition. Jeng *et al.* [25] claimed that high Nb content reduces the intergranular corrosion resistance of weldments by promoting Nb-rich phase formation. With the increase in Nb or Mo content, the segregation amount of Nb or Mo in interdendritic region increases gradually. Nb-rich phases or Mo-rich phases could precipitate in the interdendritic. Finally, it is harder to etch grain boundaries and the morphology of dendrite is easier to be revealed.

3.1.1 Effect of Nb on Precipitates

According to the XRD results, MC (*M* represents Nb, Ti) and Laves phase are present in the Nb-bearing weld metals, as shown in Fig. 4. With the increase in the Nb content, Laves phases appear in the high Nb weld metal. This phenomenon suggests that high Nb content promotes the formation of Laves phase during the welding of alloy 52M. Figure 5 shows the specific morphology characteristic of the precipitates within several Nb-bearing weld metals. The

Fig. 3 Optical micrographs of six weld metals: **a** WM-A(0Mo), **b** WM-B(2Mo), **c** WM-C(4Mo), **d** WM-D(6Mo), **e** WM-E(1.8Nb), **f** WM-F(4Nb)

precipitates mainly distribute at the grain boundaries and interdendritic spaces. Table 3 lists the composition of certain precipitates in the weld metals determined by XEDS. (Nb, Ti)C is the common second phase in three Nb-bearing weld metals. Unlike XRD results, which do not show any diffraction peak of $M_{23}C_6$, some fine Cr-carbide ($M_{23}C_6$) precipitates are noted at the grain boundaries in the WM-E(1.8Nb). In the previous study, Cr-carbide ($M_{23}C_6$) has also been found at grain boundaries in the low Nb weld metals [23, 26]. The dissolution of $M_{23}C_6$ in the process of precipitates extracting may result in such inconsistency. Little amount of $M_{23}C_6$ is hard to be extracted from the weld metals using electrolysis method. Finally, XRD analysis is unable to catch any trace of $M_{23}C_6$. With Nb addition, $M_{23}C_6$ is prevented to precipitate at the grain boundaries, and therefore the grain boundaries will become clean. This result indicates that Nb addition

effectively reduces the amount of $M_{23}C_6$. The research work of Jeng and Chang [26] showed the same phenomenon in the high Nb weldments. Compared with $M_{23}C_6$, MC generally precipitates in the matrix at higher temperature range during solidification. The supersaturation C will be consumed by Nb at high temperature and then little amount of C can be combined with Cr at relative low temperature to form Cr-carbide ($M_{23}C_6$) precipitating at the grain boundaries. Hence, with high Nb addition, Cr-carbide ($M_{23}C_6$) is suppressed and disappears from the weld metals. As shown in Fig. 5c, the abundant of Laves phases, with large sizes, forms in the interdendritic regions in the weld metal as the Nb content is up to 4 wt%. It is obvious that the morphology of Laves phase is irregular. XEDS microchemical analysis indicates that Laves phase contains more Ni and less Nb compared with that of MC, as shown in Table 3b₁, c₁.

Fig. 4 XRD pattern of the precipitates electrochemically extracted from the Nb-bearing weld metals: **a** WM-E(1.8Nb), **b** WM-A(2.5Nb), **c** WM-F(4Nb)

3.1.2 Effect of Mo on Precipitates

XRD was applied to perform phase analysis of the precipitates extracted from Mo-bearing weld metals. The results show that there are MC and Laves phase precipitating in the weld metals, as shown in Fig. 6. In addition, when Mo content is over 4 wt%, Laves phase forms in the matrix. The varieties of precipitates in four weld metals with Mo addition are shown in the Fig. 7. Table 4 lists the composition of certain precipitates distributing in the interdendritic space of the weld metals. Laves phase, which contains high amount of Nb and Mo contrasting to the matrix content, can be found in the weld metals when the content of Mo increases to 4%–6%, as shown in Fig. 7c, d. This phenomenon is consistent with the XRD results. The Laves phase has a large particle size; e.g., the sizes of Laves phases in the WM-D(6Mo) (Fig. 7d) are about 5 μm . The XEDS results (Tables 3c₁, 4c₂, d₁) show that the amount of Nb in the Laves phase tends to decrease with the increase in the content of Mo, which implies that Mo can replace certain Nb atoms during the Laves phase formation.

In the present study, the Laves phase with large size appears in the interdendritic of high Nb or high Mo weld metals. According to the work of DuPont *et al.* [27], Nb tends to promote the precipitation of two types of Nb-rich second phase in the process of the Ni-based alloy solidification. A higher C content accelerates the formation of NbC, while Laves phase forms readily with a high Nb and a low C composition in the austenitic alloy [28]. In addition, the interdendritic segregation of Nb also plays a key role in the formation of Laves phase. With high Nb adding to the

Fig. 5 Precipitates in the weld metals: **a** WM-E(1.8Nb), **b** WM-A(2.5Nb), **c** WM-F(4Nb). The compositions of a₁, b₁, c₁ are presented in Table 3

Table 3 Compositions of certain precipitates in the weld metals in Fig. 5 (wt%)

Label	Ni	Cr	Fe	Nb	Mo	Ti
a						
a ₁	54.47	34.01	9.07	2.36	–	–
b						
b ₁	29.54	18.98	4.63	43.25	–	3.59
c						
c ₁	48.78	17.77	4.79	28.67	–	–

Fig. 6 XRD patterns of the precipitates electrochemically extracted from the weld metals: **a** WM-A(0Mo), **b** WM-B(2Mo), **c** WM-C(4Mo), **d** WM-D(6Mo)

Table 4 Compositions of the precipitates in the weld metals in Fig. 7 (wt%)

Label	Ni	Cr	Fe	Nb	Mo	Ti
a						
a ₁	29.54	18.98	4.63	43.25	–	3.59
b						
b ₁	41.19	18.18	4.64	25.66	5.11	–
b ₂	8.08	6.59	1.39	58.34	1.14	14.59
c						
c ₁	14.77	13.42	2.83	34.05	2.72	26.86
c ₂	38.68	22.85	5.76	19.96	8.93	–
d						
d ₁	42.20	26.21	7.72	13.83	10.03	–

Ni-based alloy, the amount of Nb in the interdendritic space becomes higher and the precipitation of Laves phase is promoted dramatically. Mo is able to reduce the Nb content that is required to form Laves phase in the Ni–Cr–Fe alloy and promotes the Laves phase formation [29]. Even though the Nb content is not high enough, Laves phases still form in the weld metals with high Mo addition.

Fig. 7 Intermetallic precipitates in the Mo-bearing weld metals: **a** WM-A(0Mo), **b** WM-B(2Mo), **c** WM-C(4Mo), **d** WM-D(6Mo). The compositions of a₁, b₁, b₂, c₁, c₂ and d₁ are presented in Table 4

Fig. 8 Average ultimate strength of the Nb-bearing weld metals at room temperature and 350 °C

Fig. 9 SEM fractographs of the weld metals under tension at room temperature: **a** WM-E(1.8Nb), **b** WM-A(2.5Nb), **c** WM-F(4Nb)

Fig. 10 Micrographs of longitudinal section of the tensile rupture for the weld metals under tension at room temperature: **a** WM-E(1.8Nb), **b** WM-A(2.5Nb), **c** WM-F(4Nb)

Fig. 11 Average impact energy of Nb-bearing weld metals

Fig. 12 Average ultimate strength of Mo-bearing weld metals

Herein, the high Nb or Mo addition promotes the Laves phase formation in the austenitic weld metals.

3.2 Mechanical Properties

3.2.1 Effect of Nb on Tensile Strength and Impact Energy

Figure 8 illustrates the average ultimate strength, σ_m of the weld metals with varying concentration of Nb. The ultimate strength of weld metal gradually increases as the level of Nb rises at room temperature and 350 °C. All specimens rupture with a mode of ductile fracture. Figure 9 shows the fractographs of the tensile samples tested at room

temperature. The dimples are observed on the fracture surface and change from deep to shallow with increasing the Nb content. This phenomenon indicates that Nb addition improves the deformation resistance of the weld metals. However, the longitudinal sections of the tensile fracture present that many voids, which can be found in Fig. 10 easily, forming around the precipitates in the WM-F(4Nb) contains large precipitates. The result implies that large precipitates tend to make the samples rupture easily.

The impact energy of weld metal shows a negative correlation with the element Nb, which is given in Fig. 11. It is noted that Nb addition reduces the toughness of Ni–Cr–Fe weld metals. Furthermore, the impact toughness decreases rapidly as the Nb content is over 2.5 wt%. The large Laves phase, which appears in the interdendritic of the high Nb weld metal, might expose an apparent weakening effect on the toughness of weld metal.

3.2.2 Effect of Mo on the Tensile Strength and Impact Energy of the Weld Metals

The average ultimate strength of the as-welded weld metals were measured at room temperature and 350 °C. The results are plotted in a broken line graph in Fig. 12. The tensile strength increases gradually both at room temperature and 350 °C with the increase in Mo content. Due to the softening of austenite matrix, the ultimate strength at 350 °C decreased about 130 MPa compared with that at room temperature.

Fig. 13 SEM fractographs of the weld metals under tension at room temperature: **a** WM-A(0Mo), **b** WM-B(2Mo), **c** WM-C(4Mo), **d** WM-D(6Mo)

Fig. 14 Micrographs of longitudinal section of the tensile rupture for the weld metals under tension at room temperature: **a** WM-A(0Mo), **b** WM-B(2Mo), **c** WM-C(4Mo), **d** WM-D(6Mo)

Figure 13 shows the fractographs of the tensile specimens containing different Mo contents. Dimples are observed on the fractographs of weld metals. The fractography of the weld metals WM-C and D indicates that high Mo addition encourages interdendritic fracture in the weld metals. The longitudinal section of the tensile fracture, as shown in Fig. 14, shows that many voids formed around the large precipitates in the interdendritic area.

With Nb or Mo addition, the solution of Nb or Mo and precipitates enhance the tensile strength of weld metals. However, high Nb or Mo addition leads to large Laves phase, which can cause the stress concentration around precipitates under the tensile loading. Leng and Chang [26] reported that there were less slip systems to active with the solution of Mo increase and the deformation mainly occurred around the interface between the precipitates and the matrix material. The voids may occur around the large Laves phase easily during deformation, as shown in Figs. 10c and 14d. These voids gradually connect with each other and finally result in rupture. The interdendritic spaces, where large second phases distribute will become a weak zone and then the rupture shows an interdendritic fracture feature. Therefore, large Laves phase might reduce the strength of weld metals. The investigation of Lee *et al.* [28] also shows that large precipitates tend to have a weakening effect on the strength of welds and make the welds more fragile in the Nb-bearing austenitic weldment. However, the strengths of the weld metals with high Nb or Mo are increased in this study. This might result from the solid solution strengthening of Nb or Mo. With the increase

in Nb or Mo concentration, the solid solution strengthening might be the main contributor to the increase of strength.

Figure 15 shows the average impact energy of the weld metals. It is obvious that the impact energy decline rapidly with Mo addition. Further, high Mo content has a remarkably weakening effect on the impact toughness of weld metals.

Figures 11 and 15 show that high Nb or Mo addition results in a great decrease in the impact toughness of weld metals, which implies that large Laves phase tends to reduce weld metal's ability to absorb energy under impact loading. The Laves phases fragment during the impact testing to form many voids, which lead to the samples to

Fig. 15 Average impact energy of each as-welded deposited metal

Fig. 16 SEM micrographs of $M_{23}C_6$ distributing along the grain boundary **a**₁, **b**₁, **c**₁, **d**₁ and optical micrographs of longitudinal section of tensile rupture **a**₂, **b**₂, **c**₂, **d**₂; **a**₁, **a**₂ weld metal with 0 wt% Nb from the published article [23, 30], **b**₁, **b**₂ WM-E(1.8Nb), **c**₁, **c**₂ WM-A(2.5Nb), **d**₁, **d**₂ WM-F(4Nb)

rupture easily. It is obvious that large Laves phases result in a great weakening effect on the impact toughness of weld metals.

3.3 Effect of Nb on the Ductility-Dip Cracking

DDC is commonly viewed as a defect in ERNiCrFe-7A weldments and is too small to be found using conventional nondestructive evaluation methods. It is necessary to find an effective way to alleviate or even eliminate the DDC in Ni–Cr–Fe welds for the safety of nuclear power plant. In the previous investigation [8], the effect of $M_{23}C_6$ on the DDC has been investigated. Results suggested that $M_{23}C_6$ can induce the initiation and propagation of DDC in the weldments. $M_{23}C_6$ has a cube-on-cube coherent relationship with one side grain at the grain boundaries. The incoherent interface between $M_{23}C_6$ and γ is prone to crack initiation, which exacerbates DDC initiation in Ni–Cr–Fe alloy at intermediate temperatures. In this study, DDC is not observed on the weld metal with different content of Nb ($Nb \geq 1.8\%$) by using optical microscope. It can be seen that high Nb addition can eliminate DDC from the alloy 52M effectively. By contrasting to the previous investigation [23, 30], it is easy to find that the amount of Cr-carbide ($M_{23}C_6$) distributing along the grain boundaries decreases with the rise of the level of Nb, as shown in Fig. 16a₁, b₁, c₁, d₁. Furthermore, Cr-carbide ($M_{23}C_6$) is hard to be found when the Nb content is more than 2.5 wt%, which is shown in Fig. 16c₁, d₁. Element Nb has a fantastic affinity to C and forms the precipitate of NbC. Due to the consumption of the supersaturation C, the amounts of Cr-carbide decreased in the welding process. With further increasing the content of Nb, the amount of $M_{23}C_6$ is controlled to a very low level and DDC is also eliminated from the Ni–Cr–Fe weld metals. Meanwhile, Nb-rich precipitates forming with Nb addition also enhance the DDC resistance of weld metals [14]. On the other hand, DDC can promote intergranular fracture in the tensile process, like Fig. 16a₂. The longitudinal sections of the tensile rupture of high Nb weld metals do not show any distinct characteristic of intergranular fracture induced by DDC, as shown in Fig. 16b₂, c₂, d₂. Results imply that the DDC resistance of the weld metals becomes better as Nb content increases.

4 Conclusions

1. Columnar dendrites are dominant features of the microstructure in Ni–Cr–Fe weld metals. With the

increase in Nb and Mo contents, the size and quantity of precipitates become bigger and higher. Fine Cr-carbide ($M_{23}C_6$) prevents to precipitate along the grain boundaries with high Nb addition. However, high Nb or Mo addition results in the large Laves phases distributing at the interdendritic regions in the weld metals.

2. Due to the strengthening of solution of Nb or Mo and the precipitation, the ultimate strengths of the weld metals increase gradually with the Nb or Mo addition. However, large Laves phase precipitates tend to result in voids in the weld metals and may make weld metals more fragile in the process of deformation. The strengthening of solution of Nb or Mo may be the main contributor to the increase in strengths of high Nb or Mo weld metals. The impact energy decreases rapidly with the increase in Nb and Mo content. The large Laves phase exposes a dramatic weakening effect on the impact energy of weld metals.
3. No obvious DDC can be found in the weld metals with the Nb content more than 1.8 wt%. Cr-carbide ($M_{23}C_6$) is reduced to a very low level by addition of Nb element. High Nb addition in Ni–Cr–Fe alloy can restrain the appearance of DDC effectively.

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