Effect of CeO₂ on microstructures and mechanical properties of welded high-strength steel weld metal

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Abstract

The effect of CeO₂ on the microstructures and mechanical properties of welded high-strength steel weld metal was investigated by optical microscopy, scanning electron microscopy and mechanical testing. The results demonstrate that the addition of CeO₂ can promote the refinement and spheroidization of inclusions, refine the grains, and form acicular ferrites in the weld metals. When the addition of CeO₂ increased from 0% to 3%, the content of bainites gradually decreased, and the lath structure disappeared. The formation of the acicular ferrite ductile phase inhibited the formation of bainites and other strengthened phases in the weld metals, and the microstructure of acicular ferrites was excessive. When the CeO₂ content was 1%, the tensile strength was 903 MPa, the yield strength was 848 MPa, and the low-temperature impact toughness was 61 J at −40 °C. When the CeO₂ content was 3%, the low-temperature impact toughness of the weld metal gradually increased to a maximum value of 71 J. The weld metal had the highest toughness but the lowest strength. The addition of CeO₂ changed the pattern of crack generation, and the fracture mode changed from quasi-cleavage fractures to ductile fractures with dimples. To achieve a good matching of strength and toughness, the wire No. 2 with 1% CeO₂ had the best comprehensive mechanical properties.

1. Introduction

With the progress of science and technology and the development of industrial production, high-strength steels have gradually replaced low-alloy steel and are widely used in various engineering fields, such as aerospace, offshore platforms, and oil pipelines. However, the wide application of high-strength steels is inseparable from the welding technology, so the development of high-strength steel welding consumables and the study of the effects of alloying elements on the microstructure and mechanical properties of weld metals and heat-affected zones are hotspots in the current welding field [1]. Zhang et al [2] studied the effect of acicular ferrite (AF) on the mechanical properties of weld metals. Inclusions can act as inert nucleation surfaces and reduce the activation energy to promote AF nucleation. Mao et al [3] studied the effect of Ni on the microstructure of the weld metals and pointed out that Ni could promote the transformation of the microstructure from granular bainite (GB) to lath bainite and lath martensite, and appropriate Ni could improve the impact toughness. Keehan et al [4] found coalesced bainite in high-strength steel weld metals and noted that coalesced bainite was a coarse and brittle phase, and its presence deteriorated the toughness of the weld metal.

Many scholars [5, 6] have studied the microstructure transformation of the weld metals and mainly focused on C, Mn, Cr, Ni, etc. Regarding the role of rare earth elements in metal materials, the research direction focuses on steel modification. At present, there have been few, if any, studies on the effects of rare earth elements on the strength and toughness of welded high-strength steel weld metal. Therefore, rare earth oxide is the research object in this paper, and we conduct a systematic study on the effect of CeO₂ on the microstructures and mechanical properties of welded high-strength steel weld metal.
2. Experimental procedure

To study the effect of CeO$_2$ on the microstructures and mechanical properties of welded high-strength steel weld metal, three different contents of CeO$_2$ were added to the metal-powder cored wire: wire No. 2, wire No. 3 and wire No. 4 were added to 1%, 2%, and 3% CeO$_2$, respectively. The wire 1 (CeO$_2$ 0%) was the control group. The diameter of the wires was 1.2 mm, and the base material was made of Q235 carbon steel. Weld metals were prepared according to the AWS standard A5.29/A5.29 M [7]. An 80Ar + 20CO$_2$ shielding gas was adopted because the composition is widely used in practical engineering applications. The pass arrangement and welding parameters are shown in figure 1(a) and table 1, respectively. The specimens were prepared according to AWS standards A5.29/A5.29 M [7] and B4.0 [8]. The groove weld test assembly for mechanical properties is schematically shown in figure 1(a). The round tensile specimens and Charpy V-notch impact specimens are shown in figures 1(b) and (c). After preparing the standard specimens, the mechanical properties of the weld metals were tested with a WAW-6000 tensile testing machine, and the results of the yield strength, tensile strength, shrinkage and elongation were recorded. The Charpy impact test was performed by an JB30B impact testing machine after the impact specimens were cooled to $-40^{\circ}$C. Five impact values were recorded, and the average value was taken after removing the maximal and minimal values. The chemical compositions of the weld metals were determined with a Q4 optical emission spectrometer. Metallographic specimens were prepared and etched with 4% natal. The microstructures of the weld metals were observed using optical microscopy (OM), and the proportion of the microstructure was calculated. The Vickers hardness of the weld metal was measured using a HXD-1000FMC/LCD Vickers hardness tester. The fracture surfaces were examined by a TESCAN VEGA3 scanning electron microscope (SEM), and the inclusions in the dimples were analyzed by energy dispersive spectrometry (EDS).

Table 1. Welding parameters used in experiments.

| Voltage(V) | Current (A) | Wire stick-out (mm) | Welding speed (cm·min$^{-1}$) | Shielding gas (%) | Heat input (kJ·mm$^{-1}$) | Preheat/interpass temperature (°C) |
|-----------|------------|---------------------|-----------------------------|-----------------|-----------------|-------------------------------|
| 30        | 230        | 17                  | 25                          | 80Ar+20CO$_2$   | 1.80                 | 150                           |

Figure 1. Groove weld test assembly for mechanical properties: (a) test plate showing location of test specimens; (b) dimensions of round tensile specimen; (c) dimensions of charpy V-notch impact specimen.
3. Results and discussion

3.1. Microstructure and toughness of weld metals

The chemical compositions and mechanical properties of the weld metals are shown in Table 2 and Figure 2, respectively. Figure 2 illustrates the yield strength and tensile strength curves of the weld metals with the addition of various CeO₂ contents. With the addition of 1% CeO₂, the tensile strength of the weld metal increased from the initial value of 843 MPa to 903 MPa, and the yield strength increased from 793 MPa to 848 MPa. When the content of CeO₂ further increased, the strength of the weld metals gradually decreased. With the addition of 3% CeO₂, the weld metal obtained the lowest strength with a tensile strength of 808 MPa and a yield strength of 742 MPa. The low-temperature impact toughness of the weld metal is shown in Figure 2. With the content of CeO₂ increased, the low-temperature impact toughness of the weld metals obviously increased. The impact value of wire No. 1 without CeO₂ was the lowest, which was 45 J. The impact value of wire No. 4 with 3% CeO₂ was the highest, which was 71 J.

The elongation, shrinkage and Vickers hardness of the weld metals are shown in Figures 3 and 4, respectively. Figure 3 illustrates the elongation and shrinkage curves of the weld metals with the addition of various CeO₂ contents. With the addition of CeO₂, the weld metals had basically identical trends of elongation and shrinkage. Wire No. 3 with 2% CeO₂ had the lowest elongation and shrinkage, which were 9% and 8%, respectively. Wire No. 4 with 3% CeO₂ had the highest elongation and shrinkage, which was 19.6% and 29%, respectively. Figure 4 shows a scatter plot of the Vickers hardness of the weld metals with different CeO₂ contents. Wire No. 3 with 2% CeO₂ had the highest Vickers hardness, which was 279 Hv. Wire 4 with 3% CeO₂ had the lowest Vickers hardness, which was 241 Hv. Without CeO₂ and with 3% CeO₂, the Vickers hardness value of the weld metal had a large dispersion, which indicates that the microstructure of the weld metal was not uniform. With the addition of 1% CeO₂, the dispersion of the weld metal Vickers hardness value was the smallest, which indicates that the weld metal microstructure was uniform.

The microstructure quantitative statistical analysis and microstructure of the weld metals with different CeO₂ contents are shown in Table 3 and Figure 5. The addition of CeO₂ promoted the nucleation and growth of AF, and the microstructure in the weld metal was gradually refined. Without CeO₂, the lath microstructure in the weld metals was relatively coarser, as shown in figures 5(a) and (e). With the addition of 1% CeO₂, the weld metal was composed of martensite (M), B (bainite) and AF, the block microstructure and lath microstructure were reduced, and the microstructure was obviously refined, as shown in figures 5(b) and (f).
addition of CeO₂, the nucleation of M and degenerate upper bainite (DUB) decreased, and the AF content increased and became refined, as shown in figures 5(c) and (g). With the addition of 3% CeO₂, the proportion of AF was the largest, and the M and DUB basically disappeared, as shown in figures 5(d) and (h).

The addition of CeO₂ can refine the grains and promote the formation of AF in the weld metals. Fine grains have a larger grain boundary area, which helps hinder the dislocation movement. The dislocation movement

| Wire no. | Acicular ferrite | Martensite | Granular bainite | Degenerate upper bainite |
|----------|-----------------|------------|------------------|--------------------------|
| 1        | 32              | 15         | 28               | 25                       |
| 2        | 60              | 8          | 20               | 12                       |
| 3        | 76              | 4          | 12               | 8                        |
| 4        | 88              | 2          | 6                | 4                        |

Figure 3. Effect of CeO₂ on the elongation and shrinkage of weld metals.

Figure 4. Effect of CeO₂ on the Vickers hardness of weld metals.
Figure 5. Micrographs of weld metals: (a) and (e) wire Ce 0%; (b) and (f) wire Ce 1%; (c) and (g) wire Ce 2%; (d) and (h) wire Ce 3%. (AF acicular ferrite, DUB degenerate upper bainite, M martensite, GB granular bainite).
cannot easily cross the grain boundaries and accumulate at the grain boundaries, which results in a higher strength of the weld metals [9, 10]. In addition, with a more tortuous grain boundary, more energy is required to extend the crack, which is not conducive to the crack extension and can improve the impact toughness of the material. AF is considered an excellent microstructure component to improve the toughness through effective grain refinement, and AF grains can divide large austenite grains into fine individual regions to form a mixed microstructure of fine particles [11]. The AF microstructure has a large-angle grain boundary, which can effectively increase the crack extension path during fracture. The increase in energy required for crack extension helps improve the low-temperature impact toughness of the weld metals, which can explain the higher strength and impact energy of wire 2 than those of wire 1. The grain refinement degree of the weld metal decreases with the addition of excessive CeO$_2$, the addition of CeO$_2$ can be used as the core of AF heterogeneous nucleation, the grain core preferentially attaches to the surface of these impurities and promotes the formation of a large amount of AF, and the formation of a large AF content inhibits the formation of M, B and other phases. M and DUB are the main strengthening phases, which can improve the strength and hardness of the weld metal. AF and GB are toughening phases in the weld metal that mainly improve the low-temperature impact toughness of the weld metal [12]. The excessive formation of AF causes a decrease in M and DUB strengthening phases in the weld metal, so further addition of CeO$_2$ can cause a decrease in strength and an increase in toughness. This phenomenon was reflected in the micrographs of wire 2, wire 3 and wire 4.

### 3.2. Fracture morphology of weld metals

The fractures of different Charpy V specimens at $-40^\circ$C are shown in figure 6. With the addition of CeO$_2$, the fracture mode changed from quasi-cleavage fracture to ductile fracture with dimples, the tear ridges were scattered between the cleavage fracture and the dimple, the width of the dimples decreased, the depth of the dimples increased, and the distribution of the dimples was even. Dimples could improve the low-temperature impact toughness of the weld metals, which is consistent with the low-temperature impact curve in figure 2.

Without CeO$_2$, the dimples had the largest width and smallest depth, and the distribution of dimples was extremely uneven and concentrated in a few areas, which shows that the energy absorbed was low and the toughness was poor, as shown in figure 6(a). With the increase in CeO$_2$, the dimple width in the fracture of the specimen decreased, the depth increased, and the distribution was more uniform, as shown in figures 6(b) and (c). With the addition of 3% CeO$_2$, the number of dimples in the fracture reached the maximum, the dimples were narrow and deepest, and the distribution was the most uniform. There were inclusions in dimples that promoted the nucleation of AF, and wire 1 had the fewest inclusions, as shown in figure 6(a). With the addition of 1% CeO$_2$, the number of inclusions increased, and the inclusions were significantly refined, as shown in figure 6(b). With the addition of 3% CeO$_2$, there were the most inclusions, and the distribution of inclusions was the most uniform, as shown in figure 6(d). The area of cleavage fracture where the crack source was located in wire 1 was the largest, and the river pattern was smooth and continuous. The cracks started from between the B clusters and propagated along the adjacent B clusters, and the arrow direction was the crack propagation direction, as shown in figure 6(e), which is consistent with the micrograph in figures 5(a) and (e). The left side of two continuous cracks was the crack source generated between the B clusters, the right side was the crack source generated by the decohesion of an inclusion, and the pit after the inclusion fell off appeared at the crack source on the right, as shown in figure 6(f). When the CeO$_2$ content increased from 1% to 3%, the average area of the cleavage fracture where the crack source was located gradually decreased, the river pattern gradually decreased, the crack source in wire 4 had the smallest average area and smallest river pattern, as shown in figures 6(f)–(h).

Figure 7 shows the EDS of inclusions on the fracture surface. The inclusions in dimples were basically spherical. The EDS analysis indicates that the inclusions were mainly composed of Cr, Al, Si, Zr, Ti and trace Ce. The addition of Ce reacted with Al, O, Ti and other elements to form rare earth composite inclusions [13]. The addition of Ce promoted Al$_2$O$_3$ inclusions to be replaced by CeAlO$_3$ inclusions to form Al$_2$O$_3$-coated Ce–Al–O rare earth type composite inclusions, which can be used as the nucleation core of TiC and finally form TiC-CeAlO$_3$ inclusions. Rare earth composite inclusions were formed, and the curved surface of the inclusions was gradually smooth [14].

Figure 6(e) shows the cleavage fracture caused by the boundary cracking of B clusters, which extended to the critical microfracture surface and through the entire specimen. The control condition for crack growth is: $\sigma_y \geq \sigma_f$ ($\sigma_y$ is the normal stress at the front of the gap, and $\sigma_f$ is determined by the size of the critical fracture surface). The cleavage fracture mechanism was different from the ductile fracture mechanism; it did not depend on the density of the second phase particles but on the size of the critical microfracture surface controlled by the phase composition of the microstructure. The coarsest B cluster was the weakest area in the microscopic composition, and the critical event of cleavage fracture was confirmed as crack propagation through the boundary of the B cluster. The lath structure was refined with high cleavage fracture stress, which delayed the occurrence of cleavage fracture and improved the impact toughness of the material [15]. In addition, Mao [3]
Figure 6. SEM images of dimples and cleavage facets in weld metals. (a) and (e) wire 1; (b) and (f) wire 2; (c) and (g) wire 3; (d) and (h) wire 4.
Figure 7. SEM image of inclusion and EDS spectrum in weld metal: (a) and (e) wire 1; (b) and (f) wire 2; (c) and (g) wire 3; (d) and (h) wire 4.
found that the impact toughness of the weld metal increased, and the river pattern area at the fracture was smaller. He noted that the high-angle grain boundaries in the closely packed B clusters effectively prevented cracks. Figures 6(f)–(h) show the cleavage fracture caused by the decohesion of an inclusion, and the crack initiation was surface shear stress-induced decohesion of the inclusion. The dislocations accumulated at the inclusion particles during material deformation, the cracks initiated and propagated in the inclusion particles at the end of the dislocation accumulation, and the cracks of the inclusion particles that started to crack immediately extended into the matrix grains; then, the cracks extended through the entire specimen. With the addition of CeO₂, CeO₂ promoted the formation of inclusions and combined with Al, O, Ti and other elements to form rare earth composite inclusions. The thermal diffusivity of rare earth inclusions was more consistent with the matrix, which helped improve the comprehensive mechanical properties of the weld metal. Rare earth inclusions can avoid the generation of additional stress, cause the inclusions to be difficult to separate from the matrix, increase the crack extension energy, and improve the crack extension resistance of the weld metal [16]. In addition, inclusions can promote the formation of AF, and the effective inclusion size for AF nucleation was mainly 1–4 μm [17–19]. The observed size of the inclusions in the dimples was 1–2 μm, and the size of the inclusions was within the range of effectively promoting the formation of AF in the weld metal. AF was preferentially nucleated in the grains, which played a role in the segmentation of austenite grains. The growth of DUB was inhibited, the lath of grain was refined, the occurrence of cleavage fracture was delayed, and the toughness was improved.

4. Conclusions

(1) The Charpy V notch impact toughness of the weld metals in four groups with different CeO₂ additions at −40 °C was 45 J, 61 J, 64 J, and 71 J, and the tensile strength of the weld metals was 843 MPa, 903 MPa, 870 MPa, and 808 MPa, respectively. Wire No. 2 with 1% CeO₂ had the best tensile strength, and wire No. 4 with 3% CeO₂ had the best toughness but the lowest strength. The comprehensive mechanical properties of wire No. 2 wire were considered the best.

(2) The addition of CeO₂ promoted the formation of rare earth composite inclusions and AF in the weld metal, refined the lath structure, inhibited the formation of M and B, changed the pattern of crack generation and improved the impact toughness of the weld metal.

(3) The optimal proportion of microstructure was found in the weld metal with 1% CeO₂: AF was 60%, M was 8%, GB was 20%, and DUB was 12%.

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Statement: the data of this study is openly available.

Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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References

[1] Liu S 2014 Effect of micro-alloy elements on weld metal and heat-affected zone microstructure and properties in high strength low alloy steel World Iron & Steel 14 64–72
[2] Zhang T L, Li X Z, Young F, Kim H J, Li H, Jing H Y and Tillmann W 2014 Global progress on welding consumables for HSLA steel ISIJ Int. 54 1472–84
[3] Mao G J, Cao R, Cayron C, Logé R, Gou X L, Jiang Y and Chen J H 2018 Microstructural evolution and mechanical property development with nickel addition in low-carbon weld butt joints J. Mater. Process. Technol. 262 638–49
[4] Keehana E, Karlsson L, Bhadesia H K D H and Thuvandera M 2008 Three-dimensional analysis of coalesced bainite using focused ion beam tomography Mater. Charact. 59 877–82
[5] Wu D, Liu Z J, Qiu R P and Su Y H 2018 Effect of Cr content in flux cored wire on mechanical properties of WQ960 high strength steel welded joint Hot. Work. Technol 47 43–51
[6] Kong H Y, Zhu G P, Zeng Z W, Guo C and Yao R G 2017 Influence of Mo in flux cored wires on the mechanical properties of low alloy steel weld metal Devel. Appl. Mater. 32 18–22
[7] AWS A5.29/A5.29M 2005 Specification for low-alloy steel electrodes for flux cored arc welding American Welding Society
[8] AWS B4.0 2007 Standard methods for mechanical testing of welds American Welding Society
[9] Garrison W M and Maloney J L 2005 Lanthanum additions and the toughness of ultra-high strength steels and the determination of appropriate lanthanum additions Mater. Sci. Eng. A 403 299–310
[10] Hufenbach J, Helth A, Lee M H, Wendrock H, Giebeler L, Choe C Y, Kim K H, Kühn U, Kim T S and Eckert J 2016 Effect of cerium addition on microstructure and mechanical properties of high-strength Fe85Cr4Mo8V2C1 cast steel Mater. Sci. Eng. A 674 366–74
[11] Wan X L, Wang H H, Cheng I and Wu K M 2012 The formation mechanisms of interlocked microstructures in low-carbon high-strength steel weld metals Mater. Charact. 67 41–51
[12] Zhang T, Li Z, Ma S, Kou S and Jing H 2016 High strength steel (600–900 MPa) deposited metals: microstructure and mechanical properties Sci. Technol. Weld. Join. 21 186–93
[13] Lv Y, Peng J, Cai C K, Ren H Z, Zheng L L and An S L 2019 Rare earth Ce on thermodynamics of titanium containing inclusions in steel and its experimental research Iron Steel Vanadium Titanium 40 93–8
[14] Dong J L, Zhu L L and Xin L 2020 Study and control of inclusions in bearing steel with rare earth Special Steel Technol 26 31–4
[15] Chen J D and Cao R 2017 Micromechanism of cleavage fracture of weld metals Acta Metall Sin 53 1427–44
[16] Wang L L, Liu Y Q, Wang O and Chou K C 2015 Evolution mechanisms of MgO Al2O3 inclusions by cerium in spring steel used in fasteners of high-speed railway ISIJ Int. 55 970–5
[17] Shi M H, Du K, Gao P and Zhang J 2018 Microstructure evolution and toughness variation of simulation HAZ with large heat input welding for E40 ship plate steel IOP Conf. Ser. Mater. Sci. Eng. 382 032010
[18] Jiménez A J, Mercado A M P, Hirata V M L, Bórquez A G, Torre A S D I D L, García C M, Muñoz M L S and Díaz E M 2019 Improvement of the toughness and ductility of the weld beads by inducing growth of acicular ferrite with TiO2-nanoparticles during submerged arc welding Mater. Res. Express 6 106534
[19] Gao X Z and Du K P 2019 Effect of carbon and manganese contents on intra-granular acicular ferrite nucleation in steel containing nanoparticles J. Phys. Conf. Ser. 1347 012065