Solidified Microstructure of Wear-Resistant Fe-Cr-C-B Overlays

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Iron-based alloy overlays are widely utilized in industry to extend the service life of components subjected to wear and corrosion attack. Welding is an overlay process commonly employed because of its low cost and high efficiency. The microstructure of an as-welded chromium carbide overlay and a new Fe-Cr-C-B overlay containing multiple alloying elements has been characterized by optical microscopy, scanning electron microscopy, X-ray diffraction, and electron backscatter diffraction (EBSD). The microstructure of the chromium carbide overlay consists of large M_7X_3 primary carbides and austenite and carbide eutectic phases. The microstructure of the new overlay consists of granular MX-type primary carbide ($M = Nb, Ti, Mo$; $X = C$ and B), dendritic δ -ferrite/austenite, eutectic phases of austenite and M_2B boride ($M = Fe$ and Cr). The austenite portion of the microstructure has been subsequently transformed into martensite and retained austenite. The fine MX-type hard particles and refined eutectic and matrix microstructure lead to the high hardness of the overlay. The non-equilibrium solidification process for the complex microstructure is discussed using ThermoCalc.

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I. INTRODUCTION

OVERLAY is a surface treatment that applies a metallic coating on the engaging surface of a given component. There are various methods to apply the overlay, among which welding overlay is efficient and inexpensive. Common welding overlay methods include, but are not limited to, flux-cored arc welding,^[1,2] submerged arc welding,^[3,4] plasma transferred arc welding^[5,6] and gas metal arc welding.^[7,8] Depending on the composition, the as-solidified overlay can have a hypoeutectic, eutectic or hypereutectic microstructure. The hypereutectic overlay contains higher levels of alloying elements and is often used in severe wear applications. The hypereutectic microstructure consists of carbides as the primary phase, austenite or ferrite as one eutectic component and often a networked carbide as the other eutectic component.^[9] The main hard phase for the chromium carbide overlays (CCO) is generally the M_7C_3 carbide. The strong but brittle M_7C_3 carbide

has a hexagonal cross section and can grow relatively long in the c -axis to form a “pencil”-shaped morphology. It has been reported that the hypereutectic overlays tend to contain relief cracking in the transverse direction of the overlay because of the contraction of the weld pool upon solidification cooling^[10] and poor ductility of the carbides; as a result, the cracked coating will show premature failure in a corrosive environment when the corrosive solution can penetrate into the base metal through the cracks.

The nickel tungsten carbide (Ni-WC) system is another commonly used hypereutectic system. Due to the high volume fraction of tungsten carbide hard particles and nickel matrix in the overlay, it usually shows good wear and corrosion resistance.^[11] However, it is generally also the most expensive system. Compared with the Ni-WC overlays, the iron-based chromium carbide overlay system has a lower cost, great flexibility as a welding filler metal and potential for excellent erosion-corrosion resistance due to its high chromium content.

The hypoeutectic overlay, on the other hand, consisting of a lower level of alloying elements, solidifies into primary austenite or ferrite and the eutectics. As a consequence, the hypoeutectic overlay shows a superior weld cracking resistance but relatively low wear resistance due to the lower volume fraction of hard phases.^[12]

The chemical composition of major alloying elements and additions of minor alloying elements have complex effects on the eutectic alloy microstructure. The effect of Ti, Nb and Mo on the steel welding

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coating was studied in strong carbides for the enhanced wear resistance.^[2,13,14] As strong carbide formers, these elements would consume the carbon in the molten pool in the early stage during cooling. The eutectic phases would form after the formation of primary hard phases. It is reported that the oriented M_2B boride (M represents Cr and Fe) crystals would show excellent slurry erosion resistance compared with carbides.^[15–17] If controlled well, the M_2B eutectic structure has the potential to provide an excellent combination of properties. However, the challenge is still how to design an overlay solidified microstructure that has

welding crack resistance as good as that of a hypoeutectic system and wear performance as good as that of a hypereutectic system.

A balance is required considering that a higher content and greater size of carbides in an overlay would give greater wear resistance but also would lead to a higher crack tendency, higher cost and often a lower corrosion resistance.^[18] Thus, the optimized overlay system should contain a fair amount of primary hard phases that provide the wear resistance. In addition, the morphology and size of the primary hard particles need to be optimized so that they will not break easily during

Table I. Lattice Parameters of Phases for Rietveld Refinement of the Raw XRD Data

Phase	<i>a</i> (nm)	<i>b</i> (nm)	<i>c</i> (nm)
Austenite	0.35919 ± 0.00028	—	—
Martensite	0.28655 ± 0.00007	—	0.28997 ± 0.00008
NbC	0.44363 ± 0.00006	—	—
TiC	0.45565 ± 0.00031	—	—
Fe ₂ B	0.51679 ± 0.00023	0.51679 ± 0.00023	0.42195 ± 0.00035

Fig. 1—Optical microstructure of the Wire 1 overlay, showing a traditional chromium carbide overlay structure, (a) at low magnification and (b) at higher magnification.

Fig. 2—Optical microstructure of the Wire 2 overlay top surface at (a) lower magnification and (b) higher magnification.

Table II. Microhardness (HV0.5) of Typical Chromium Carbide Overlay by Wire 1 and Overlay by Wire 2

Wire 1 Overlay Top Surface	Wire 2 Overlay		
	1st Layer	2nd Layer	Top Surface
867 ± 32	600 ± 23	732 ± 32	825 ± 33

Fig. 3—X-ray diffraction patterns of typical CCO overlay by Wire 1.

abrasion wear. The overlay should contain a significant fraction of primary austenite (a steel matrix) that is relatively ductile to help prevent crack formation. The networked eutectics should be uniformly distributed, which bridges other phases together. A good selection of the chromium level is also critical to the corrosion performance.

In this work, we report a novel iron-based overlay system produced by flux-cored arc welding to apply in the wear and corrosion environment. The primary MX-type hard particles are formed first, followed by formation of austenite dendrites and lastly M_2B -containing eutectics. The overlay microstructure is fully characterized as hypereutectic based on the primary carbide dendrites; paradoxically, it can also be considered hypoeutectic based on the solidification of primary δ -ferrite/austenitic dendrites. The formation mechanism of this complex microstructure in the novel overlay is discussed.

II. EXPERIMENTAL PROCEDURE

Two custom-designed overlay weld wires were applied to a 1/2 inch (12.5 mm)-thick mild steel tube inner diameter by self-shield flux-cored arc welding. The chemical composition of Wire 1 is that of a typical chromium carbide overlay (CCO), consisting of 30.0 pct Cr, 4.0 pct C, 2.0 pct Mn and Si each, and balance Fe. The main alloying elements in Wire 2 include 20.0 pct Cr, 1.0 to 2.0 pct C, 1.0 to 4.0 pct Nb, 2.0 pct Mn and Si

Fig. 4—X-ray diffraction patterns of Wire 2 overlay (a) without matrix dissolution and (b) with matrix dissolution by deep etching.

each, < 2.0 pct Ti, Mo and B each, and balance Fe. The 2-mm-diameter wires were welded using 200-240 A and 20 V arc voltage without any preheat or post-weld heat-treatment. Multiple welding layers were deposited. The cross-sectional samples of the overlaid steel tube were cut using the abrasive saw with water cooling.

The samples were then mounted in epoxy followed by a series of grinding processes using #80, #180, #320, #600 and #1200 sandpaper and polishing with 3 μ m, 1 μ m and 0.25 μ m diamond suspension. The polished sample was then etched with Vilella reagent, which consisted of 1 g picric acid, 5 ml hydrochloric acid and 100 ml ethanol. The microhardness tests across the weldment cross section were conducted with test force of 0.5 kgf and dwell time of 10 s on a Wilson VH3100 hardness tester. A matrix of indentations was made to collect multiple hardness data across different zones including base metal, HAZ, the first overlay layer and second overlay layer. The microstructure of the sample was examined by an optical microscope (Zeiss Axio Vert.A1) and scanning electron microscope (Zeiss Evo). Combined energy-dispersive X-ray spectroscopy and electron backscatter diffraction (EBSD) analysis of the

Fig. 5—SEM images of the overlay top layer of Wire 2: (a) backscattered electron image before deep etching, (b) secondary electron image after deep etching, under the same magnification as for (a).

Fig. 6—Secondary electron image after deep etching, showing the lamellar eutectic network with primary carbides under higher magnification.

same view field were carried out on the un-etched samples, which were further polished down to $0.05\text{ }\mu\text{m}$ with a colloidal silica suspension, using Zeiss Sigma FESEM equipped with Oxford EBSD and X-Max EDX systems under an operating voltage of 20 kV, 80 to 140

nm step size and $60\text{ }\mu\text{m}$ objective aperture). The EBSD data processing (reconstructed maps) was carried out using Channel 5 data processing software.

The area fractions of various phases and carbide size were measured using ImageJ software. Phase identification of the top surface of the overlay was performed by X-ray diffraction (XRD) with a scanning angle (2θ) of 20 to 100° with a sampling width of 0.05° on a Rigaku Ultima IV X-ray diffractometer with a Cu K α source at a scan rate of $1^\circ/\text{min}$. XRD can obtain all the phase information of the bulk material inclusively; therefore, it is helpful to extract the matrix phase signal and improve the identification of primary carbide and eutectic phases. The overlay specimen was immersed in the Villela reagent for over 2 days at room temperature. It was then washed and dried and underwent XRD scanning on the same surface to compare with the bulk analysis. Rietveld refinement of raw XRD data was carried out using an open-source software Profex-BGMN^[19] to de-convolute the peaks sharing similar 2θ ranges and to identify and quantify the phases present in the overlay system. The lattice parameter values for the key phases used in the Rietveld refinement procedure are shown in Table I.

III. RESULTS

The microstructure of the chromium carbide overlay by Wire 1 is shown in Figure 1. The as-solidified overlay shows a typical hypereutectic microstructure with large hexagonal M_7C_3 carbide particles surrounded by a matrix of finer eutectic M_7C_3 carbide and eutectic austenite.

However, the microstructure of the Wire 2 overlay displays a complex hypereutectic microstructure that consists of a fine granular primary phase, a dendritic austenite (which later has transformed into martensite) and a eutectic network of austenite and secondary hard phases (Figure 2). Measured using the ImageJ software, the primary hard phase and eutectic phases have an area percentage of $4.7 \pm 0.4\text{ pct}$ and $22.4 \pm 0.2\text{ pct}$, respectively. The primary hard phase has an average size of $2.1 \pm 0.1\text{ }\mu\text{m}$ and is found to have the MX-type ($M = \text{Nb, Ti, Mo}$; $X = \text{C, B}$) crystal structure in later sections. The dendritic austenite regions appear bright in Figure 2(b). No cracks are observed in any of the overlay cross sections.

The microhardness values measured on the top surface of the Wire 1 overlay and various locations at the first layer, second layer and top surface of the Wire 2 overlay are summarized in Table II. The average hardness of the Wire 1 overlay is 867 HV0.5. For the Wire 2 overlay, due to the base metal dilution effect, the hardness of the first overlay layer is less than that of the second layer with an average Vickers hardness of 600 and 732 HV0.5, respectively. At the top surface, the dilution effect is minimized, which results in a higher microhardness value of 825 HV0.5.

Fig. 7—EBSD and SEM/EDS analysis of the top overlay layer of Wire 2. (a) SEM secondary electron image, (b) EBSD phase map of the selected area in which red represents BCC phases, green represents MX phases, yellow represents M_2B phases, blue represents other carbides including $M_{23}C_6$ and M_7C_3 , and black represents non-indexed areas. (c) Kernel average misorientation map of the selected eutectic area. (d)–(i) EDS maps of various elements, C, B, Cr, Nb, Ti and Mo, respectively (Color figure online).

XRD phase identification was conducted on both the Wire 1 and Wire 2 overlay samples. First, in the Wire 1 overlay, it was confirmed that M_7C_3 is the primary and eutectic carbide (Figure 3). The strongest diffraction peak is for austenite $(111)_\gamma$; the second strongest diffraction peak is for martensite $(110)_{\alpha'}$.

The Wire 2 overlay sample, on the other hand, shows the strongest diffraction peaks for the $(110)_{\alpha/\alpha'}$ and $(211)_{\alpha/\alpha'}$ planes of martensite, and minor peaks for Fe_2B , NbC and austenite (Figure 4(a)). Deep etching of the Wire 2 overlay was carried out to dissolve the matrix martensite. Figure 4(b) shows that when the matrix has been dissolved by deep etching, the NbC and Fe_2B peaks are prominent. No peaks from martensite are observed anymore; however, weak austenite peaks are still visible after deep etching.

Figure 5(a) shows the backscattered electron image of the top layer of the Wire 2 overlay with a hypereutectic microstructure consisting of primary MX-type carbide particles, austenite dendritic grains and eutectic

networks. After deep etching, the austenite dendrites have been dissolved and left with a three-dimensional eutectic network and some remnant primary particles adjacent to the eutectic (Figure 5(b)). At a higher magnification, the lamellar structure of the eutectic consisting of eutectic carbides and austenite is clearly seen in Figure 6.

The distribution of various elements and phases in the Wire 2 overlay is shown in Figure 7. The EBSD phase map in Figure 7(b) confirms that the primary hard phases consist of MX phases (green region). To evaluate the local strain distributions in the overlay, a kernel average misorientation map is plotted in Figure 7(c). It shows that there is large misorientation in the prior austenitic dendritic regions, indicating that the BCC ferrite detected by EBSD (red regions in Figure 7(b)) is martensitic in nature, further confirming the XRD results. As shown in Figure 7(d)–(i), the primary MX carbide is found to be rich in Nb, Ti, Mo, C and B, while the eutectic M_2B boride is found to be rich in Cr and B.

◀Fig. 8—SEM backscattered electron (BSE) image of Wire 2 overlay, showing MX-type particles (a), and corresponding line-scan (b) of various elements, C (c), Cr (d), Ti (e), Nb (f), Al (g) and O (h).

An EDS line-scan analysis of the primary carbide particles in the Wire 2 overlay is presented in Figure 8. It confirms that the MX phases are rich in C, Nb and Ti, while it also finds that the core of the MX particle is rich in Al and O, which suggests the core to be Al_2O_3 . It is noted that some MX primary particles contain a darker core, which indicates a segregation of lighter elements under the backscattered electron mode. Some studies have identified similar structures in the M_7C_3 -type primary carbide.^[9,20]

The size of Al_2O_3 is found to be up to 1 μm. Except for the Al_2O_3 found in the core of primary carbides, there were no isolated Al_2O_3 particles detected in the microstructure of the bulk overlay. Aluminum was not intentionally added as an alloying element, but rather as a common deoxidizer to the flux-cored welding wire to protect the overlay from oxidation. It is believed that the high melting point (2072 °C) Al_2O_3 inclusions may have served as the first solid phase during welding solidification.^[21] These inclusions may act as nucleation sites for the primary MX particles.

Figure 9 shows the SEM micrograph and EBSD analysis of the selected area containing an eutectic colony in the Wire 2 overlay. As shown in Figure 9(b), M_2B ($M = \text{Fe}, \text{Cr}$) is confirmed as the main hard phase in the eutectic. Figure 9(c) confirms the large misorientation in the martensite of the prior austenite regions of the eutectic. Enrichment of B and Cr, and depletion of Fe are clearly visible in the EDS maps of the eutectic, shown in Figure 9(d) and (e).

IV. DISCUSSION

ThermoCalc analysis of Wire 1 overlay and Wire 2 overlay is shown in Figs. 10 and 11, respectively. Compared with the new Wire 2 overlay system, the traditional overlay Wire 1 shows a simpler hypereutectic system, as shown in Figure 10(a). The M_7C_3 primary carbides nucleate first at 1420 °C from the liquid followed by an eutectic reaction. The eutectic reaction occurs at a narrow temperature range around 1300 °C where liquid transforms into carbide M_7C_3 and austenite (FCC_A1). Upon further cooling, an allotropic reaction at 860 °C produces a ferritic phase (*i.e.*, if rapidly cooled, martensite phase) from austenite.

Figure 10(b) shows the calculated equilibrium solidification and Scheil solidification curves of the Wire 1 overlay. In ThermoCalc calculations, a complete mixing of all elements in liquid and solid-state diffusion of two fast diffusing elements, carbon and boron, are assumed. The Wire 1 overlay shows the formation of 32 pct mole fraction of primary M_7C_3 carbide and 68 pct mole fraction of eutectics. Some mismatch is expected between phase fractions from the equilibrium ThermoCalc predictions and the actual phase fractions

Fig. 9—Secondary electron image of an eutectic colony in the Wire 2 overlay (a); EBSD phase map of the selected eutectic area in which red represents BCC phases. Green represents MX (Nb(C,B) and Ti(C,B)) phases, yellow represents M_2B phases, blue represents other carbides including $M_{23}C_6$ and M_7C_3 , black represents non-indexed areas (b), Kernel average misorientation map (c) and (d)–(f) EDS maps of boron, chromium and iron (Color figure online).

following the non-equilibrium flux cored arc welding process. The Fe-Cr-C hypereutectic system has been well studied for the last half century.^[20,22–24] Most researchers have focused on the refinement of the microstructure and the control of M_7C_3 carbides.^[25,26] As reported by Berns and Fischer,^[27] TiC and NbC carbides can reach 2500 HV and 1900 HV hardness, respectively. Nevertheless, the M_7C_3 phase only showed hardness values varied from 1190–1800 HV. Some studies^[28,29] of longitudinal growth mechanisms of M_7C_3 have shown that the M_7C_3 can easily grow to over 50 microns in size. The hard and brittle primary phase can cause welding cracks and premature wear failure of the hypereutectic hardfacing.

In Wire 2 overlay, the solidification starts from 1480 °C when the MX-type primary phase (with the FCC_A1#3 crystal structure, and mole fractions of 0.48 C, 0.34 Nb, and 0.18 Ti for chemical composition) forms (Figure 11(a)). At 1357 °C, the δ -ferrite (with the BCC_A2 crystal structure) dendrites start to form. At 1275 °C, a peritectic reaction starts to transform the δ -ferrite and liquid to austenite (with the FCC_A1 crystal structure). By 1200 °C, all δ -ferrite has been consumed. Between 1170 °C and 1145 °C, an eutectic reaction transforms the remaining liquid to austenite and two borides (M_2B _TETR crystal structure with a chemical composition of mole fractions of 0.33 B, 0.32 Cr, 0.27 Fe, 0.07 Mo and 0.01 W and Cr_2B _ORTH crystal structure with a composition of mole fractions of 0.40 Cr, 0.33 B and 0.27 Nb). In solid state, at a

temperature near 1040 °C, $M_7(C, B)_3$ -type carbide starts to precipitate, and at a temperature near 938 °C, $M_{23}C_6$ -type carbides start to precipitate, associated with the dissolution of Cr_2B _TETR boride and growth of Cr_2B _ORTH boride. Although the Cr_2B tetragonal to orthogonal phase transformation mechanism is not clear, the total M_2B content is almost a constant at about 10 pct mole fraction. Upon further cooling, an allotropic reaction at 840 °C produces a ferritic phase (*i.e.*, if rapidly cooled, martensite phase) from austenite.

Scheil calculations of Wire 2 overlay shown in Figure 11(b) indicate a 3 pct mole fraction of primary carbide, 63 pct mole fraction of δ -ferrite and austenite, and 34 pct mole fraction of eutectics. This microstructure composition seems to agree with the actual micrograph in Figure 2(b). There are $M_{23}C_6$ and $M_7(C, B)_3$ forming and $M_{23}C_6$ dissolving at the final, eutectic, stage of liquid-to-solid transformation based on the Scheil solidification model in Figure 11(b), which is not predicted in the equilibrium phase calculations in Figure 11(a). When the δ -ferrite dendrites were forming, they engulfed the primary carbide particles, as shown by the distribution of the primary carbides within the δ -ferrite/austenite dendritic regions (Figure 2(b)). It clearly shows that the eutectic distributes at the δ -ferrite/austenite dendritic interfacial regions, not continuously, but in a fragmented and networked pattern. A continuous networked eutectic would have resulted in a deteriorated toughness and crack resistance.

Fig. 10—Thermo-Calc analysis of chromium carbide overlay by Wire 1. (a) Equilibrium phase constitution as a function of temperature and (b) Scheil solidification diagram.

Fig. 11—Thermo-Calc analysis of the Wire 2 overlay sample, (a) equilibrium phase constitution as a function of temperature, and (b) Scheil solidification diagram.

V. CONCLUSIONS

In this study, a novel Fe-Cr-C-B overlay system has been characterized and compared with conventional Fe-Cr-based chrome carbide overlays using XRD, EBSD and associated thermodynamic calculations. Results showed that the novel Fe-Cr-C-B overlay system has primary MX ($M = \text{Nb, Ti}$; $X = \text{C, B}$) type carbides/borides with an eutectic of M_2B and austenite in a martensite/retained austenite matrix. Compared with the conventional chromium carbide overlay system, where coarse M_7C_3 acts as a primary carbide, the proposed Fe-Cr-C-B overlay system has a finer, harder and uniform distribution of MX primary carbides, more ductile austenitic dendrites and a networked eutectic matrix, which may offer an improved welding crack and overlay wear resistance. The novel overlay microstructure is fully characterized as hypereutectic based on the primary carbide dendrites; paradoxically, it can also be considered hypoeutectic based on the solidification of primary δ -ferrite/austenitic dendrites. These conclusions are mostly based on thermodynamics analysis; the significant effect of welding parameters, and associated chemical kinetics, including oxidation loss of alloying elements during welding, will need to be further quantified.

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