Flux Composition Dependence of Microstructure and Toughness of Submerged Arc HSLA Weldments

CaF$_2$-CaO-SiO$_2$ system fluxes produce good quality niobium microalloyed HSLA weldments with very low oxygen content

BY C. B. DALLAM, S. LIU, AND D. L. OLSON

ABSTRACT. Twenty eight reagent grade fused fluxes from the CaF$_2$-CaO-SiO$_2$ system were used to produce bead-on-plate and double-V-groove submerged arc welds on a quenched-and-tempered niobium HSLA steel. An E70S3 welding wire was used with two different heat inputs—namely, 1.9 and 3.3 kJ/mm (48.3 and 83.8 kJ/in.). A niobium microalloyed steel was selected because of its fine grained microstructure, high yield strength, and high toughness at low temperatures. Fluxes from the CaF$_2$-CaO-SiO$_2$ system were selected because of their low oxygen potential, and their ability to produce low oxygen (80-450 ppm) welds. Quantitative metallography and chemical analysis were performed on the welds. The chemical behavior of this flux system has been characterized with respect to manganese, silicon, niobium, and sulfur.

The lower heat input welds showed predominantly fine microstructure of acicular ferrite. At high oxygen content, a higher percentage of grain boundary ferrite (ferrite veining) was observed. By reducing the oxygen in the weld metal, the amount of acicular ferrite was increased. With further reduction of weld metal oxygen, the main microstructural feature, instead of acicular ferrite, became bainite. Using higher heat input, the weld metal microstructure transition with oxygen level was not so clear.

In spite of the essentially similar optical microstructure and similar chemical composition (other than oxygen), the mechanical properties of the various welds were observed to be very different. Toughness data (upper shelf energy and transition temperature) were found to correlate with weld metal oxygen content. The upper shelf energy decreased with increasing oxygen level in the weld metal.

Introduction and Background

HSLA steels were developed to achieve high yield strength and at the same time maintain a reasonable level of toughness with a minimum of alloying. Due to the possibility of increased design loading and strength to weight ratio, more and more structural applications using HSLA steels are being seen. Some examples are line pipes for gas and oil transportation and off-shore structures. The physical metallurgy of these microalloyed steels for the optimization of their microstructure and properties has already been treated extensively in the literature (Refs. 1-4) and are not discussed in this paper.

Most applications of HSLA steels involve structures where welding is used. Two major consequences of this fabrication process are the deterioration of the base metal properties due to the welding thermal cycles and the introduction of a solidification structure which is heterogeneous (compared to the base metal) both chemically and microstructurally. In the fusion zone, the weld metal composition, heat input and cooling rate, solidification characteristics, and reheating thermal cycles (in multiple pass welds) contribute to the final properties of the weld joint. Adjacent to the fusion zone is a thermally affected region (heat-affected zone, i.e., HAZ) within which the base metal microstructure is altered by the high temperature of the molten weld pool. Martensite or other low temperature transformation products may be formed impairing the toughness of these regions.

As noted below, under separate heading, the presence of oxygen can influence weld metal microstructure and properties. With this in mind, the behavior of CaF$_2$-CaO-SiO$_2$ flux systems was studied with the purpose of reporting on weld metal performance when using these fluxes on a niobium microalloyed steel.

Some Background

Typical Microstructure of C-Mn Steel Weldments

Several different microstructures may be obtained in the weld metal of low carbon microalloyed steels. They are grain boundary ferrite (BF), side plate ferrite (SPF), acicular ferrite (AF), upper bainite (AC), and micro-constituents such as pearlite, cementite, and martensite.

Figure 1 shows some of the main constituents of a C-Mn steel weldment. A comparison of the various classifications of low carbon, low alloy steel weld metal microstructure is shown in Table 1.

Factors Affecting Weld Metal Microstructure

Factors affecting weld metal toughness have been studied and acicular ferrite
was found to be the constituent responsible for the high toughness (Refs. 6, 7). Acicular ferrite is formed intragranularly, resulting in randomly oriented short ferrite needles with a basket weave feature. This interlocking nature, together with its fine grain size, provides the maximum resistance to crack propagation by cleavage. For this reason, it has become increasingly important to understand the factors which would maximize the volume fraction of acicular ferrite in the weld metal.

Weld metal composition (alloying elements and oxygen), prior austenite grain size, and welding heat input (cooling rate) are the three main factors that determine the microstructure of a weld metal. It is shown (Ref. 8) that an increasing cooling rate progressively refines the resulting microstructure from grain boundary ferrite to side plate ferrite, acicular ferrite, bainite, and eventually to martensite.

Alloying elements in the weld metal may come from the base metal, the welding electrode, and the welding fluxes. Hardenability agents such as manganese, chromium, and molybdenum will shift the austenite decomposition transformation to longer delay times. Superimposing a cooling curve on the transformation curves, one notices that the refining of the final weld microstructure can be explained. This is shown schematically in Fig. 2. Composition control is necessary in order to maximize the volume fraction of acicular ferrite, because excessive alloying elements can cause the formation of bainite and martensite.

A number of recent investigations (Refs. 9-13) indicate that oxygen affects the weld metal microstructure and the mechanical properties in the form of inclusions. These inclusions may be a product of the deoxidation process or result from some solid state transformation (Ref. 14). Using Charpy testing data, Ito and Nakanishi (Ref. 15) observed that both high and low weld metal oxygen content weldments (with >300 ppm or <200 ppm) showed poor impact properties. Only the intermediate level oxygen

Table 1—Classification and Terminology of Microstructures in Low C-Low Alloy Steel Weld Metal (Ref. 5).

<table>
<thead>
<tr>
<th>Dube, C.A.</th>
<th>Widger, D.J.</th>
<th>Abson, D.J.</th>
<th>Japanese Researchers[a]</th>
<th>Others[b]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Primary and secondary ferrite sideplates*</td>
<td>Lamellar component (product)</td>
<td>Acicular ferrite</td>
<td>Polygranular ferrite</td>
<td>Polygranular ferrite</td>
</tr>
<tr>
<td>Intragranular ferrite plates*</td>
<td>Acicular ferrite</td>
<td>Acicular ferrite</td>
<td>Acicular ferrite</td>
<td>Ferrite islands</td>
</tr>
<tr>
<td>Massive ferrite</td>
<td>Pearlite</td>
<td>Ferrite-carbide aggregate</td>
<td>Pearlite</td>
<td>Martensite</td>
</tr>
<tr>
<td>Pearlite</td>
<td>Martensite</td>
<td></td>
<td>Martensite</td>
<td>M-A constituent</td>
</tr>
<tr>
<td>Lath martensite</td>
<td></td>
<td></td>
<td></td>
<td>High-C martensite</td>
</tr>
<tr>
<td>Twinned martensite</td>
<td></td>
<td></td>
<td></td>
<td>Upper bainite</td>
</tr>
<tr>
<td>Retained austenite</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Upper (occasionally lower) bainite</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

welds (200 ppm to 500 ppm) produced tough acicular ferrite structures. Kikuta et al. (Ref. 16) reported the same trend for electroslag weldments.

Cochrane and Kirkwood (Ref. 10) studied the effects of weld metal oxygen on acicular ferrite formation by producing a series of weld metals of similar chemical composition, with the exception of oxygen level. Intermediate weld metal oxygen content (200-300 ppm) gave a primarily acicular ferrite microstructure, while side plate ferrite predominated at high oxygen contents (500 ppm). They concluded that weld metal oxygen variation affected the inclusion size distribution and the surface energy of the inclusion/matrix interface which resulted in different microstructures. Abson, Dolby and Hart (Ref. 9) observed the same kind of microstructural evolution as Cochrane and Kirkwood. They also postulated that oxygen-rich inclusions can directly nucleate acicular ferrite. In the absence of these inclusions, lath type structures (bainite) will form.

For higher oxygen systems (>500 ppm), Indacochea and Olson (Ref. 17) obtained predominantly grain boundary ferrite and blocky ferrite (intragranularly nucleated coarse ferrite grains). Some evidence of the direct nucleation of the acicular ferrite phase on inclusions was observed. Increasing weld metal oxygen content, and thus the amount of inclusions, should move the transformation curve in Fig. 2 to shorter delay times.

Harrison and Farrar (Ref. 18) reported that the austenite to ferrite transformation temperatures can be altered by the interaction of inclusions with austenite grain boundaries. A high inclusion concentration tended to reduce the austenite grain size, favoring grain boundary ferrite and side plate ferrite formation. Ferrante and Farrar (Ref. 19) determined that the inclusions control the austenite grain growth by pinning of the grain boundaries, following the Zener equation of precipitate-boundary interaction. They also found that acicular ferrite directly nucleates from the inclusions.

Keville (Ref. 14) and Pargeter (Ref. 20) showed that the inclusions' size, distribution, and type (composition) can be correlated to the microstructure and the toughness of the weld metal. Keville (Ref. 21) also indicated that the inclusions are of complex nature and that distinct geometrical shapes could be associated with the type of welding flux used. A change in inclusion shape was always observed with a change in the chemical composition of the inclusion.

The types of inclusions were also catalogued using particle analyzer scanning electron microscopy (Ref. 22). The idea that the differential thermal contraction of the inclusions and the austenite matrix during cooling of the weldment may provide favorable conditions for the ferrite nucleation is also being investigated (Ref. 23).

Sources of Oxygen in the Submerged Arc Welding Process

The four main sources of weld metal oxygen can be identified as the flux, the base metal, the electrode welding wire, and the atmosphere. In general, the oxygen and nitrogen pickup from the atmosphere can be considered as contamination and constitutes only a minor part of the weld metal oxygen (Ref. 24). However, it may become more significant in welds made with high basicity fluxes.

The lower viscosity of high basicity fluxes may be the cause of the increase in weld metal oxygen pickup (Ref. 24). The base metal and welding electrode contain approximately 100 ppm oxygen each; the amount is determined by the melting process. The amount that the base metal and electrode contribute is appreciably less than that generated from the welding flux. The major components
of a flux are generally oxides, and a reasonably high oxygen potential of the flux would be expected. Therefore, the main source of oxygen in the submerged arc welding process is the flux. Some commercial flux systems based on FeO, MnO and SiO₂ may contribute up to 1000 ppm of oxygen to the weld metal.

The requirement of low weld metal oxygen to achieve high toughness (high volume fraction of acicular ferrite) leads to the reformulation of fluxes attempting to reduce the oxygen transfer. One way is to substitute non-oxygen carriers (such as fluorides) for some of the oxides in the fluxes. Decreasing the amount of oxygen in the flux, a reduction of weld metal oxygen would be expected. A second way is to replace weak oxides (e.g., silica and ferrous oxide) partially by stronger oxides such as calcium oxide and magnesia oxide. Since the Ca-O bonds are much stronger than the Si-O bonds, the degree of dissociation of calcium oxide in the molten flux would be less than that of silica, resulting in a lower weld metal oxygen content. Another possible alternative is the addition of metal powders (e.g., aluminum, titanium) to the fluxes to reduce the weld metal oxygen pickup (Refs. 11, 25, 26).

Experimental Procedure

Twenty-eight reagent grade fused fluxes were produced from the CaF₂-CaO-SiO₂ system, using nominal composition at every 10% increment on the ternary phase diagram—Fig. 3. The details of the flux preparation were reported in a previous paper (Ref. 27). Two quenched-and-tempered niobium microalloyed steel plates of similar grade were used to make the welds, and a 3/₄ in. (2.38 mm) diameter E70S3 welding electrode was used. The chemical composition of the plates and the welding electrode are given in Table 2.

Two sets of experiments were performed:

1. Bead-on-plate welds were made on 4 X 10 X 0.5 in. (100 X 250 X 13 mm) plates (machined from plate 1) with each one of the fluxes. Instead of using the flux hopper, a flux bin was set up along the torch path. The welding process parameters are shown in Table 3. The welds were used to study variables such as arc instability, bead morphology and penetration, and contact angle for interfacial energy calculations.

   Metallographic analysis and Rockwell hardness measurements were performed on each weldment. Chemical analyses were performed using a Baird Atomic SpectroVAC Model 1000 Emission Spectrometer. Weld metal oxygen, nitrogen, sulfur, and carbon contents were determined using Leco Analyzers. The chemical interaction between the molten flux and weld pool was also studied.

2. Double-V-groove welds were made on plates machined from the plate II. The dimensions of the welding plates were 4 X 11 X 0.625 in. (100 X 280 X 16 mm). Specific fluxes, indicated in Fig. 3, were used for this second set of welds. The joint design is shown in Fig. 4; the welding conditions are shown in Table 3.

   The welds were radiographically examined before standard Charpy specimens were prepared. Approximately nine Charpy bars were machined out of each weld. The Charpy V-notch toughness measurements were done using a Tinius Olsen Charpy machine, with the testing temperatures ranging from —100 to 120°C (—148 to 248°F). At least eight temperatures were tested for each weld. Fractographic analyses were also done using AMR scanning electron microscope.

   Carbon extraction replicas were made on some of the welds in order to examine the inclusions and other second phases present in the weld. The replicas were examined using the Philips 200 transmission electron microscope.

   In an attempt to characterize the crystalline nature of the inclusions, an acid extraction was performed. Some drilling shavings from the weld were chemically decomposed with hydrochloric acid (10:1). To accelerate the process of dissolution, the solution was heated to close to boiling with an argon cover gas. After the extraction, the fluid was filtered through a Millipore filtration set-up (0.025 micron pore size). The filtered particles were examined using a Philips x-ray diffractometer and spectrometer.

Results and Discussion

Chemical Behavior of the Fluxes

The final weld composition for a particular element is made up of contributions from the welding electrode, the base metal, and the weld metal-flux reactions. The chemical behavior will be expressed in terms of a quantity called delta (Δ); this is the difference between the composition of a particular element determined analytically and the amount of that element which could be present in the weld if no elemental transfer from the weld pool to the flux or vice versa had occurred. The effects of dilution with the base metal were also taken into account. A negative value of delta means that, during welding, a particular element has been transferred from the weld metal to the slag; a positive delta would indicate the element going from the molten flux to the weld metal.

The chemical composition and the elemental delta values of the first set of welds are presented in Tables 4A, B, and 5. The delta quantities of the elements were then plotted as a function of flux composition. Negative delta values were seen for manganese and niobium—Figs. 5 and 6. These delta quantities were also insensitive to flux compositional changes. This observation implies the activities of manganese and niobium are nearly constant for these systems.

High temperature activity data of MnO (Ref. 28) in the MnO-CaO-SiO₂ system indicate that the MnO activity is almost constant with 40% SiO₂ content in the flux providing the conditions of a constant delta manganese in the weld metal for that system. Comparing the two curves in Fig. 5, the same behavior would be expected for welds made with CaF₂-CaO-SiO₂ fluxes, resulting in the constant delta manganese. A constant delta quantity in this case also suggests that small flux variation will not significantly alter the weld metal chemical composition.

The delta sulfur quantity is plotted as a function of the ratio CaO/SiO₂ in Fig. 7. The deviations were all negative, indicating weld pool desulfurization. With increasing CaO-SiO₂, an increasing sulfur loss was observed. This is related to the oxygen potential of the molten flux in contact with the molten weld pool and can be explained by the desulfurization reaction:

\[ \frac{S_{\text{metal}} + Fe_{\text{metal}} + O^2-_{\text{slag}}}{S^2_{\text{slag}} + FeO_{\text{slag}}} \]  

and:

Table 3—Welding Conditions for the 1st and 2nd Set of Experiments

<table>
<thead>
<tr>
<th></th>
<th>1st</th>
<th>2nd</th>
</tr>
</thead>
<tbody>
<tr>
<td>Potential, volts</td>
<td>30</td>
<td>28</td>
</tr>
<tr>
<td>Current, amperes</td>
<td>500</td>
<td>520</td>
</tr>
<tr>
<td>Travel speed, mm/min</td>
<td>480</td>
<td>265</td>
</tr>
<tr>
<td>Heat Input, kj/mm</td>
<td>187</td>
<td>3.29</td>
</tr>
</tbody>
</table>

Fig. 4—Joint geometry of the double-V-grooved welds; measurements given in inches
where $K'$ is known as the equilibrium index of the sulfur reaction, $\frac{(\%S)}{[\%S]}$ is the sulfur distribution ratio between the metal and the slag, and $\frac{([\%FeO]_k)}{[\%FeO]}$ is the total iron oxide content in the slag.

The increase of $K'$, with increasing basicity in Fig. 8, indicates that the sulfur is being partitioned more to the slag. This translates into a negative delta sulfur quantity and agrees with the trend shown in Fig. 7. By increasing CaO activity in the flux, the sulfur will be removed.
more readily. This can occur by reducing the silica content in the flux for the CaF₂-CaO-SiO₂ system. Silicon had both positive and negative delta values (gained or lost) as shown in Fig. 9. When the SiO₂ content is approximately equal to the CaO content, no silicon transfer was observed. This corresponds to the zero delta silicon region (shaded region indicated in Fig. 9). Above that region (%SiO₂/%CaO greater than one), there was an increase of silicon in the weld pool indicating that silicon was transferred from the flux to the weld. When %SiO₂/%CaO is less than one, a

Fig. 5 — Delta manganese plotted as a function of weight-percent CaF₂ for two different flux systems

Fig. 6 — Delta niobium plotted as a function of weight-percent CaF₂ for different SiO₂ contents in the CaO-CaF₂-SiO₂ flux system

Fig. 7 — Delta sulfur plotted as a function of basicity index CaO/SiO₂

Fig. 8 — Variation of sulfur equilibrium index (K').

\[ B = \frac{\%CaO + 1.4 \times \%MgO}{\%SiO₂ + 0.84 \times \%P₂O₅} \]

Data were collected from various sources using basic open hearth furnaces.
Welds made with silica free fluxes showed the lowest oxygen contents (<100 ppm). Increasing the silica content in the flux, the weld metal oxygen was found to increase. To a lesser degree, an increase in CaO content also increased the oxygen content in the weld metal.

Traditionally, weld metal oxygen and mechanical properties had been related to the basicity index. Tuliani et al. (Ref. 31) showed that higher basicity fluxes resulted in lower weld metal oxygen, sulfur, and silicon. Weld metal toughness was also said to improve with increasing basicity index (Ref. 32). However, it is still questionable whether the parameter basicity index expresses any fundamental relationship. The flux basicity index (B.I.) equation proposed by Tuliani (Ref. 31) is:

\[ B.I. = \left[ \frac{1}{2} \left( \frac{\text{CaO} + \text{CaF}_2 + \text{MgO} + \text{BaO} + \text{SrO} + \text{K}_2\text{O} + \text{Li}_2\text{O} + \frac{1}{2}(\text{MnO} + \text{FeO})}{\text{SiO}_2 + \frac{1}{2} (\text{Al}_2\text{O}_3 + \text{TiO}_2 + \text{ZrO}_2)} \right) \] (3)

Eagar (Ref. 24) modified this relationship by omitting the CaF\(_2\) term. Using the equation developed by Eagar (Ref. 24), the weld metal oxygen contents were plotted as a function of basicity index—Fig. 12. The data points showed large scattering without following the usual trend as indicated by the solid curve proposed by other authors (Refs. 24, 31). However, if the weld metal oxygen content were plotted using all the terms in equation (3), the data points fell smoothly in the band as seen in Fig. 13 and reported in the literature (Ref. 24). This complicates the interpretation of the relationship between the basicity index and weld metal oxygen. It is also known that weld metal oxygen content increases with an increasing heat input.

**Microstructure**

A definite trend of microstructural variation was observed within the series of weld metals. For the purpose of analysis, the following discussion will be based upon three particular weldments. Figure 14 shows the light micrographs of the three weldments. Their oxygen contents are 350, 250, and 140 ppm, respectively (each containing similar manganese and silicon contents).

The results of the oxygen analysis on the weld beads are shown in Fig. 11.
phy showed that acicular ferrite constituted more than 90% of the microstructure—Fig. 14B. Decreasing the oxygen further to 140 ppm, a finer acicular ferrite was not obtained; instead long and aligned ferrite laths were formed with an aspect ratio of approximately 10:1 to 12:1. Carbide precipitation could be seen between the ferrite plates and were thus classified as bainite—Fig. 14C.

The results depicted in Fig. 14 suggest that, for a given flux system, there is an optimum level of weld metal oxygen producing the maximum amount of acicular ferrite. In the CaF₂-CaO-SiO₂ flux system, the optimum weld metal oxygen content ranged from 200-250 ppm corresponding to a microstructure approximately 90% acicular ferrite.

Physical Properties

The weld penetration, interfacial tensions, and arc stabilities were quantitatively measured and analyzed. The results have been reported in a previous paper (Ref. 27), and are not discussed here.

Recommended Fluxes from the Fluorspar-Lime-Silica System

From the results of the first set of welds, important conclusions can be reached with respect to the applicability of CaF₂-CaO-SiO₂ flux system. There is a flux compositional region which showed very good performance during welding; this appears as the shaded area in Fig. 15. Fluxes on the CaF₂-CaO side of the ternary system would not be advisable because of its lack of a silicate network. Weld pool protection and the weld surface quality were observed to be poor. The region of the CaF₂-SiO₂ side should be avoided because of the two liquids region. Flux behavior may be unpredictable. Welds made in the upper left region should be avoided in the welding of HSLA steels because the

Fig. 14 — Microstructural variation with weld metal oxygen content: A - 350 ppm of oxygen, mixed microstructure of grain boundary ferrite and acicular ferrite; B - 260 ppm of oxygen, microstructure predominantly acicular ferrite; C - 107 ppm of oxygen, microstructure predominantly upper bainite.
Fig. 15—Partial ternary diagram of CaF₂-CaO-SiO₂ system showing as the shaded region the flux compositions which resulted in good quality welds

"higher" weld metal oxygen content (low compared to many welds made with other flux systems) would yield a larger amount of grain boundary ferrite and side plate ferrite than desired for the maximum toughness. Regions to the left side ought to be avoided because of the steep ridges in the melting temperature of the fluxes.

**Toughness**

From the indicated region in Fig. 9, nine fluxes could be selected along the zero delta silicon quantity line for the second set of welds. The chemical composition of the second pass (the untempered bead) of this second set of weldments was determined and presented in Table 6.

This criterion of flux selection proved adequate, because with the exception of oxygen, the welds showed similar composition. A higher heat input of 3.3 kJ/mm (83.8 kJ/in.) was utilized to achieve deeper penetration such that the Charpy specimens were made entirely of weld metal. Due to the slower cooling rate of these weldments, the microstructural variation was not as clear as the lower heat input welds. High magnification light micrographs showed that slightly finer acicular ferrite was associated with lower oxygen welds. However, no significant difference in the microstructure could be found to distinguish the welds. In spite of the similar microstructure of the different welds, their toughness measurements showed quite different results. The upper shelf energies and the ductile brittle transition temperatures (DBTT) of the weldments are given in Table 7.

With an increasing weld metal oxygen content, the upper shelf energy absorbed at fracture decreased. This relationship is shown in Fig. 16. In the upper shelf region, the Charpy specimens fracture by ductile failure mode, absorbing a maximum amount of energy which is related to the magnitude and distribution of strain at notch root. It is mainly influenced by the presence of inclusions (population, spatial distribution, and shape). Due to the very low solubility of oxygen in BCC iron, the total amount of oxygen in solution is negligible. At room temperature, oxygen exists in the combined form as inclusions (e.g., oxides, silicates, aluminates, oxysulfides, etc.). Based on this argument, weld metal oxygen content could be used as an estimate indicating the amount of inclusions in the weld metal as a correlating parameter. Fundamentally, however, inclusion content should be the more appropriate variable to be compared.

Transmission electron micrographs of selected carbon extraction replicas are shown in Fig. 17. High weld metal oxygen Table 7—The Upper Shelf Energies (USE) and the 100 Joules Transition Temperatures (TT<sub>100</sub>) of the Nine Welds Produced Using a Heat Input of 3.3 kJ/mm

<table>
<thead>
<tr>
<th>Weld</th>
<th>Flux</th>
<th>USE, joules</th>
<th>TT&lt;sub&gt;100&lt;/sub&gt;, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5</td>
<td>215</td>
<td>-3</td>
</tr>
<tr>
<td>2</td>
<td>8</td>
<td>220</td>
<td>-3</td>
</tr>
<tr>
<td>3</td>
<td>9</td>
<td>195</td>
<td>+15</td>
</tr>
<tr>
<td>4</td>
<td>13</td>
<td>190</td>
<td>+18</td>
</tr>
<tr>
<td>5</td>
<td>17</td>
<td>200</td>
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</tr>
<tr>
<td>6</td>
<td>18</td>
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<tr>
<td>9</td>
<td>27</td>
<td>165</td>
<td>+18</td>
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Table 6—Chemical Composition of the Second Set of Weldments—Heat Input 3.3 kJ/mm, %<sup>a</sup>

<table>
<thead>
<tr>
<th>Weld</th>
<th>Flux</th>
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<th>Mn</th>
<th>Si</th>
<th>Nb</th>
<th>Cu</th>
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<tr>
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<tr>
<td>2</td>
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<td>1.09</td>
<td>0.46</td>
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<td>0.12</td>
<td>240</td>
<td>80</td>
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<tr>
<td>3</td>
<td>9</td>
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<td>0.41</td>
<td>0.023</td>
<td>0.14</td>
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<td>74</td>
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<tr>
<td>4</td>
<td>13</td>
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<td>1.17</td>
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<td>0.021</td>
<td>0.12</td>
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<td>86</td>
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<tr>
<td>5</td>
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<td>8</td>
<td>25</td>
<td>0.088</td>
<td>1.19</td>
<td>0.44</td>
<td>0.023</td>
<td>0.12</td>
<td>338</td>
<td>81</td>
</tr>
<tr>
<td>9</td>
<td>27</td>
<td>0.079</td>
<td>1.12</td>
<td>0.51</td>
<td>0.019</td>
<td>0.12</td>
<td>432</td>
<td>75</td>
</tr>
</tbody>
</table>

<sup>a</sup> Oxygen (O) and nitrogen (N) given in ppm.
samples showed higher inclusion content than the lower oxygen welds. Finer particles and larger size variation were also seen to be associated with the higher oxygen welds. Particles' sizes ranged from 0.05 to 1 micron, and various geometrical shapes were also observed. This all seems to support, to a certain extent, the previous statement that the weld metal oxygen can be used to indicate the amount of inclusions in the weld metal for correlation purposes.

To further analyze the inclusions effect on the weld metal toughness, the highest and lowest weld metal oxygen samples were chosen. Their Charpy V-notch energy transition curves as a function of temperatures are shown in Fig. 18. Corresponding scanning electron micrographs of the fracture surfaces at the upper shelf and lower shelf regions are shown in Figs. 19 and 20, respectively.

At the upper shelf regions, dimples could be seen in the fracture surface of both the samples, indicating ductile failure by the microvoid fracture mechanism. The difference between the two samples is not so much in the number of dimples, but in the amount of strain associated with each dimple. The microvoid fracture mechanism can be described as being composed of three stages. The first stage is the initiation of a microvoid which occurs by the decohesion of a particle from the matrix material, or by the fracture of a second phase material. The second stage of the microvoid model is the growth of the dimple which is independent of inclusion parti-

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*1 micron = $10^{-3}$ mm = 0.00004 in.
The energy difference observed between the high and low oxygen welds may be explained by the presence of small regions of ductility between the flat fracture facets. They were more frequently observed in the low oxygen welds. This indicates that the crack has to change directions during propagation throughout the material, and while doing so, a small amount of ductility is seen. This is related to the finer grain size (thus, more grain boundaries) observed in the lower oxygen weld metal.

A minimum transition temperature was found in the range of 200-300 ppm of oxygen. This agrees with the upper shelf energy data shown previously. The same behavior has been observed and reported by Devillers et al. (Ref. 23). When analyzing these toughness data and comparing with the metallographic and fractographic results, the optimum weld metal oxygen content for the CaF$_2$-CaO-SiO$_2$ system was determined to be in the range of 200-300 ppm.

Particle Extraction

In an attempt to study the chemical and crystalline nature of the particles, an acid dissolution technique was used. The collected residue was analyzed using x-ray spectrometry and diffractometry. Peaks for several elements including manganese, silicon and niobium were seen. However, no diffraction pattern could be obtained. This seems to suggest that the inclusion particles could be amorphous even though they showed definite facets. The negative results using the x-ray techniques could also be due to the extremely small quantity of residue obtained being insufficient for the characterization.

Conclusions

The results of this investigation led to the following conclusions.

1. The CaF$_2$-CaO-SiO$_2$ flux system is found to produce good quality niobium microalloyed HSLA steel weldments with very low oxygen content (100-460 ppm).

2. Manganese and niobium showed negative delta values during welding, indicating a loss from the weld pool to the slag.

3. There is a region of zero delta silicon where weld pool chemistry can be easily controlled.

4. The negative delta sulfur values indicated that the CaF$_2$-CaO-SiO$_2$ flux system was effective in sulfur control.

5. The optimum weld metal oxygen content for this flux system ranged from 200-300 ppm, corresponding to a microstructure of approximately 90% acicular ferrite.

6. The refinement of the weld metal microstructure due to oxygen decrease resulted in an improvement of the weld metal toughness.

7. High weld metal oxygen welds showed high inclusion contents, but the inclusions were observed to be finer in size.

8. In the lower oxygen content welds, a larger strain was found to associate with the upper shelf ductile dimples leading to a higher energy absorbed at fracture.

9. The lower shelf failure was characterized by cleavage fracture with small regions of ductility between the flat fracture facets which account for the energy difference between high oxygen and low oxygen welds.

Acknowledgments

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References


and Consumables for Welding. The Welding Institute, London.
AN INVITATION
TO
AUTHORS

Gentlemen:

The American Welding Society will hold its 67th Annual Convention and AWS Welding Show in Atlanta, Georgia, during April 13-18, 1986. One of the most important events of our 67th Annual Convention will be its Professional Program.

It is indeed a pleasure to invite you as Authors to be participants in the Professional Program of our 67th Annual Convention. On this occasion, the Society is offering an opportunity to Authors to bring the results of outstanding work on their part to the attention of our entire membership, the welding industry, and the nation's metalworking industries.

To this end, the Society's Technical Papers Committee will be happy to receive your application for participation in our 67th Annual Convention Professional Program; the Committee is inviting 500-word summaries for, basically, two categories of papers:

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Paul W. Ramsey
Executive Director

May 1, 1985
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67th Annual AWS Convention
Atlanta, Georgia, April 13-18, 1986

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