

Limits on Ti Element Transfer in Submerged Arc Welding: Thermochemical Analysis

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TiO₂ inclusion formation in the weld pool is required to produce potent nucleation sites for acicular ferrite microstructure formation. Weld metal oxygen content must be limited to ensure acceptable materials properties, although sufficient oxygen is required for inclusion formation. Weld pool oxygen is sourced from the decomposition of flux oxides. Weld metal oxygen is controlled with flux chemistry formulation by CaF₂ dilution of oxides. In conventional submerged arc welding (SAW), the flux is the source of weld metal Ti. Transfer of Ti from the slag appears to be limited to 400 parts per million (ppm). SAW modification by metal powder additions changes the element transfer reactions. Thermochemical analysis is applied to explain the limitations in Ti transfer from the slag, compared to improved reaction conditions for Ti element transfer in the aluminum-assisted Ti alloying of weld metal. Low TiO₂ activity due to low flux TiO₂ content from CaF₂ dilution and Ti loss from Ti-fluoride gas formation limits Ti transfer from slag. Aluminum-assisted alloying of the weld metal shifts the gas composition from Ti-fluoride formation to Al-fluoride formation and lowers the system partial oxygen pressure to increase the weld metal Ti content, with acceptable ppm O remaining in the weld metal.


welding showed increased parts per million (ppm) Ti in the weld metal to 580 and 700 ppm with 292 and 283 ppm O, resulting in increased AF in the weld metal at 74% and 92%, respectively.^[3,4] The source of oxygen in submerged arc welding (SAW) is the decomposition of flux oxides in the high-temperature arc plasma of the arc cavity.^[5] Weld metal oxygen content control has two aims. First, the oxygen content must be limited to 200–500 ppm O in the carbon steel weld metal to ensure acceptable materials properties because too many oxide inclusions weaken the weld metal structure.^[6] Second, sufficient oxygen must be present in the weld pool to form enough inclusions of the required size. Weld metal oxygen is controlled mainly via the flux chemistry formulation, by diluting the flux oxide content with fluoride, typically added as CaF₂.^[5] In addition, the flux formulation must ensure slag formation of acceptable physicochemical properties such as viscosity, surface tension, and melting behavior.^[7]

1. Introduction

An acicular ferrite (AF) microstructure in carbon steel weld metal ensures excellent materials properties such as impact toughness.^[1] Nonmetallic inclusions serve as nucleation sites for AF.^[2] Titanium oxide inclusions form potent nucleation sites for AF. Formation of titanium oxide inclusions requires sufficient concentrations of Ti and O in the weld pool. Increased weld metal Ti content may increase the weld metal AF content. The application of Ti metal and alloys in powder or fiber format in shielded metal arc welding and gas metal arc

For maximum element transfer from the molten flux (slag) to the weld pool, it is generally accepted that maximum oxide activity in the slag is required by using the metal–slag equilibrium as a guideline.^[8–11] However, it is well known that metal–slag equilibrium does not hold in SAW.^[11] Recent work has demonstrated that gas–slag–metal equilibrium calculations can effectively simulate the conventional SAW process to accurately predict the weld metal ppm O from the specified input flux, base plate, and weld wire compositions and masses.^[12,13] Titanium transfer from the slag to the weld metal in conventional SAW seems to be limited to ≈400 ppm, and according to recently reported results, the transfer of Ti appears to be independent of the flux basicity.^[14] Conventional SAW refers to applying one or more solid arcing wires beneath a flux covering. The application of alternate element transfer methods in SAW, in addition to element transfer from the flux oxide, can dramatically increase the weld metal titanium content.^[15,16] Modified SAW considered in this work is the application of unconstrained metal powders that are not contained in tubular form, as in metal-cored and flux-cored wires. The objective of this work is to apply thermochemical analysis to explain titanium transfer limitations in the conventional SAW process, as compared to modified SAW process metallurgy with aluminum-assisted transfer of Ti to the weld metal. In this work, the weld metal ppm O and ppm Ti achieved in conventional and modified SAW are compared. This novel work first applies thermochemical analysis to back-test the metal–slag

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equilibrium for Ti element transfer in conventional SAW in comparison to that from Ti metal powder additions, and second applies gas–slag–metal thermochemical analysis to explain how the changes in the gas-phase influence Ti element transfer in conventional SAW versus Ti metal powder added to SAW versus aluminum-assisted Ti powder alloying in SAW.

2. Data Set

The data set used in the thermochemical analysis is summarized in **Table 1** in terms of the fluxes applied to attain the weld metal Ti and O content. The carbon steel base plate and weld wire compositions used in each study are summarized in **Table 2**. The data set includes mostly conventional SAW results made with various flux chemistries, both pre-melted and agglomerated fluxes were applied with up to two arcing weld wires and at different welding heat input values.^[12,14–17] Three nonconventional welding results

were reported with added Ti metal powder to attain higher Ti levels in the weld metal, as indicated by the mass percentage numbers in **Figure 1**.^[15,16] From the conventional SAW results, it is seen that a narrow band of ppm Ti was obtained at various ppm O, as displayed in Figure 1, with the maximum weld metal Ti content of 410 ppm Ti at 150 ppm O. Interestingly, it is seen that about 300 ppm Ti was obtained in weld metal with 300 ppm O and also at 700 ppm O. These numbers indicate that the weld metal ppm O does not correlate with weld metal ppm Ti and that the WM ppm O is not the only determining factor in setting weld metal ppm Ti. Applying Ti metal powder to achieve higher Ti content in the weld metal, at ≈ 500 ppm O, indicates a shift from the accepted slag–metal equilibrium approach in weld metallurgy theory. Several studies on the transfer of Ti in conventional SAW were not used in the data set because the results are not complete in terms of ppm Ti and ppm O in the weld metal, or the complete flux analyses were not reported. This is typically due to the study aim being different from the process metallurgy

Table 1. Input flux compositions.

Compound [mass%]	Flux 4 ^[12,16]	Flux 5 ^[12]	Flux W ^[15]	F1 ^[17]	F2 ^[17]	F3 ^[17]	F4 ^[17]	T1 ^[14]	T2 ^[14]	T3 ^[14]	T4 ^[14]	T5 ^[14]
SiO ₂	19.6	18.6	17.0	16.3	16.3	15.8	15.0	0	0	0	0	0
TiO ₂	1.0	10.7	10.0	1.0	5.9	10.8	15.9	20	25	30	35	40
Al ₂ O ₃	24.9	36.0	34.0	19.7	18.7	17.6	16.9	0	0	0	0	0
FeO	2.4	5.4	5.0	1.3	1.4	1.3	1.3	0	0	0	0	0
MnO	6.8	12.3	8.0	2.9	2.6	2.5	2.4	0	0	0	0	0
CaO	0.1	2.3	1.0	1.0	1.3	1.1	1.0	0	0	0	0	0
MgO	22.2	4.9	14.0	17.0	16.6	15.8	15.0	0	0	0	0	0
Na ₂ O	1.6	2.2	0.0	1.8	1.7	1.9	1.8	0	0	0	0	0
K ₂ O	0.2	0.5	0.0	0.6	0.6	0.6	0.8	0	0	0	0	0
ZrO ₂	0	0.2	4.0	0.03	0.04	0.05	0.03	0	0	0	0	0
CaF ₂	17.9	4.2	8.0	38.3	35.1	32.8	29.9	80	75	70	65	60
Basicity index (BI)	1.43	0.55	0.72	2.28	2.00	1.80	1.61	8.00	6.00	4.67	3.71	3.00
Heat input [kJ mm ⁻¹]	2.0	2.0	4.9	5.9	5.9	5.9	5.9	6.0	6.0	6.0	6.0	6.0

Table 2. Base plate and weld wire compositions.

Compound [mass%]	Plate ^[12,16]	Wire ^[12,16]	Plate ^[15]	Wire ^[15]	Plate ^[14]	Wire ^[14]	Plate ^[17]	Wire ^[17]
C	0.120	0.110	0.05	0.041	0.052	0.127	0.035	0.105
Si	0.155	0.137	0.28	0.19	0.142	0.049	0.160	0.180
Mn	1.340	0.990	1.88	1.02	1.540	1.650	1.420	1.800
Ti	0.005	0	0	0	0.010	0.010	0.015	0
Ni	0.020	0	0.243	0.025	0.075	0.009	0.120	0
Cr	0.160	0	0.019	0.031	0.028	0.030	0.020	0
Mo	<0.01	0	0.387	0.006	0	0	0	0
P	0.019	0.009	0.002	0.006	0	0	0	0
S	0.007	0.023	0.009	0.011	0	0	0	0
Cu	0.030	0.140	0.032	0.079	0	0	0	0
Al	0.067	0	0.074	<0.005	0	0	0	0
O	0.007	0.003	0	0	0.003	0.003	0.003	0.003
Balance	Fe	Fe	Fe	Fe	Fe	Fe	Fe	Fe

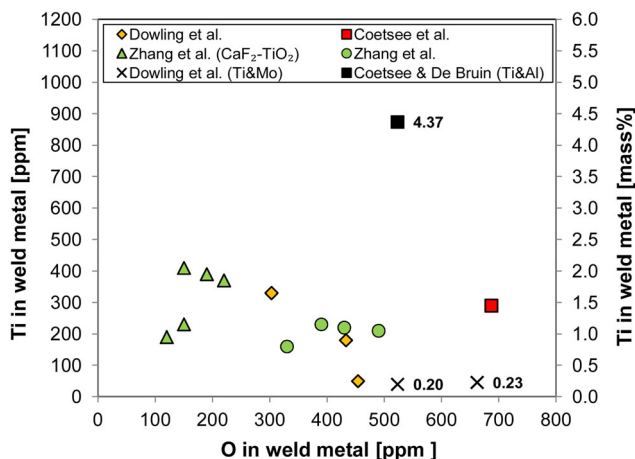


Figure 1. Weld metal ppm O versus Ti content.

consideration of SAW, for example, the aim is to study the weld metal materials properties. In some instances, the flux formulation is not stated to protect commercial interests. From these studies, it appears that the weld metal ppm Ti attained were all below the maximum value of 410 ppm Ti in the data set considered here.^[18–23]

In conventional SAW, the flux formulation is set to control the weld metal ppm O via the flux basicity index (BI) according to the empirically determined trend line of Tuliani et al.^[24] Figure 2 shows the flux BI versus weld metal ppm O for the data set, and Figure 3 shows the flux BI versus weld metal Ti content. In Figure 2, there are large deviations from the trend line of Tuliani et al.^[24] In the data displayed in Figure 3, there is no clear relationship seen between BI and weld metal Ti content. The expression for the BI is as displayed in Equation (1) with inputs of mass% of each flux compound.^[24]

$$BI = \frac{\%CaF_2 + \%CaO + \%MgO + \%BaO + \%SrO + \%Na_2O + \%K_2O + \%Li_2O + 0.5(\%MnO + \%FeO)}{\%SiO_2 + 0.5(\%Al_2O_3 + \%TiO_2 + \%ZrO_2)} \quad (1)$$

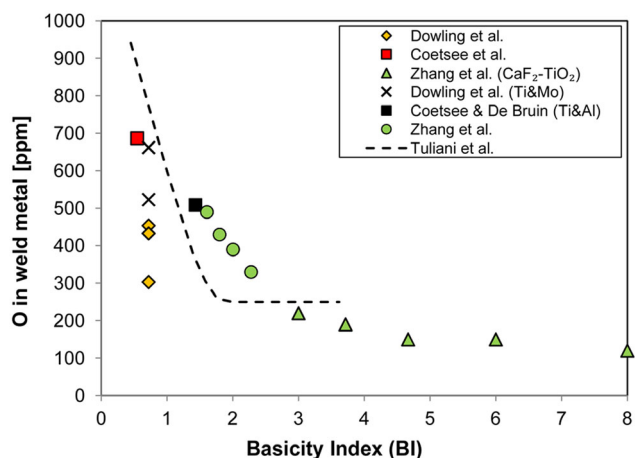


Figure 2. BI versus weld metal ppm O.

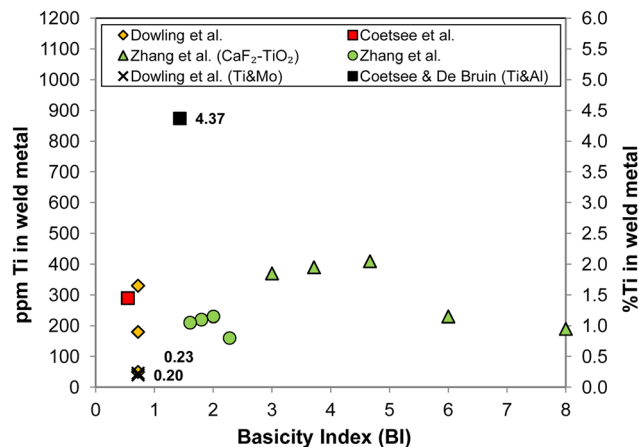


Figure 3. BI versus weld metal Ti content.

3. Discussion

Element transfer between the slag and the weld pool is often written as displayed in Equation (2).^[8] According to reaction thermochemistry, a decrease in the weld metal oxygen should shift reaction (2) to the right to transfer more Ti to the weld metal since the transfer of Ti from the flux to the weld metal occurs due to the dissociation of TiO_2 . The source of oxygen in the weld metal is the decomposition of oxides in the arc cavity.^[5] The initial oxygen content in weld wire droplets in the arc cavity was measured at 2000–3000 ppm O.^[25,26] Therefore, to increase the transfer of Ti from the flux, the initial high level of oxygen added to the weld pool must be significantly reduced via deoxidation reactions, similar to steel ladle metallurgy reactions and/or the initial source of oxygen in the arc cavity must be controlled to lower levels.



$$K = \frac{(a_{Ti})(a_O)^2}{a_{TiO_2}} \quad (3)$$

a_{TiO_2} = activity of TiO_2 in molten flux (slag)

a_O and a_{Ti} = Henrian activity of O and Ti in steel

Here, the initial thermochemical analysis consists of calculating of the activity of TiO_2 in the initial unreacted molten flux (see Table 1) to compare the driving force for reaction (2) in the metal–slag equilibrium. The Equilib module in FactSage 7.3 with the FToxid database was used in activity calculations. The FToxid-SlagA solution model was selected to calculate the activity of TiO_2 . The activity values were calculated for 1800–2100 °C, as shown in Table 3. The extrapolation of thermodynamic data beyond the typical maximum experimental temperature of 1600 °C applied in laboratory measurements is accepted in SAW equilibrium calculations.^[8]

The activity values for 2000 °C are displayed in Figure 4 since the temperature of 2000 °C was calculated previously as the effective slag–metal equilibrium temperature in conventional SAW, and this temperature is based on maximum weld pool temperature measurements.^[8] The maximum TiO_2 activity value in Figure 4 is 0.20 in the 40% TiO_2 –60% CaF_2 flux, but the weld

Table 3. TiO₂ activity ($\times 10^{-2}$) in molten flux versus temperature.

Temperature [°C]	Flux 4 ^[12,16]	Flux 5 ^[12]	Flux W ^[15]	F1 ^[17]	F2 ^[17]	F3 ^[17]	F4 ^[17]	T1 ^[14]	T2 ^[14]	T3 ^[14]	T4 ^[14]	T5 ^[14]
1800	0.212	10.8	5.72	0.223	1.34	3.06	5.30	18.2	19.9	21.4	23.0	24.6
1900	0.196	10.3	5.42	0.204	1.30	2.81	4.90	15.8	17.3	18.9	20.4	22.0
2000	0.182	9.87	5.12	0.188	1.20	2.58	4.54	13.7	15.2	16.7	19.8	19.8
2100	0.170	9.44	4.85	0.174	1.10	2.38	4.21	11.9	13.4	14.8	17.9	17.9

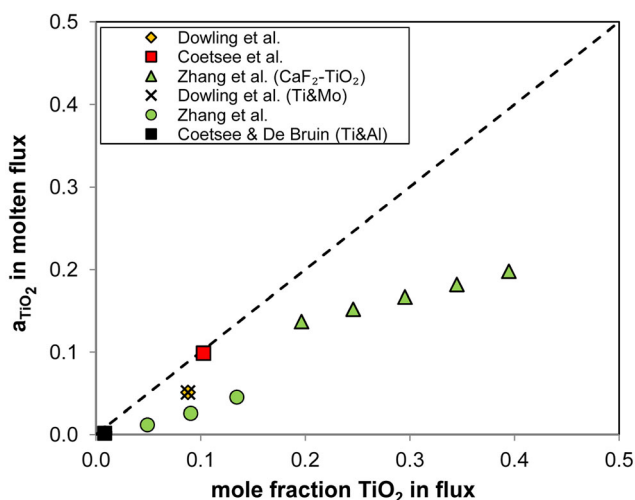


Figure 4. TiO₂ activity in molten flux at 2000 °C.

metal ppm Ti is not the highest value attained at 370 ppm Ti, at 220 ppm O.^[14] The highest Ti content is 410 ppm Ti at 150 ppm O from the 40% TiO₂–60% CaF₂ flux. Significant quantities of SiO₂ and MnO are added into commercial flux formulations to compensate for the loss of Mn and Si from the weld wire and base plate steel to the gas phase and slag phase. Therefore, the %TiO₂ in the flux formulation is limited by these required additions and flux physicochemical property requirements. Therefore, since the %TiO₂ in the flux is constrained, the activity of TiO₂ in the flux is limited by the %TiO₂ that can be added in conventional SAW.

The TiO₂ activity values in Figure 4 were used to back-test the equilibrium ppm Ti content according to Equation (2) by using 50–800 ppm O as input values. The Gibbs free energy expression for reaction (2), as displayed in Equation (4), was taken from the literature.^[27]

$$\Delta G = 646\,500 - 224.555T \text{ (J mol}^{-1}\text{)} \quad (4)$$

The ppm Ti values calculated from Equation (4) are displayed as curves of ppm O versus ppm Ti in Figure 5, with the data points from the weld tests superimposed for comparison. Since the TiO₂ activity in the molten flux does not vary significantly within the temperature range of 1800–2100 °C, as shown in Table 3, the curves in Figure 5 were calculated across the temperature range of 1800–2100 °C by using a constant input TiO₂ activity of 0.20, the maximum value in Table 3 for 2000 °C. The data in Figure 5 shows no consistent matching of weld metal

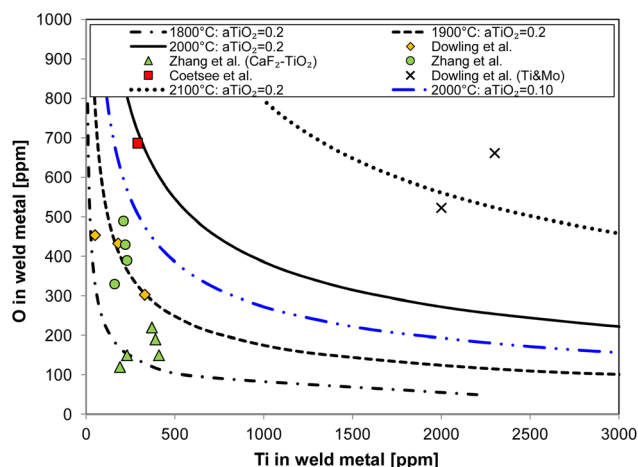


Figure 5. Ti–TiO₂ reaction equilibrium versus weld metal data.

Ti and O content with the Ti–TiO₂ slag–metal equilibrium values. Furthermore, a shift of 100 °C in equilibrium temperature significantly shifts the ppm Ti versus ppm O equilibrium curves, as does a change in TiO₂ activity from 0.20 to 0.10. The latter is shown as the blue curve in Figure 5. The thermochemical analysis results from Figure 5 show that metal–slag equilibrium, as per Equation (2), does not hold in the conventional SAW process. With the modification of the SAW process by adding Ti metal powder, the metal–slag equilibrium at 2100 °C is closest to the WM composition measurements of ppm Ti and ppm O. The data point of 4.37% Ti at 509 ppm O is not shown in Figure 5 since this point is beyond the 1% limit requirement for Henrian activity values in Equation (3).

Adding more TiO₂ in the flux formulation does not proportionally increase the Ti element transfer to the weld metal. The main reason for this effect is that TiO₂ reacts with F₂ gas in the arc cavity to form Ti-fluoride gasses. Titanium loss to the gas phase, as TiF₃ and TiF₄, works against the transfer of Ti element to the weld metal.^[13] The gas–slag–metal equilibrium model developed by Coetsee et al.^[12] as applied to the data set of Zhang et al.^[13] clearly illustrated the preferential formation of TiF₃ gas with increased TiO₂ in the flux formulation. The gas–slag–metal equilibrium model was applied successfully in calculating the weld metal ppm O and ppm Ti in conventional SAW for 2000 °C using FactSage 6.4 and for 2100 °C using FactSage 7.3.^[12,13] The latter calculation included the gas–slag–metal equilibrium for flux 5 in Table 1. The calculated gas analysis agrees in terms of the preferential formation of TiF₃ gas from the TiO₂ added to the flux.^[12]

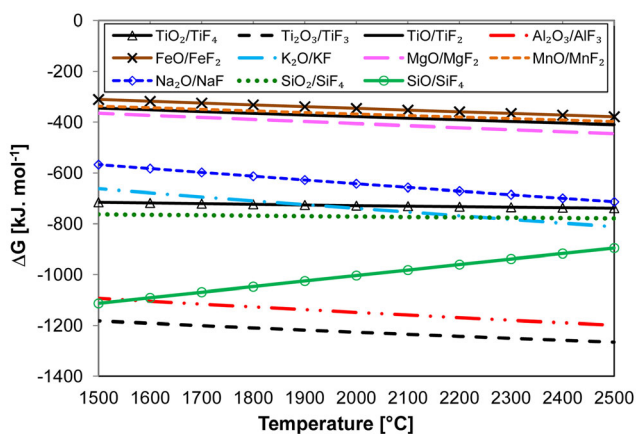


Figure 6. Oxide–F₂(g) reaction Gibbs free energy versus temperature.

A simplified thermochemical analysis of fluoride gas formation from oxides is shown in **Figure 6** in the form of Gibbs free energy lines for a reaction similar to Equation (5). The reaction module in FactSage 7.3 with FToxid and FactPS database selections were used in Gibbs free energy calculations for Figure 6. The data in Figure 6 confirms that the highest thermodynamic driving force for the typical oxides in SAW flux formulations is to form TiF₃ gas via reaction (5). This reaction is the combination of oxide dissociation in the arc cavity to release oxygen and metal, as determined in previous experimental work by Chai and Eagar^[5] and the reaction of the released metal with F₂(g) in the arc cavity. The formation of F₂(g) appears possible due to the dissociation of CaF₂ in the arc cavity since Ca and F were analyzed in the arc cavity gas phase during SAW tests made with CaF₂-based flux.^[28,29]



Although reaction (5) is written with oxygen released into the gas phase, it is unlikely that this is the only gas-phase reaction in the arc cavity. Since the source of the F₂(g) is CaF₂ added to the flux formulation, the reaction between the oxide and CaF₂ to form CaO and fluoride gas is often written in welding flux texts. Reaction (5) is a useful overall reaction description since it is well established that the flux oxides decompose at high temperatures in the arc cavity to release oxygen.^[5] This oxygen is subsequently adsorbed onto the weld wire metal droplets, and the weld wire metal droplets form the pathway of oxygen transfer into the weld pool.^[5,25,26] Therefore, adding more TiO₂ to the flux is not viable to increase the weld pool Ti content because more added TiO₂ results in more Ti loss to the gas phase as Ti-fluoride gas and also results in more oxygen released from TiO₂ decomposition in the arc cavity. The latter effect was demonstrated for CaF₂-TiO₂ flux by Chai and Eagar^[5] and confirmed in the data sets applied here.^[14,17] This trend of increased weld metal ppm O with increased flux TiO₂ content was confirmed in application of CaF₂-TiO₂-Al₂O₃ flux to achieve a maximum of 200 ppm Ti in the weld metal at 265 ppm O.^[30]

The lines in Figure 6 show that AlF₃ gas forms almost as easily as TiF₃ gas via type (5) reactions. This aspect indicates that the SAW process can be effectively modified by adding Al, as shown in several studies on the aluminum-assisted transfer of high

oxygen-affinity elements, Cr and Ti.^[31–35] The aluminum-assisted transfer of Ti with the addition of various alloying elements formed weld metals containing 4.37% Ti, 0.98% Al, 509 ppm O (as displayed in Figure 1–3) and 5.11% Ti, 5.23% Al, 371 ppm O with Cu added and 5.89% Ti, 4.84% Al, 326 ppm O with Cu and Cr added.^[16,32,34] The added Al powder lowers the oxygen partial pressure in the arc cavity and at the slag–metal interface to maintain the added Ti metal powder in the metallic state and prevent oxidation of Ti to its oxides.^[16,31–36] The alloying of the weld metal with Ti is done by Ti metal powder, and not by TiO₂ in the flux.^[16] Furthermore, as discussed elsewhere, the formation of more stable Al-fluoride gas formed from the added Al metal powder, displaces less stable metal fluoride gasses such as Ti-fluoride gas, so limiting Ti loss to the gas phase.^[35–37] The FactSage-based gas–slag–metal equilibrium model from Coetsee et al.^[12] was applied to illustrate the changes in the arc cavity gas phase when Al is added versus conventional SAW when using flux 4 in Table 1 and versus adding only Ti in SAW.^[12,16] The effect of the added Al in limiting Ti-fluoride gas formation by forming Al-fluoride gas instead is seen in the gas compositions in **Table 4** as a shift from MgF₂, CO, AlF₃, and CaF₂ as the main gas species to AlF, TiF₃, MgF₂, and Na with Ti addition to AlF, Mg, and Na with Al and Ti additions. The gas-phase oxygen partial pressure values shown in Table 4, as calculated in the gas–slag–metal equilibrium model, are lowered from 7.56 × 10^{−9} atm from the flux without added metal powders to 4.17 × 10^{−12} atm with the addition of Ti and Al metal powder. The values in Table 4 are a useful confirmation of the deoxidizer role of Al in the arc cavity, even though this model

Table 4. Gas-phase composition from gas–slag–metal equilibrium model for 2100 °C.

Compound [vol%]	Flux 4 ^[12,16]	Flux 4 + (Ti) ^[16]	Flux 4 + (Al & Ti) ^[16]
CO	13	6	2
Na	3	10	7
SiO	<0.5	2	<0.5
Mg	1	6	29
NaF	10	8	1
KF	1	1	<0.5
CaF ₂	11	3	1
MgF ₂	24	10	2
MgF	3	4	4
AlF ₃	12	7	1
AlF ₂	4	8	5
AlF	2	13	42
NaAlF ₄	7	3	<0.5
TiF ₃	1	12	1
TiF ₂	<0.5	2	1
KAlF ₄	2	1	<0.5
Mn	1	2	2
MnF ₂	1	<0.5	<0.5
Fe	1	<0.5	1
P _{O₂} [atm]	7.56 × 10 ^{−9}	1.32 × 10 ^{−10}	4.17 × 10 ^{−12}

does not adequately predict the final weld metal composition in the aluminum-assisted SAW process. An accurate prediction of the weld metal composition requires the incorporation of mass-transfer effects in the form of a timed thermodynamic model or effective equilibrium reaction zone model.^[36]

The thermochemical analysis of this varied data set shows that the element transfer of Ti from molten flux (slag) to the weld metal is limited to ≈ 400 ppm O because of the limited increase in the TiO₂ activity as set by flux formulation requirements, and the preferential loss of Ti as Ti-fluoride gas. Application of metal powders in SAW results in modification of weld metallurgy to enhance Ti transfer to the weld metal. The addition of Al metal powder as deoxidizer with Ti metal powder shifts the gas composition from Ti-fluoride formation to Al-fluoride formation and lowers the SAW process partial oxygen pressure to increase the weld metal Ti content, whilst acceptable ppm O remains in the weld metal.

4. Conclusion

1) In conventional SAW, the element transfer of Ti from the slag is limited by TiO₂ activity due to limits set on the flux TiO₂ content, namely oxide dilution by CaF₂ to control the weld metal ppm O and form the required physicochemical properties of the slag; 2) addition of Ti metal powder in SAW can overcome this element transfer limitation via the flux but does not optimize yield because Ti-fluoride gas formation causes Ti loss. 3) modification of the conventional SAW process by aluminum-assisted alloying of the weld metal can control the weld metal ppm O and negate Ti loss as Ti-fluorides in the gas phase; and 4) the added Al deoxidizer shifts the gas composition from Ti-fluoride formation to Al-fluoride formation and lowers the SAW process partial oxygen pressure to increase the weld metal Ti content, with acceptable ppm O remaining in the weld metal.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

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

Keywords

fluorides, fluxes, slags, thermochemistry, titanium, weldings

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