# Effect of different oxide fluxes on the penetration depth, microstructure and corrosion behaviour of austenitic stainless steel in A-TIG welding

Ajit Kumar Pramanick\*, Suman Modak\* and Tapan Kumar Pal#

\*Seacom Engineering College, Howrah - 711 302

#Welding Technology Centre, Metallurgical and Material Engineering Department, Jadavpur University, Kolkata-700 032

## ABSTRACT

Three single oxide fluxes and two combined oxide fluxes were used to investigate the effect of different fluxes on depth/width ratio, microstructure and corrosion resistance in type 304 stainless steel. The result showed that the type of oxide flux has a significant effect on the 304 SS weld penetration, microstructure and corrosion resistance. The uses of oxide fluxes appear to promote the formation of deltaferrite and higher oxygen content resulted fine delta ferrite. Furthermore, the corrosion resistance of austenitic stainless steel welds improved with addition of oxide fluxes.

Key Words: Oxide flux, depth/width ratio, microstructure, corrosion resistance, Gas Tungsten Arc Welding.

# **1.0 INTRODUCTION**

Improvements in weld penetration have long been sought in gas tungsten arc welding (TIG welding) as the process has relatively shallow penetration. Although the TIG welding has a good appearance and high quality, shallow penetration restrict the process for wide application. Two methods have been evolved to improve the TIG welding penetration. One is changing the raw material composition by adding some minor elements to the stainless steel, such as O, S, Se and Bi [1,2], which is of particular interest to steel manufacturers who supply the base material. The other method is from welding engineers using fluxes in TIG welding [3-6].

A – TIG was first invented in the 1960s by researchers at Paton Electric Welding Institute in Ukraine [7]. Since then the investigations of the effect of the activators on processes occurring in the arc column, behaviour of the molten metal pool, penetration and formation of the weld metal have been carried out in different countries. Many investigations of the mechanism and the application technology of the A – TIG process have been made and the two representative theories are: arc contraction [8 – 16] and the reversal of the Marangoni convection in the weld pool [11, 17 – 20]. However, there is still no common agreement about A – TIG mechanism. Even for the arc contraction, different researchers have different explanations [8]. Heipler and Roper studied on the minor elements effect on the Marangoni convection in the welding pool [17, 18, 20]. Their result showed that the active elements, such as O, S and Se, in the welding pool changed the temperature coefficient of the surface tension,  $\delta\gamma/\delta T$ , from negative to positive and hence reversed the Marangoni convection direction from outward to inward. As the convection was inward, the penetration was increased dramatically.

Together with the experimental investigation, simulation analysis of the welding pool convection and its effect on the penetration was studied by Kou, [22 - 24] T Sai, [25, 26] and Oreper [27]. The result also showed that weld pool convection by the surface tension and electromagnetic force were the main driving forces, and the active elements will change the Marangoni convection and penetration.

It is worth mentioning here that the application of flux is cost effective. The economic factors are

- Reducing of the number of passes
- Decrease in the volume of the deposited metal due to avoidance of groove preparation.
- Filler wire is not needed
- Costs associated with making, machining and repeated welding of the root weld are reduced.
- Total power and labour consumption is reduced.

Therefore, research is being continued to explore new types of fluxes which will improve performance of the weld in addition to penetration advantage. Two previous studies reported on SS – 7HP – GTAW flux for use on austenitic (316L), super austenitic (254SMO), and super duplex (SAF 2507) small diameter heavy – bore tubing for sub – sea control line applications [28,29]. In each of the alloy systems, penetration increases of over 300% when using HP – GTAW flux permitted full fusion orbital welds to be produced in 15.8 mm by 2.8 mm tube in a single pass. Consistent with previous research efforts [30] the use of HP – GTAW flux did not appear to significantly influence the microstructure or properties in austenitic or super weld metals.

However, due to slag-metal reaction in A-TIG process composition of the weld metal is expected to alter and may affect microstructure, mechanical and corrosion properties of such welds .Very little published literature is available on these aspects particularly the corrosion behaviour of weld metal. Accordingly, the focus of this investigation has been on identification of new type of flux in terms of their penetration characteristic and corrosion behaviour.

In this study, five different kinds of oxide fluxes were used in A-TIG process to examine the effect of three single fluxes and two combined oxide fluxes on the weld penetration, micro structure and corrosion behaviour of the type 304 austenitic stainless steel systematically.

# 2.0 EXPERIMENTAL PROCEDURE

A bead – on – plate weld was made on a 304 stainless steel substrate machined into rectangular plates, 100 X 50 X 5 mm, with the average composition of 0.06%C, 0.44%Si, 0.96%Mn, 8.19%Ni, 18.22%Cr, 0.027%P, 0.02%S, and the rest of Fe and the base metal microstructure is shown in **Fig 1.** A nominal 2% thoriated tungsten electrode rod of 1.6 mm diameter was

used; the electrode tip was ground to a 60° point with a small flat tip. The shielding gas of 100% argon with gas flow rate of 10 l/min was maintained constant in the present experiment.

The oxide fluxes used in the experiment were Cu<sub>2</sub>O, NiO, TiO<sub>2</sub>, TiO<sub>2</sub> + NiO and TiO<sub>2</sub> + Cu<sub>2</sub>O with the specific atomic weight and specific surface areas of different flux are given in **Table 1**. Before welding, the substrate surface was ground using 80 grit flexible abrasive papers and 50 X 5 X 1 mm slot was planed on the surface centre of the substrate as shown in **Fig.2**. The flux was manually pre – placed in the slot over a 50 mm length and uniformly dispersed with acetone.

Welding was carried using a DCEN power supply with a system (MASTER TIG AC/DC 3500W) in which the torch was carried



Fig.1 : Base metal microstructure

Table 1 : Atomic weight and specific surface area of Different Flux.

Name of the Flux	Atomic weight	[O]/mole	Specific surface area (m²/L)
NiO	75	120 X 10-5	6.6 X 103
TiO <sub>2</sub>	80	40 X 10-5	3.5 X 103
Cu₂O	143	16 X 10-5	2.7 X 103



Fig. 2 : Schematic of the 304 SS plate used in GTAW

Flux Type used	Welding Speed (mm/sec.)	Welding Current (A)	Arc Voltage (V)	Heat Input (KJ/mm)
Without Flux	4.29	166	11.22	0.217
With TiO₂ Flux	3.93	165.88	10.61	0.223
With Cu₂O Flux	3.23	166	11.2	0.287
With NiO Flux	4.43	155	11.44	0.200
With TiO <sub>2</sub> + Cu <sub>2</sub> O Flux	2.76	166	10.94	0.328
With TiO₂ + NiO Flux	3.42	165.8	11.38	0.275

Table 2 : Parameters used for TIG Welding.

manually. The time taken for each weld deposit for each flux type was noted. **Table 2** shows the welding parameters used.

After welding, the cross – section of the bead was etched by  $HCl + Cu_2SO_4$  solution and was photographed using an optical microscope (Make: Carl ZEISS India Pvt. Ltd.; Model: Imager. A1m). The etched samples were examined for oxygen content and other elements present in the weld metal by EDS. The depth of penetration and width of the weld bead was measured

with the help of AUTO Desk Software using suitable scale. Micro-hardness was taken on flat metallographic specimen across the weld using Vickers's microhardness testing machine (Make: Lieca, Germany; Model: A- 1170) using 50 gf load.

Potentiostatic polarization study was performed at the cross sections of six different weld metals, exposed to 3.5% NaCl solution for corrosion study. The potentiostatic polarization curves for weld metals were determined in 3.5% NaCl solution using AUTOLAB PGSTAT 302 Potentiostatic machine at a scan rate of 0.5 mv/s. Ag/AgCl electrode was used as reference and platinum was used as counter electrode. Taffels slopes were derived from the polarization curves.

## 3.0 RESULTS AND DISCUSSION

## 3.1 Weld Bead Penetration

Cross - section of the welding beads produced using different types of oxide fluxes were photographed. The fusion zone shapes without flux, and with different single and double component fluxes are shown in **Fig.3**. It is clear from **Fig.3** that all the five oxide fluxes used such as  $Cu_2O$ , NiO, TiO<sub>2</sub>, TiO<sub>2</sub> + NiO and TiO<sub>2</sub> +  $Cu_2O$ , attributed deep penetration compared to the weld metal produced without flux shown in **Fig.3** (a).



Fig.3 : Fusion Zone morphology with without flux and with fluxes

Name of Sample	Without Flux	With Cu2O	With NiO	With TIO2	With TiO2 + NiO	With TIO2 + Cu2O
Depth of Penetration, D (mm)	1.14	4. <b>4</b>	3.21	1.63	2.19	2.71
Bead Width, W (mm)	5.792	5.54 <del>9</del>	5.395	5.319	6.493	7.1852
Depth to Width ratio (D/W)	0.1968	0.7929	0.5949	0.3064	0.3372	0.3771
Depth of Penetration/ Heat Input	5.253	19.73	16.05	7.30	7.96	8.26

Table 3 : Weld bead geometry with and without flux of TIG weld

## 3.2 Weld D/W ratio

Based on the photograph of cross – section, the depth/width (D/W) ratio of the fusion zone was measured. **Table 3** Shows the characteristics weld geometry of the type 304 stainless steel in TIG welding produced with and without flux. It is clearly observed that D/W ratio increases for all five different types of flux composition used. However, the extent of increment differs and fluxes can be arranged according to their D/W ratio in descending order as follows: Cu<sub>2</sub>O, NiO, Cu<sub>2</sub>O + TiO<sub>2</sub>, TiO<sub>2</sub> + NiO and TiO<sub>2</sub>.

Since the welding was performed manually where variation of heat input can not be avoided, D/W ratio has been divided by the heat input used to nullify the effect of heat input and is shown in **Fig. 4**. Interestingly the trends in contributing penetration for each type of flux remains unaltered. These results therefore indicate that slight variation in heat input due to Manual A-TIG does not have any significant effect on weld penetration. In other words, the change in weld penetration is solely due to the addition of different oxide fluxes.

The behaviour of different oxide fluxes could be rationalised from their physico – chemical properties of the oxides. From the Ellingham diagram ( $\Delta G^{o}$  - T), the Cu<sub>2</sub>O oxide is least stable and will easily decompose under the arc releasing oxygen. Thermodynamic calculations of the reactions for the oxides formation are estimated under the assumption that the weld pool is considered as a Fe- M–O system. The equilibrium reactions of Cu, Ni and Ti with oxygen in the liquid iron for the oxide products such as Cu<sub>2</sub>O, NiO and TiO<sub>2</sub> are given by the following equations, respectively [18, 20, 21, 25].

# For Cu<sub>2</sub>O system

$$Cu_2O(S) = 2Cu(1) + 1/2O_2(g)$$
 (1)



Fig.4 : Depth of penetration/heat input for different oxide fluxes and without flux

$$\Delta G0 = -RTlnK = -RTln a_{\alpha z 0 (s)} / a^{2}_{\alpha z (s)} X a^{1/2}_{\alpha z} = -188300 + 88.48T$$
(2)

$$2NIO(S) = 2Ni(I) + O_2(g)$$
(3)

$$\Delta G^{\circ} = -RTInK = -RTIn a^{2}NiO(s) / a^{2}Ni(s) X$$
$$a_{o2} = -471200 + 172T$$
(4)

#### For TiO<sub>2</sub> system

$$TiO_2(s) = Ti(s) + O_2(g)$$
(5)

$$\Delta G^{\circ} = -RT \ln K = -RT \ln a_{\pi o 2(s)} / a_{\pi(s)} X a_{o 2}$$
(6)

(T=1500K, R= Constant)

Using Eqs. (1-6), the equilibrium oxygen content in the liquid iron and the oxygen partial pressure are calculated and shown in **Table 4** under 1500K.

Table 4	: Equilib	rium o	xygen	xygen content and		
	oxygen	partia	al press	ure		

Oxide Fluxes used	Equilibrium oxyger content at temp. 1500K		
Cu₂O	1.34X10 <sup>4</sup>		
NiO	3.7X10 <sup>-8</sup>		
TiO	22.89X10 <sup>-15</sup>		
TiO <sub>2</sub> + Cu <sub>2</sub> O	1.3X10⁴		
TiO <sub>2</sub> + NiO	3X10 <sup>-8</sup>		

For Cu <sub>2</sub> O	oxide	formation	reaction	Eqs.	(1),	the	equ	ilibri	um
oxygen co	ontents	in liquid	pool are h	igher	than	that	for	the I	NiO

and  $TiO_2$  oxide formation reactions (Eqs. (3) and (5)) as shown in **Table 4**. Similarly the equilibrium oxygen contents in liquid pool are higher for NiO than  $TiO_2$ . Although it was not possible to measure the oxygen content of the weld metal by Leco Analyser, which provides more accurate results, it is reasonable to predict, at least the trend based on EDS results as shown in **Fig 5**. The oxygen content in the weld metal will be higher with least stable oxide used as flux such as  $Cu_2O$  and will be lower with more stable oxide flux such as  $TiO_2$ .

The deoxidant present in the base metal such as Cr is sufficient to keep oxygen content at the initial levels. However, due to characteristic features of arc welding process, the chemical interactions between the liquid metal and slag (Flux) take place within a very short time. As a result, deoxidation reaction may not complete and for an oxygen content above the limit of solubility (Approximately 0.2 wt pct. As per Fe – O diagram [31]), deoxidation products such as SiO<sub>2</sub> and Cr<sub>2</sub>O<sub>3</sub> are possibly



Fig. 5 : Analysis of Oxygen content and other elements present in weld metals made with different flux by EDS

formed as an immiscible phase with liquid iron. During solidification under quasi – steady state conditions deoxidation products separate and incorporated in the slag. Small amounts of deoxidation may be trapped as an inclusion in the weld metal.

# 3.3 Marangoni convection in weld pool

Weld penetration in GTAW was determined by the fluid flow mode in the weld pool. The surface tension gradient on the weld pool surface is a principle variable that changes the convection mode. Generally, the surface tension decreases with increasing temperature,  $\delta v / \delta T < 0$ , for pure metal and many alloys. In the weld pool for such materials, the surface tension is higher in the relatively cooler part of the pool edge than that in the pool centre under the arc, and hence the fluid flows from pool centre to the edge. The heat flux is easily transferred to the edge and the weld pool shape is relatively wide and narrow as shown in Fig.6 (a). Heipler and Roper [18, 20] proposed that surface active elements such as oxygen, sulphur and selenium can change the temperature coefficient of surface tension for iron alloys from negative to positive,  $\delta y/\delta T > 0$ , and furthermore, change the direction of fluid flow in the weld pool as illustrated in Fig.6 (b). In such case, a relatively deep and narrow weld will be produced.

In our experiments, the oxygen content in the weld from the decomposition of the oxide flux is considered to be played the important role as an active element and is expected to change the marangoni convection mode of the liquid weld pool. Comparing the results of the  $Cu_2O$  flux with that of NiO and  $TiO_2$ , it was found that there was increased oxygen content for which D/W ratio is higher. With decreasing oxygen content the Marangoni convection probably becomes week and weld pool penetration decreases. The oxide type and behavior on the liquid pool under arc column thus play an important role in the Marangoni convection of the liquid pool.

# 3.4 Effect of oxide flux on microstructure and hardness

Careful examination of the micrographs shown in **Fig. 7** reveals that the delta ferrite grain size is significantly smaller in the welds produced with oxide flux (**Fig. 7a**). It is probable that weld metal oxygen concentration has a direct influence on the delta ferrite grain size. Small delta ferrite grain size is observed in the welds produced using the oxide flux releasing higher oxygen. Slightly larger grain size was observed in the oxide welds having lower oxygen content. Delta ferrite was also observed in the welds produced without flux. The formation of ferrite in weld metal compared to the base metal



Fig.6 : Schematic of Marangoni convection mode in welding pool.

which is free from delta ferrite is likely due to the faster cooling rate experienced in the bead - on - plate welds. However, slight change in chemical composition in weld metals due to slag-metal reaction as shown in SEM-EDS results does not affect the microstructure significantly.

**Fig. 8** presents the experimental results for the micro hardness of the TIG weld metal with and without flux. The results show that weld metals produced with the different oxide fluxes though attribute higher hardness compared to base metals, do not show any significant difference in hardness among the 304 stainless steel weld metal except weld metal formed with  $Cu_2O+TiO_2$ . This is expected as the formation of delta ferrite in austenitic stainless steel should increase the hardness. In case of weld metal with  $Cu_2O+TiO_2$ , amount of delta ferrite is very low (**Fig. 8**) resulting in lower hardness.

# 3.5 Corrosion study

Potentiostatic polarization study has been done for six different weld metals across the cross sections in 3.5% NaCl solution to evaluate corrosion behavior of different weld metals. The potentiostatic curves obtained for different weld metals are shown in **Fig.9**. Comparison of the five different welds reveals that nearly identical polarisation behaviour is obtained in each case. However, variations in corrosion rate and  $E_{\rm our}$  among the weld metals have been observed and these are evaluated as given in **Table 5**.

Weld metals made with different oxide fluxes in general show higher corrosion resistance than the weld metal made without











SI No	Weld Metal with different oxide fluxes	Ecorr (V)	Corrosion Rate (mm/Year)
1	Cu₂O	- 0.223	0.02
2	NiO	- 0.202	0.08
3	TIO <sub>2</sub> + Cu <sub>2</sub> O	- 0.186	0.1
4	TiO <sub>2</sub> + NiO	- 0.163	0.15
5	TiO <sub>z</sub>	- 0.196	0.2
6	Without Flux	- 0.183	1.116

Table 5 : Data for Corrosion rate and Ecorr values derived from Potentiostatic polarization curve

flux. Again, out of five different oxide fluxes used,  $Cu_2O$  flux shows highest corrosion resistance than the other oxide fluxes. However, the extent of corrosion resistance differs from flux to flux and fluxes can be arranged according to their corrosion resistance in ascending order as follows:  $TiO_2$ ,  $TiO_2$  + NiO,  $Cu_2O$ +  $TiO_2$ , NiO and  $Cu_2O$ .

Published information indicates that the effect of delta ferrite on corrosion resistance vary with steel composition. In austenite steels weld metals without molybdenum; delta ferrite itself does not appear to reduce corrosion resistance [32 - 34]. Furthermore, chemical change occurring in the weld metal of stainless steels can affect corrosion resistance. Again, there can also be segregation effects in the weld metal [35, 36]. In such cases, composition heterogeneity and insufficient alloy content are seen to be principal causes of attack [37, 38].

Oxidation can cause loss of chromium and stabilizing elements such as titanium from weld metal [39]. However, the solid Cr<sub>2</sub>O<sub>3</sub> having melting point of 2538K should form at the periphery area on the weld pool surface as the peak temperature of the pool should be in the range of 2200K -2500K [40]. It has been reported that higher Cr-oxide increases the passivity of the bulk alloy system by reducing the dissolution rate in the chloride medium [41]. Also the film thickness of the passive oxide film increases with the anodic potential. Other elements such as Cu, Ni etc which are being picked up in the weld metal due to slag- metal reaction from their respective oxides in A -TIG process, should take part in inhibiting corrosion as well. Several authors [42, 43] have pointed out that the mechanism of copper's beneficial effect is based on the suppression of anodic dissolution by elemental copper deposition on the steel surface immersed in the corrosive medium. For instance, some authors [44, 45] have established that Cu improves pitting corrosion resistance through redeposition of previously dissolved copper. According to these studies, Cu diminishes the current density of dissolution once the pitting potential has been reached and reduces the corrosion rate as a consequence of the passivation of the active pits caused by the enrichment of the stainless steel surface with metallic Cu. Further, Hermas et al. [46] concluded that Cu additions diminish the corrosion rate and the stainless steel dissolution in diluted acid chloride solutions as a result of the accumulation of metallic Cu on the surface and the later formation of CuCl, which protects the oxide film. Similarly the beneficial effects of nickel on the increase of the passive region was also observed by Tavara et al. [47] to have resulted from an enrichment of nickel in the passive film.

These results indicated that  $Cr_2O_3$  played an important role in addition to the microstructure (delta ferrite) and other alloying elements present such as Cu and Ni transferred from slag. With increasing oxygen content more amount of  $Cr_2O_3$  is expected resulting in improved corrosion resistance. This agreed well with the results on corrosion study as the weld metal made with Cu<sub>2</sub>O having higher oxygen content showed highest corrosion resistant. However, the least corrosion resistance in weld metal made with TiO<sub>2</sub> flux could be due to the fact that apart from lower oxygen content, Ti oxides which may be contaminated with molten pool, acted as a non-metallic inclusion in the corrosive environment and the interface between the Ti-oxide and the matrix is the preferential site for pitting corrosion [48].

#### 4.0 CONCLUSIONS

- In GTA welding, type of oxide flux has a significant effect on the 304 SS weld penetration. The depth/width ratio increased with the type of flux as TiO<sub>2</sub>, TiO<sub>2</sub> + NiO, Cu<sub>2</sub>O + TiO<sub>2</sub>, NiO and Cu<sub>2</sub>O.
- The oxygen in the weld pool from the decomposition of flux is considered to be responsible for changing the Marangoni convection direction resulting in deep penetration. Higher oxygen in the weld increased the depth/width ratio.
- The uses of oxide fluxes appear to promote the formation of deltaferrite and higher oxygen content resulted fine delta ferrite.
- 4. The corrosion resistance of austenitic stainless steel welds improved with addition of oxide fluxes. The improvement of corrosion resistance with fluxes such as  $TIO_2$ ,  $TIO_2$  +  $NiO_2$ ,  $Cu_2O$  +  $TiO_2$ , NiO and  $Cu_2O$  is in that order.

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