Effect of Acetone/Isopropanol as Solvent TiO$_2$ Active Flux on Penetration Characteristics Activated Tungsten Inert Gas Welding Incoloy 825

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Abstract. The purpose of this study was to investigate the effect of flux solvent on the depth-to-width ratio (DWR), microstructure, and the hardness of Incoloy 825 with activated tungsten inert gas (A-TIG) welding process. Effect of solvents on active flux of TIG welding for geometry characteristics were investigated by metallographic and defense tests. The arc constriction and Marangoni convection are considered as the two main factors for increasing the penetration of the A-TIG weld pool. Marangoni convection provided change to fluid flow in the anode area of welding it formed a thermionic electron condition because of the large positive ion collisions in the welding arc. This study investigated the effect of acetone/isopropanol as a TiO$_2$ active flux solvents on A-TIG incoloy 825 autogenous welding. Metallography observations carried out give the best results on the addition solvents of 40% isopropanol can provide DWR 0.549 while 100% acetone solvent has DWR 0.321 and for 100% isopropanol solvent has a much smaller DWR which is 0.276. In addition to providing penetration rates, this isopropanol have an effect increasing of hardness especially in the fusion line at 40% isopropanol 198.79 HV and 100% isopropanol 202.52 HV.

1. Introduction

Incoloy 825 is alloy Ni–Fe-Cr that is widely used because of its good corrosion, oxidation resistance, and creep resistance. The alloy has good mechanical properties and presents the desirable combination of strength and toughness. The high nickel content of the alloy enables it to resist corrosion caused by many organic and inorganic compounds and also gives it high resistance to chloride ion stress corrosion cracking. The chromium content provides a resistance to sulfur compounds and to various oxidizing environments at high temperatures or in corrosive solutions [1, 2]. Tungsten Inert Gas (TIG) welding is one of the mainly applied welding processes in industry to stainless steels and nonferrous metals and its alloys such as aluminum, magnesium, and Ni-based alloys for high quality weld and low investment. Basically, TIG weld quality is strongly characterized by the weld pool geometry. This is caused the weld pool geometry plays an important role in determining the mechanical properties of the weld [3, 4].

Activated Tungsten Inert Gas (A-TIG) welding is one of the novel approaches in the fusion welding techniques which utilizes the flux layer, a mixture of inorganic material suspended in a volatile medium and produces cloud flux [5]. This cloud was able to capture the electrons from the outer sections of the arc column, since these electrons were relatively weaker, with lower kinetic energy for which these requirements give rise to electron thermionic emission requirements. This contributes to a smaller
diameter arc, with a significantly higher energy density [6, 7]. The activated flux effect be used in A-TIG is related to the surface tension force that will be formed in the weld pool. Surface tension gradient becomes positive be used activated flux and hence fluid flows radially inward into the weld pool. Initial studies on this topic showed that surface activated elements like sulfur and oxygen affect the penetration positively. Similar type of study done by other researchers, and reported increased penetration depth be used A-TIG, the same was attributed to reversal of Marangoni convection in the weld pool [6, 8, 9, 10].

In this study, the addition of isopropanol to dissolve TiO$_2$ into the acetone to improve the thermionic emission conditions of electrons which can be attributed to the cloud flux that can improve penetration geometry formed in the welding process be used A-TIG, in order to increase weld penetration depth and reduce the size of the penetration beads formed.

2. Experimental procedure

2.1 Base metal

Incoloy 825 is a nickel alloy which is one kind of austenite steel that has a crystalline structure Face Centered Cubic (FCC) and has phase amplifier gamma prime (γ'-Ni$_3$ (Al, Ti)) or gamma double prime (γ''-Ni$_3$ (Nb, Ti, Al, Mo)) [11, 12, 13, 14]. This alloy have stabilizer elements of Ni, Fe, Cr, Mo, and Ti with excellent resistance to sulfuric, phosphate acids also acid oxidants, pitting corrosion, stress corrosion crack and intergranular corrosion [9, 14].

| Material    | Chemical Composition (wt%) |
|-------------|----------------------------|
| Ti          | Cr | Mn | Fe | Ni | Cu | Mo |
| Incoloy 825 | 1.10 | 22.02 | 0.37 | 34.63 | 46.21 | 1.67 | 3.37 |

In order to investigate the composition, chemical composition testing was carried out using Positive Material Identification (PMI) S1 Sorter SRT1160 method. nickel alloy specimens were prepared for the A-TIG welding process with a test coupon marking.

2.2 Solvent and activated flux

Solvents are one of the determining factors for activated fluxes that have useful properties can determine the results of homogeneous distribution of layers due to solvent chemical properties. As the level of solvent viscosity will determine the strength of the mixture which will have an impact on the distribution of homogeneous layers. Solvent vapor pressure will determine the volatile power, which is to accelerate the drying time of the coating flux on the base metal at ambient temperature [4].

| Chemical Properties          | Acetone | Isopropanol |
|------------------------------|---------|-------------|
| Vapor Pressure (25°C, kPa)   | 30.8    | 6.02        |
| Viscosity (mPa.s)            | 0.30    | 2.04        |
| Density (g/mL)               | 0.7845  | 0.7809      |
| Molecular Weight (g/mol)     | 58.079  | 60.095      |
| Moment Dipole (D)            | 2.88    | 1.56        |
| Thermal Conductivity (25°C, W/mK) | 0.169 | 0.135       |
| Dielectric Constant          | 21.01   | 20.18       |

Cleaning the surface of the Incoloy 825 with acetone is one of the most common procedures for determining research result. After that, the coated of activated flux is done by repeating 3 times be used
a brush to maintain the same layer on various variables additional of isopropanol to the base metal Incoloy 825. Activated flux TiO$_2$ be used in this study is EMSURE® Reag. Ph Eur Titanium(IV) Oxide identified by Merck with number CAS 13463-67-7 which oh these product does not explain what phase is formed. The characterization of Titanium(IV) Oxide was conducted to find out the phase formed be used X-Ray Diffraction (XRD) X’Pert PANanalytical Cu Kα.

![Figure 1. XRD Titanium(IV) oxide](image)

Based on XRD characterization TiO$_2$ as shown in Figure 1, these results suggest that anatase phase because it has a pattern (101). Activated flux TiO$_2$ provided in the form of powder which is then weighed with Mettler Toledo MS204S and this study has solvent variables used were 100% acetone, 10, 20, 30, 40% isopropanol in the acetone and 100% isopropanol to dissolve TiO$_2$.

![Figure 2. Coated sample of TiO$_2$ activated flux paste](image)

In these variables are used the mixing ratio of ± 15 gram mass of activated flux TiO$_2$ and 30 mL solvent to dissolve TiO$_2$ which has become a variable in calculating the volume of solvent for the content percentage for the addition of isopropanol. For the mixing ratio of flux with solvent to dissolve activated flux it is not very important as long as it can be coated on the anode or base metal [4, 5]. The activated flux TiO$_2$ coated on the Incoloy 825 has a width of about 10 mm as shown in Figure 3 and produces a thickness approximately 130 µm tested using Eddy Current Testing (ECT) thickness test.
2.3. Welding procedure
The welding process to be used in this study is the autogenous TIG with the A-TIG welding technique and be used DCEN polarity, this welding process does not use a filler electrode with single-pass mode. Autogenous welding is carried out along the lines of the test specimens to produce penetration geometry along with the morphological results of weld capping.

The electrodes to be used in this welding use Tungsten electrodes with a mixture of Thorium oxide 2% or known as EWTh-2 electrodes. The electrode uses a conical shaped tip or commonly referred to as the vertex with an angle of 60°. Shielding gas uses Argon UHP gas with a purity level of 99.999% at a constant flow rate of 15 l/minute. Welding current be used of 140 A on all variables with output which of course the heat input will be the same for all the variables of addition of isopropanol to solvent activated flux TiO₂.

3. Result and discussion

3.1. Effect of solvent on geometric shape of weld
The autogenous A-TIG welding with the Incoloy 825 produced a fine beads geometry. Then the samples were etched in a 10% oxalic acid by electrolytic using 6 V DC, and current density of 1.6 A/cm² after the processes of rough grinding, fine grinding, and polishing. This provides evidence that there is an increase in penetration depth and an increase in Depth-to-Width Ratio (DWR) value in the variable that adds isopropanol to solvent acetone which is be used to dissolve the activated flux TiO₂ as shown in Figure 3. The increase in the DWR value can be attributed to the narrowing of the arc and the mode of fluid flow in the weld pool which is influenced by Marangoni convection, Lorentz force, Buoyancy force, and aerodynamic drag [6, 10].

![Figure 3. Penetration value A-TIG autogenous against addition of isopropanol](image_url)

The higher the depth of penetration of A-TIG welding can be due to the effect of narrowing of the arc. The activated flux arc constriction effect is also related to the evaporation of re activated elements such as Oxygen or Sulfur found in activated flux and preferential ionization, this can increase current density at the anode and arc force acting in welding [16]. Fluid flow that is directed inward at the weld pool is intended to form a beads that does not widen and deeper when be used activated flux. This is due to the fact that the change in surface tension gradient from negative becomes positive, while be used activated flux it will result in a reversal of Marangoni convection which is a fluid flow that forms centripetal and hence the resulting penetration is smaller and deeper [6, 10, 17]. The electrode tip tip angle is also a parameter that can affect penetration depth and DWR beads geometry results in the TIG welding process, but in the results of this study be used a vertex angle electrode tip or conical which is...
considered to have satisfactory results for penetration depth and forming DWR beads geometry and DWR factors an important role in determining the quality of the welding joint [10].

The penetration geometry observations formed DWR as shown in Figure 4 and explains various variables be used in this study provide a DWR which is increasing with the solvent addition of 10, 20, 30 and 40% isopropanol in the acetone. However, the use of 100% isopropanol solvent has a DWR result that is far from the other, that is this variable gives a DWR value of 0.276. This proves that when be used 100% isopropanol solvent is not recommended without other solvent combinations.

![Figure 4. DWR Penetration A-TIG autogenous against addition of isopropanol](image)

The result of the high viscosity of isopropanol is 2,04 mPa.s so it is quite difficult to completely dissolve the activated flux TiO₂ into a paste coated to the base metal because the mass density of each unit area is higher so that the autogenous welding current be used 140 A is possible not strong enough to do a maximum penetration due to the weak arc welding formed in the weld column.

3.2. Interface and weld microstructure

Observation of microstructure in the welding area shows that epitaxial grain growth has been seen in the fusion line area, because the welding process carried out by the Incoloy 825 base metal uses the autogenous welding method [18, 19]. This epitaxial mechanism can be seen from the results of the microstructure in all A-TIG variable processes in various variables of addition of isopropanol solvent.

![Figure 5. Microstructure A-TIG autogenous welding solvent 40% isopropanol](image)

This epitaxial mechanism can be seen from the results of the microstructure on A-TIG in various variables of addition of solvent isopropanol. Autogenous TIG welding observations using optical microscope with 200X magnification on the fusion line area were identified as shown in Figure 5.
containing columnar dendrite due to solidification behavior of the weld pool. During the solidification process the grains will grow toward the weld center due to the high thermal gradient effect on the weld interface from the center of the weld pool and this supports the growth of columnar dendrite [1, 11]. In the same way because the cooling process is fast and the thermal gradient is lower in the center of the weld pool, equiaxed dendrite is formed in the weld center and it is also found that deposited microstructure addresses a homogeneous cellular structure that grows epitaxial along the weld direction. The columnar dendrite growth is come from the fusion line area interface. This direction of growth of columnar dendrite grain is perpendicular to the fusion line with a flat shape extending to the beads weld interface and will expand its flat shape towards the center of weld metal.

3.3. Hardness of the weldments

The hardness of weld areas was measured by an DMTH FLC50UC type digital Vickers micro hardness tester with method SNI 8390:2017 standard which refer to ASTM E384:2005. This tested provides five different points in the welding area with two transverse lines. From the results of Vickers micro hardness it has been shown that welding be used the A-TIG process almost all variables in this study show that fusion lines have the highest distribution of hardness as shown in Figure 6. In the 100% acetone has the average hardness value at 176.08 HV fusion line while in the weld metal has a value of 181.88 HV and the 100% isopropanol has a hardness value of 202.52 HV in the fusion line while 204.14 HV for weld metal.

![Figure 6. Distribution of hardness A-TIG welding Incoloy 825](image)

For variables that provide the best DWR value, namely in the variable 40% isopropanol has an average hardness of 198.79 HV on the fusion line while 187.90 HV for weld metal. From the results of Vickers micro hardness testing it can be concluded that the addition of isopropanol solvent in the acetone has influenced the level of hardness formed in the solidification rate that can determine the hardness value. Therefore, hardness which has a high value on the fusion line can be associated with the formation which is formulated by the formation of the columnar dendrite solidification. The microstructure formed in the results of autogenous welding has several kinds of solidification that is the columnar dendrite structure shows a higher hardness value than the equiaxed structure which is present in many fusion line areas [5]. In addition, the hardness in this fusion line area can be associated with phase precipitation in the dendrite and interdendrite regions of weld metal.
4. Conclusion
After analyzing the results of the study, it can be concluded that the addition of solvent isopropanol in the acetone to dissolve TiO$_2$ active flux which is used to strengthen the reversed Marangoni convection in weld pool fluid flow or weld arc constriction can increase penetration DWR values however, it is not the case when using 100% solvent isopropanol. This can be caused from very concentrated or high concentrations of active flux paste coating on the Incoloy 825 so that it can result in weakening of the weld arc to penetrate the TiO$_2$ active flux paste layer. A-TIG welding has a microstructure perpendicular to the fusion line with cellular solidification and columnar dendrite. The existence of this cellular solidification due to temperature gradients higher than the grain growth rate in other words solidification has a rapid cooling rate, and for columnar dendrite is the same as cellular but the cooling is slower. The addition of isopropanol in the acetone has the effect of increasing the value of hardness especially on the fusion line resulting from cellular solidification and the formed columnar dendrite.

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