Influence of heat treatment on microstructure and mechanical properties of Inconel-625 clad deposited on mild steel

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The present work investigates on the microstructure and mechanical properties of Inconel-625 clads deposited through tungsten inert gas on mild steel (MS). The microstructure of the deposited clads reveals perfect metallurgical bonding with the substrate. The deposits are dense with almost negligible porosity. To enhance the mechanical properties, Inconel-625 clads are subjected to three different post heat treatments for 4 h. Compared to the as-deposited clad, the microstructure at 650°C heat treated specimens reveals no dissolution of the Laves phase. However, the 850°C and 950°C heat treated samples exhibit partial dissolution and complete dissolution of the Laves phase in the fusion zone, respectively. The 850°C heat treated clads exhibit higher microhardness compared to the other clads (as-deposited, 650°C and 950°C heat treated clads). This is attributed to the precipitation of γ″ strengthening phases due to availability of more Nb in the fusion zone microstructure.

Keywords: Inconel 625, TIG cladding, Microstructure, Heat treatment, Laves phase

Mild steel (MS) is one of the most widely used engineering material due to its adequate strength, weldability, plasticity, and low cost. However, mild steel has poor corrosion and wear resistance. On the other hand, Ni-based alloy, Inconel-625, has been widely used in high temperature applications, such as aerospace, heat exchangers, electrochemical industry, industrial boilers, gas and steam turbines, petrochemical, chemical and bellow material of nuclear power plants due to excellent resistance to high temperature corrosion during prolonged exposures to aggressive environments. In nickel based superalloy, Ni and Cr provide the resistance in the oxidizing environment. The presence of Cr continuously passivates the external surfaces of Inconel-625 by forming Cr2O3 and thus providing resistance to corrosion. The strengthening in Inconel-625 is due to the solid solution strengthening effect of the refractory metals, Mo and Nb in the Ni-Cr matrix. This alloy is also strengthened due to precipitation of the Ni3(Al,Ti,Nb) coherent precipitates abbreviated as γ', metastable Ni3Nb phase abbreviated as γ″ and the blocky MC (M indicates Nb, Mo, Ti) carbides particles in the Ni-Cr matrix. The γ″ has a body centered tetragonal DO22 structure. The meta-stable precipitates γ″ (Ni3Nb) are formed in the Inconel-625 after annealing over a long period in the temperature range 550-850°C.

Therefore, Inconel-625 can be suitably utilized to enhance the surface properties of MS. In the present work, the Inconel-625 clads were deposited on MS using the TIG cladding technique. The TIG cladding offers stable function, easy operation, low cost, non-oxidation process due to use of inert gas (Ar or He), safety and stable energy concentration. Thus, high quality clad layer can be deposited using the TIG arc. The high quality clad possesses high wear resistance, hardness, corrosion resistance and other improved properties. The deposited clad also provides protection to the engineering components over the service life and allows for the integrity of the base material without degradation of the wall strength. However, the formation of Laves phase is inevitable due to segregation of high concentration refractory elements such as Nb, and Mo in the interdendritic regions in the cast and welded processes of Inconel-625. Laves phase is a brittle intermetallic compound represented as (Ni, Cr, Fe)2 (Nb, Mo, Ti). Presence of Laves phase was reported to be detrimental to the weld mechanical properties, such as fatigue, tensile ductility and creep.
rupture properties. The previous investigations reveal that the distribution, morphology and volume fraction of Laves phase is highly dependent on segregation of high concentration refractory elements, such as Mo, and Nb in the interdendritic regions during weld metal solidification. The segregation of high atomic diameter elements such as Nb is easy due to non-equilibrium solidification condition prevails during the TIG cladding process. The Nb concentration in the Laves phase varies from 10 to 30%. A lot of work has been reported to control the formation of Laves phase by reducing the segregation of Nb in the interdendritic regions. The Laves phase can be controlled by reducing the Nb content in the base metal or filler metal. It has been reported that in the Inconel-718 cast material having lower Nb contents (4%) were easily homogenized compared with those having more Nb contents (5%), which required extended homogenization treatments. Further, the replacement of Nb by tantalum (Ta) and iron (Fe) with the optimum level of cobalt has been suggested to reduce the tendency for the formation of Laves phase in Ni-based superalloy. A churning effect similar to the case of EBW oscillation technique can be applied in GTA welding using magnetic arc oscillation for reducing the segregation of Nb in the interdendritic regions. Once the Laves phase is formed in the matrix, they can be dissolved back into the matrix by using suitable heat treatment. Therefore to improve the mechanical properties, the deposited clads were further subjected to three heat treatments at 4 h at 650°C, 850°C and 950°C, respectively and were allowed to cool in the furnace. Further, the developed clads were characterized using various characterisation techniques to correlate the microstructural features with their mechanical properties.

Experimental Procedure

Cladding of Inconel-625 was carried out on mild steel (MS) samples using TIG arc. Commercially available Inconel-625 wire (ERNiCrMo-3) with a diameter of about 2.4 mm was used as a clad material. The average chemical composition of the filler rod as provided by the supplier (in wt%) was 64.22 Ni, 22.59 Cr, 8.71 Mo, 3.52 Nb, 0.01 C, 0.2 Ti, and 0.18 Al. The MS plates were used as the substrate. The plates were machined to a size of 150×100×5 mm³. The MS plates were cleaned thoroughly ultrasonicated in an acetone bath prior to the deposition of the clads. The optimized parameters used for cladding was electric voltage 14 V and operating current of 112 A. An argon gas having purity 99.99 % at 16 L/min was also used as shielding gas to avoid the Inconel-625 wire and substrate from oxidation during cladding. The developed clads were further subjected to different heat treatment for improving its microstructural properties by precipitation of strengthening phases in the matrix. The as-deposited samples were post heat treated at 650°C, 850°C and 950°C in a muffle furnace with a 4 h hold time and then slow cooled in a furnace up to room temperature.

Characterization of the samples

The various phases present in the as-deposited and heat treated clads were analyzed using X-ray diffraction (XRD) with Cu-Kα radiation in scan range of 30° to 120°. The cross-sectional microstructure features of the clad specimens (as-deposited and heat treated) was investigated using optical and electron microscopy. The specimens were etched with an acid solution (acetic acid: 10 mL, hydrochloric acid: 10 mL and nitric acid: 10 mL) to reveal the microstructural features. The elemental composition of the clads specimens was investigated by energy dispersive spectrometer (EDS) on a FEI scanning electron microscope. The microhardness on the clad specimens was evaluated on Vicker’s microhardness tester (model: Economet VH1 MD, make: Chennai Metco) at a load of 50 g load applied for 15 s.

Results and Discussion

XRD results

The XRD spectrum of the as-received Inconel-625 filler wire shows the presence of major peaks corresponding to 20 value of 43.38°, 50.59°, 74.45°, 90.28° and 95.8° (Fig. 1a). In comparison to this, the peak positions of the pure Ni-based were observed at 20 value of 44.5°, 51.9°, 76.45°, 92.5° and 98.5°. The Ni peaks were observed at a slightly higher 20 value compared to as received Inconel-625 wire. This is due to the presence of the alloying elements in the Inconel-625, which significantly affects the inter-planar spacing of the γ-nickel present in the filler. The XRD spectra of the Inconel 625 clad in the as-deposited and heat treated conditions are shown in Fig. 1b. The as-deposited clad shows the presence of γ (200) phase corresponding to 20 value of 50.897° and heat treated samples shows the presence of γ (200) phase corresponding to 20 value of 50.702°. Thus, there is a slight shift in the peaks of the heat treated samples compared to as-deposited Inconel-625 clad. The peaks
of γ matrix and γ' phase match with each other because they possess similar lattice constants and a small lattice misfit\textsuperscript{11}. Li \textit{et al.}\textsuperscript{12} reported that the in case of Ni-based superalloy capturing of precipitates from the XRD diffraction is too difficult. However, the changes or shift observed in the XRD pattern of γ matrix could be used to indicate indirectly the presence of strengthening precipitates in the matrix\textsuperscript{2}. The shift or changes observed in the peaks indicate the precipitation of strengthening phases occurred in the heat-treated samples\textsuperscript{13}.

**Microstructural characterizations**

The optical macrograph of the as-deposited clad specimens is shown in Fig. 2. The dilutions and geometry size of the clad layer is illustrated in Table 1. The dilution of the cladding area is to be calculated by the ratio of the cladding layer area to the total area of the molten metal. The dilution percentage was calculated using the following formula\textsuperscript{14}:

\[
\text{Dilution (\%)} = \frac{A}{(A+B)} \times 100
\]

The dilution of the clad layer was found to be about 41.86%. The clads show good metallurgical bonding with the substrate and it is about 2.5 mm thick (reinforcement). The optical micrograph of the as-deposited clad is shown in Fig. 3. The columnar dendritic microstructure was formed in the clad zone, which grew epitaxially from the base material near the fusion boundary zone (FBZ). The FBZ is a partial melted zone and it is formed between the heat affected zone (HAZ) and the deposited Inconel-625 clad. In TIG cladding, the base material (MS) acts as a heat sink. Thus, during solidification of the melt pool,
cooling occurs mainly through the base material. Thus, grains grow in a direction opposite to the heat flow direction and it results in the formation of columnar microstructure in the fusion zone. The low porosity of the clad was due to high heat input characteristics inherent in the TIG welding process. The TIG arc helps in perfect metallurgical bonding of the clad with the substrate through complete melting of the filler wire and melting of the interfacing surface of the MS substrate. The bonding between the substrate and the overlay material (Inconel-625) is perfect with an obscure boundary as shown in Fig. 3. The optical micrograph of the fusion zone of the clad at higher magnification at various conditions is shown in Fig. 4. The SEM micrograph of the Inconel-625 clads at different heat treatment conditions are shown in Fig. 5 and their corresponding fusion zone at higher magnification are shown in Fig. 6. The microstructure of the as-deposited Inconel-625 clad shows the precipitation of Laves phase and MC carbide in the interdendritic regions (Figs 4 and 6). In TIG cladding, the solidification in the Inconel-625 materials begins with the primary liquid→ γ reaction, resulting in the enrichment of Mo, Nb, Ti and C elements in the grain boundaries and interdendritic liquid. Thus, precipitation of carbide particles (TiC and NbC) and Laves phase occurred in these regions. Later, the subsequent L→ (NbC + γ) eutectic reaction consumes most carbon available until another eutectic reaction L→ γ + Laves occurs, completing the solidification process\textsuperscript{15}. The Laves phase consumes significant amount of strengthening elements, mainly Nb, and Mo originally dissolved in the base material, thus reducing the Nb for the precipitation of principal strengthening
phase $\gamma''$-$\text{Ni}_3\text{Nb}$ in the matrix. Compared to the as-deposited Inconel-625 clad, the microstructure in the 650°C post heat treated (650HT) clad varies insignificantly, i.e., the morphology of interdendritic Laves phase and size of dendrites formed remained same as in as-deposited Inconel-625 clad (Figs 4 and 6). Thus, the 650°C post heat treatment does not dissolve any Laves phase in the matrix except the precipitation of strengthening phases. With the increase in post heat treated temperature, the Laves phase starts dissolving in the matrix. The 850°C post heat treated (850HT) clad samples indicate partial dissolution of the Laves phase. On the other hand, the 950°C post heat treated (950HT) samples exhibited higher extent of the dissolution of the Laves phase and almost uniform and homogeneous microstructure was formed after this treatment (Figs 4 and 6). Thus, it has been found that the extent of micro segregation was not significantly changed from the as-deposited condition to heat treatment temperatures up to 850°C, but decreased substantially at 950°C and an almost uniform microstructure was formed after 950°C heat treatment. It has already been reported that during solidification of nickel based alloys the distribution of a particular element depends on the value of $K$ ($K = C_s/C_l$; where $C_s$ represents the solid composition and $C_l$ indicate the liquid composition at a particular temperature). If the value of $K$ for a particular element is less than 1, then the amount of this element in the solid phase is lower compared to the liquid one. If the value of $K$ is greater than 1, the amount of the element in the solid phase is more compared to liquid phase and the segregation process proceeds towards the opposite direction. In Inconel-625, the Nb, Mo, and Si elements have value of $K<1$, and thus these elements segregate to interdendritic regions, and Ni, Fe, and Cr has value of $K>1$ and these elements segregates into the dendritic core. The value of $K$ for Nb ($K_{\text{Nb}} = 0.5$) is significantly higher than the value of $K$ for Mo ($K_{\text{Mo}} = 0.86$). Thus Nb has the strongest tendency for segregation in the interdendritic regions compared with Mo. Thus Nb enriched Laves phase formed during Inconel-625 cladding can be dissolved by using suitable heat treatment.

**Elemental distribution**

The elemental distribution of the different phases present in the clad zone was investigated through EDS.
There are mainly three phases present in the clad zone; matrix, white phase and black particles as shown in Fig. 6b. The elemental distribution was carried out these phases and corresponding EDS spectra are shown in Fig. 7. From EDS spectra, it has been found that there is enrichment (Nb, Mo, Ti) and depletion (Ni, Fe, Cr) of the elements on white phase; the results confirm that the white phase were Laves phase, which were formed during the segregation of Nb, Mo and Ti in the interdendritic regions at high temperature. The results show that the matrix was enriched in Ni, Fe and Cr and depleted by Nb, Mo and Ti. The black particles were Nb and Ti enriched carbide particles depicted from Fig. 7. Further to determine the distribution of elemental across the clad substrate zone a line scan (AB) was used as shown in Fig. 7d for the as-deposited Inconel-625 clad. The distribution of elements across the line scan area was superimposed as shown in Fig. 7d and its corresponding higher magnification is shown in Fig. 7e. It is clear from Fig. 7e that there is a diffusion of elements occurred across the clad-substrate interface. The Fe shown in red color line has a higher amount in the substrate and it reduces towards the clad zone.

Microhardness study

The microhardness measurement across the clads for different heat treatment conditions were carried out using Vickers microhardness tester at a load of 50 g for a dwell time of 15 s. The reported values were average of 10 measurements, as shown in Fig. 8. It is clear from Fig. 8 that the heat treated clads exhibit higher microhardness due to precipitation of strengthening.
phases after heat treatment (Fig. 8). The 850°C heat treated clad has higher microhardness followed by 950°C heat treated clad followed by 650°C heat treated clad and then followed by as-deposited clad. The heat treatment releases Nb and Mo and thus results in increase in solid solution strengthening and precipitation hardening (γ', γ'') of as-deposited Inconel-625 clads. The hardness of sample increase in case of 650°C and 850°C heat treated sample due to precipitation of meta-stable coherent γ'' (Ni₃Nb) in the austenite matrix. At 950°C the hardness of the deposited clad decreases due to formation of orthorhombic intermetallic incoherent δ (Ni₃Nb) precipitates in the fusion zone microstructure. The incoherent particles do not increase the hardness of the matrix.

Conclusions

In the present work, TIG arc was used for depositing Inconel-625 clads on mild steel. The as-deposited clads specimens were further heat treated at different temperatures to improve its microstructure and mechanical properties. The following conclusions are drawn from this work:

(i) The Inconel 625 clad was successfully deposited on mild steel using the TIG arc. The deposited clads has good metallurgical bonding with the MS with no evidence of defects or cracks.

(ii) The columnar dendritic microstructure was developed in the as-deposited Inconel-625 clad. The heat treated clads exhibit better microstructure and mechanical properties due to precipitation of strengthening particles.

(iii) Compared with as-deposited samples, the 650°C heat treatment did not dissolve any Laves phase, 850°C heat treatment slightly dissolve Laves phase, and 950°C heat treatment resulted almost complete dissolution of the Laves phase in the clad layer microstructure.

(iv) The 850°C heat treated clad has higher microhardness followed by 950°C heat treated clad and then followed by 650°C heat treated clads compared with as-deposited Inconel-625 clads.

References