Microwave cladding of Inconel-625 on mild steel substrate for corrosion protection

Gurbhej Singh¹, Hitesh Vasudev², Amit Bansal¹, Sachit Vardhan³ and Shubham Sharma⁴

¹ Mechanical Engineering Department, CT University, Ferozepur Road, Sidhwan Khurd, 142024-India
² School of Mechanical Engineering, Lovely Professional University, Phagwara, 144411-India
³ Mechanical Engineering Department, IKGPTU, Jalandhar, 144603-India
⁴ Mechanical Engineering Department, CT University, Ferozepur Road, Sidhwan Khurd, 142024-India
⁵ CSIR-Central Leather Research Institute, Regional Centre for Extension and Development, Jalandhar, Punjab, 144021-India

E-mail: gurbhejsingh612@gmail.com, hiteshvasudev@yahoo.in, amit.bansal978@gmail.com, sachit.mechanical@gmail.com and shubhamsharmacsirclri@gmail.com

Keywords: superalloys, microwave cladding, metals and alloys, inconel-625, corrosion

Abstract
In the present work, Inconel-625 cladding was performed on a mild steel (MS) substrate through a cost-effective microwave technique. Cladding was performed in an industrial microwave oven operated at 1.2 kW and 2.45 GHz. The XRD observation indicates the presence of Laves and carbide phases in addition to a Ni-Cr-Fe based face-centered-cubic (fcc) matrix in the fusion zone microstructure of the clad specimen. The microstructural characterization shows a homogenous and dense microstructure with a porosity value of less than 1%. The columnar dendrites grew perpendicularly to the substrate was observed in the clad zone microstructure. The average microhardness of the deposited clad was significantly higher than that of the base metal (MS), indicating improved surface properties of the base material. Further, corrosion testing indicates that the clad specimen has significantly higher corrosion resistance than that of the base metal (MS) in a 3.5 wt% NaCl solution.

Introduction
Surface engineering is the most widely used technique for improving the surface properties and functionality of a material without changing its bulk properties. This technique is beneficial tool for modifying a material’s surface to yield surface properties that are significantly different from the material’s bulk properties [1]. Protection against corrosion for ferrous alloys (mild steel, stainless steel) used in the oil and gas industry is a critical field of research. Mild steel is the most commonly used material in engineering applications; but it has very poor corrosion resistance. The surface properties of a ferrous alloy can be modified through various methods, such as heat treatment, PVD, thermal spraying, epoxy coating, enamel coatings, laser cladding, and microwave cladding [2–6]. Thermal spraying is the most commonly used technique due to its ease of operation and ability to deposit a range of materials on a substrate. However, the weak mechanical bonding between splats and the significantly higher porosity are the major limitations associated with the thermal spray technique [7–9]. Epoxy coatings are widely used in natural gas and crude oil pipelines, but these coatings exhibit weak bonding with their steel substrates and therefore they are subjected to under-film corrosion [6]. Enamel coating has excellent resistance to abrasion and mechanical shocks particularly in extreme wear and erosion applications. However, these coatings have a tendency to lose their properties as a consequence of thermal cycle and cleaning which they are subjected to on a daily basis. Laser cladding is the most widely used technique for cladding small components with high accuracy; but it is expensive. Further, there is a tendency of crack formation during the rapid melt pool solidification stage of the laser cladding process [10, 11] and corrosion (in the form of pitting and crevices) often originates at these crack formation sites.
Now-a-days microwave processing of materials is gaining popularity due to its specific advantages such as uniform heating, volumetric heating and better mechanical properties due to the improved microstructural characteristics of the microwave processed products. Further, microwave processing of the material is also one of the most environmental friendly process [12]. In microwave processing of materials, the heat is produced at the atomic level inside the material which resulted in lower energy consumption and increased productivity. In microwaves, the volumetric heat is generated inside the material owing to the atomic level interaction compared to the conductive mode of heat transfer in conventional surface heating technique; it results in a reduced thermal gradient inside the material which leads to reduced residual stresses inside the material and improved functional properties [13, 14]. The application of microwave heating in the form of microwave cladding to enhance the functional properties of metallic material was first given by Sharma et al [15] in the form of patent. The authors used principles of microwave hybrid heating (MHH) technique for the development of microwave clad. The authors reported that microwave induced clads exhibit improved tribological and mechanical properties. The developed clads exhibits the perfect diffusion bonding with the substrate without any interfacial cracking [15]. Afterwards a lot of research work is carried out to improve the surface properties of metallic material using cost effective microwave cladding technique due to its specific advantages such as low material wastage, low power consumption and ability to produce quality cladding having better tribological performance [16–20].

Inconel-625, a Ni based superalloy is one of the most commonly used material having excellent combination of mechanical properties and corrosion resistance. The outstanding mechanical properties of the Inconel-625 mainly depend upon the solid solution hardening provided by the niobium and molybdenum in the Ni–Cr based matrix. The addition of titanium and niobium acts as stabilizing elements to tie up carbon against sensitization to intergranular corrosion. Further, the Ni–Cr matrix provides high corrosion resistance in oxidizing environment by forming the passivating oxide layered structure on the surface of the Inconel-625, which protects the material from further corrosion [21–24]. Abioye et al [25] reported the corrosion performance of laser cladded Inconel-625 on the stainless steel (AISI-304) substrate. The authors reported that the corrosion performance of the coatings decreases with an increasing of iron (Fe) dilution; but, it is better than that of the wrought AISI-304 and very close to the wrought Inconel-625. The corrosion performance of the microwave cladded Inconel-625 on the mild steel substrate has not been reported till now. The corrosion resistance of ferrous based alloy such as mild steel can be improved by depositing a layer of Inconel-625 on its surface through microwave cladding technique. Therefore, in the present work, the surface properties of mild steel were improved using microwave cladding which is based on the MHH technique. Further, the characterization of the clad specimens was performed in terms of its microstructural, mechanical and corrosion behavior using various characterization techniques. The corrosion behavior of the clad specimens was determined in a 3.5 wt% NaCl solution using the Potentiodynamic polarization technique.

Materials and methods

Material detail
The commercially available Ni-based superalloy powder (Inconel-625) exhibit high hardness and corrosion resistance was selected as a clad powder. A typical SEM micrograph indicating the morphology of the Inconel-625 powder is shown in figure 1. The shape of the powder particles is mainly spherical with a size distribution of 40 ± 5 μm diameter. The mild steel, which is one of the most commonly used engineering materials was selected as a base material. The chemical compositions (wt %) of the substrate (MS) and Inconel-625 powder as obtained by the optical emission spectrometer (OEM) are given in table 1.

Experimental procedure
Inconel-625 powder was deposited on a mild steel substrate using an Industrial Microwave oven (Power = 1.2 kW and frequency = 2.45 GHz). A detailed description of the microwave cladding procedure has been explained elsewhere [15, 26]. The clad powder (Inconel-625) having an average thickness of about 1 mm was uniformly spread on the substrate upper surface. An alumina plate (separator) having a thickness of about 0.3 mm was kept on the preplaced powder and further the susceptor powder (charcoal) was placed on the alumina plate. After-words the whole assembly was placed in the microwave oven cavity and exposed to electromagnetic microwave radiation. The optimized process parameters (Power: 1.2 kW; Time = 10 min) were selected through various sets of trial runs. The principles of microwave hybrid heating (MHH) were successfully utilized for the deposition of Inconel-625 clad on the base material (MS) surface. During microwave exposure, the susceptor powder (charcoal) having a high loss tangent value easily couples with microwave radiation at room temperature and it transfers heat to the clad powder through the alumina plate (separator) by conduction mode of heat transfer. The temperature of the clad powder starts increases up to the threshold temperature and later, the powder particles themselves start absorbing microwave radiation resulting in melting of the clad powder.
However, the melting of the base metal was restricted to a thin depth only due to its low skin depth during microwave exposure. After the completion of the experiment, the melted pool becomes the solidified clad layer deposited on the base metal (MS) substrate surface. The schematic diagram illustrates the experimental setup used for the deposition of the clad through cost effective microwave technique is shown in figure 2. The power absorbed for completion of the experiment due to the internal electric field component of the microwave radiation can be calculated using the following relation.

$$P = 2\pi f \varepsilon_0 \varepsilon''_{\text{eff}} |E|^2$$

where: $\varepsilon_0 = 8.86 \times 10^{-12}$ F m$^{-1}$, permittivity of free space $\varepsilon''_{\text{eff}}$ = relative effective dielectric loss vector $f$ = frequency of microwaves (Hz) $E$ = electric field (V/m)

The corrosion behavior of the base metal (MS) and the microwave induced clad specimen were revealed using Potentiodynamic polarizations Tafel curves. The schematic diagram indicates the set-up used for investigating the corrosion behavior of the clad specimen is shown in figure 3. The tests were performed in a Gamry’s Interface 1000 Potentiostat equipped with DC-105 software. The sample surface area of 0.15 cm$^2$ was
exposed to 3.5 wt% NaCl solution at room temperature. The electrolyte de-aerated was performed by purging nitrogen gas about \( \frac{1}{2} \) h duration to ensure that experiment was performed in an oxygen free environment. In a 3 electrode cell configuration, the sample consists of the working electrode. The saturated calomel electrode (SCE) was used as a reference electrode and graphite rod acts as a counter electrode. With the help of Potentiostat, Potentiodynamic polarization scans were plotted for both base metal (MS) and microwave induced clad specimens with a sweep rate of 1 mV s\(^{-1}\) from the 1 h open circuit potential. The potential was scanned in the range of \(-500\) to \(+500\) mV. For each scan, the current between the counter and working electrode was recorded. Further, the surface characterizations of tested specimens (MS and clad) were examined using SEM/EDS to examine the corrosion mechanism.

**Microstructural and mechanical characterization**

The various phases formed in the clad specimen were probed through XRD operating at Cu-k\(\alpha\) radiation with coupled \( \theta-2\theta \) configuration. For microstructural analysis, the clad samples were cut along the transverse cladding direction using a slow speed diamond cutter. The cut samples were further mounted on epoxy and prepared as per standard metallographic procedure. The polished samples were ultrasonicated in alcohol before doing cross-sectional microstructural analysis. The SEM/EDS were utilized for investigating the elemental compositions of the microstructural features (phases) present along the cross-section of the prepared samples. The micro-indentation technique was used to evaluate the microhardness along the depth in the transverse cross-sectional of the clad specimens. The micro-indentation was performed at a normal load of 50 g for 10 s using Vickers indenter as a function of distance from the top surface towards the clad deposited direction. The porosity analysis was performed on the acquired optical images of the cladding with the help of an image analysis software, Dewinter Material Plus, Version 4.3. The magnification was chosen such that the cladding microstructure image covers the screen and allows the resolution of the voids that contribute notably to the total porosity area percentage. The pore area size in the view field was determined by converting the pore areas (grey-level areas) into a background color such as red while the rest of the microstructure remains in its original color. A total of ten readings were taken at different locations across the cross-section of the Inconel-625 cladding for calculating porosity. Finally the average value for the porosity measurement was reported.

**Results and discussion**

In the present work, the Inconel-625 clad was successfully deposited on mild steel (MS) substrate through a novel microwave hybrid heating (MHH) technique. The results obtained after performing various characterizations on deposit Inconel-625 clad are discussed in the following sections:

**Microstructural study**

A typical optical micrograph (figure 4(a)) illustrates the clad layer deposited on the mild steel substrate through electromagnetic microwave radiation. The clad layer having a thickness in the range of approximately 1 mm was seen to deposit on the MS substrate surface. It is clear from figure 4(a) that the perfect diffusion bonding between clad powder and the substrate was obtained through complete melting of powder particles. The metallurgical bonding was occurred between the substrate and the deposited clads though mutual diffusion of elements across the deposited clad and the substrate interface. The developed microstructure in the clad regions (figure 4(b)) was homogenous and dense with a very few voids and pores due to uniform cooling attributes of microwave hybrid heating (MHH) phenomenon. The SEM micrograph of the as-deposited Inconel-625 clad on MS substrate is
also shown in figure 4(c). From figure 4(c), a well bonded clad without any defect or interfacial cracking was observed. Inconel-625 is a heavily alloyed material and it generally solidifies in a dendritic mode. The columnar dendritic grain grew epitaxially from the substrate in a direction counter to heat flux direction was observed in SEM micrograph as shown in figure 4(a). In microwave cladding, the substrate acts as a heat sink. Thus, during solidification of clad powder, cooling of the melt pool, mainly occurs through the substrate. Thus the dendrites grain growth perpendicular to the substrate was observed. The dendrites are seems to be uniformly distributed in the clad regions of the microstructure due to a uniform cooling phenomenon associated with microwave hybrid heating (MHH) process.

XRD analysis
Figure 5 illustrates a typical XRD spectrum of the microwave induced Inconel-625 clad specimen. The XRD spectrum of the clad specimen indicates the presence of Laves, and carbides (NbC) phases in addition to main fcc solid solution matrix in the clad fusion zone. The formation of various carbides occurred due to the strong affinity of highly reactive elements such as Nb and Cr towards carbon at high temperature. The melting of the Inconel-625 powder was occurred at high temperature along with the melting of the upper surface of the substrate (MS). These melted elements react with each other at high temperature and it results in the formation of various secondary phases such as Laves and carbides in the clad zone of the microstructure. The various phases formed in the clad microstructure during melt pool solidification were further analyzed using relative peak intensity ratio. The normalized intensity ratios (NIR) were calculated using the relation as given in equation (2). Peak intensities of the different phases and normalized intensity ratio are shown in table 2.

\[ \text{NIR}_i = \frac{I_1 - I_{\text{back}}}{I_1 + I_2 + I_3 - 3I_{\text{back}}} \]  

where \( I_1, I_2, I_3 \) represent the intensities of 1st, 2nd, and 3rd phases, respectively, and \( I_{\text{back}} \) corresponds to the background intensity. The same relation equation (2) was also applied for calculating the normalized intensity ratio values for the other phases. For calculating the NIR value for a particular phase, the major peak
corresponding to that phase was taken. The NIR value, however, may not indicate the accurate amount of phases present; it provides a relative representation of the amount of phases. Thus, during microwave irradiation of the Inconel 625 powder, approximately 26.33% Inconel-625 powder got converted into NbC and Laves phases.

Porosity and elemental analysis

The porosity in the microwave induced clad specimens were determined as per the procedure explained in the above section. The porosity in the microwave induced clad specimens were found to be less than 1% due to uniform and volumetric heating phenomenon associated with MHH exposure. The observed porosity is, in general, very low while compared to many widely practiced surface treatment processes. The low value of porosity in microwave clads is attributed to the reduced thermal gradient in the process and low solidification rate. Further, there are two types of phase to be observed in the MHH induced clad microstructure; the matrix and the segregated dendritic phase as shown in figure 6(a).

The results of the EDS analysis along with compositions of the major elements present at the two regions marked ‘1’ and ‘2’ is shown in figures 6(b) and (c) respectively. It is clear from figure 6(b) that the grey regions marked 1 corresponding to matrix phase is rich in Ni, Fe, and Cr. The precipitates (light contrast) marked ‘2’ at the boundaries in between the dendritic core indicates the significant presence of Mo, Nb and Ti as shown in figure 6(c). This indicates that the dendritic core regions are mainly consist of Laves phase indicated by MN2 (M: Nb, Mo, Ti; N: Ni, Cr, Fe), they are formed due to segregation of Nb, Mo and Ti in the interdendritic spaces at high temperature during melt pool solidification. The presence of Laves phase was also confirmed by XRD spectrum as shown in figure 5.

Observation on microhardness

The microhardness measurement of the deposited cladding was obtained as per the procedure explain in section. There are two types of regions to be developed in the deposited clad cellular (matrix) and dendritic regions. The calculated value of the hardness of the substrate, cellular and dendritic regions was illustrated in table 3. The dendritic region has a significant high hardness followed by cellular regions and then followed by the substrate. The high hardness of the clad imparts the wear and corrosion resistance to the substrate surface. The

Table 2. Relative phase intensity of the different phases present in the clad zone.

| S. No. | Phases   | I1 | I2 | I3 | Iback | NIR% |
|-------|----------|----|----|----|-------|------|
| 1     | NbC      | 32 | —  | —  | 6     | 1.34 |
| 2     | Laves    |    | 470| —  | 6     | 23.96|
| 3     | Matrix   |    |    | 1452| 6     | 74.84|

Figure 5. A typical XRD spectrum of the deposited Inconel-625 clad specimen on MS.
The high hardness of the dendritic regions was due to presence of refractory elements Mo and Nb which are hard and brittle in nature.

**Corrosion performance**

The various corrosion parameters ($E$ corrosion potential, $i$ corrosion current density) were calculated by plotting Tafel plots (figure 7) are illustrated in table 4. The $E$ value of the clad specimen is significantly high indicates improved corrosion resistance of the clad specimen as compared to base metal (BM). Further, the corrosion current density $i$ is significantly high for BM indicating the BM corrodes severely in the 3.5wt% NaCl solution. The SEM micrograph of the corroded surfaces of the BM and clad specimen are shown in figure 8. From figure 8,
it is clear that the severe corrosion in the BM was occurred when it was exposed to 3.5 wt% NaCl solution. Further, the pits of size varies from 20 to 50 μm were also observed in the tested clad specimens. The corrosion in Inconel-625 was occurred mainly due to segregation of high atomic weight elements (Nb and Mo) in the interdendritic spaces.

### Conclusions

In the present work, corrosion behavior of the Inconel-625 cladding synthesized using the cost effective microwave technique was investigated. The deposited cladding was characterized in terms of phase identification, microstructure evolution and corrosion performance in a 3.5 wt% NaCl solution. The conclusions drawn from the present work are as follows:

1. Inconel-625 cladding on mild steel (MS) was successfully developed using the cost effective microwave technique.
2. The deposited Inconel-625 clad was well diffusion bonded with the substrate and growth of dendrites perpendicular to the substrate was seen in the fusion zone clad microstructure.
3. The normalized intensity ratio (NIR) indicates that the approximately 26.33% powder was converted into various phases (carbides and Laves) during microwave exposure.
4. The microhardness of the dendrites was significantly higher than the matrix phase followed by substrate (MS).
5. The clad specimen exhibit significantly higher corrosion resistance than base metal (MS) in 3.5 wt% NaCl solution.

### ORCID iDs

Hitesh Vasudev  https://orcid.org/0000-0002-1668-8765
Amit Bansal  https://orcid.org/0000-0002-0133-2897
Shubham Sharma  https://orcid.org/0000-0001-9446-8074
References

[1] Vijayakumar K, Sharma A K, Mayuram M M and Krishnamurthy R 2002 Response of plasma-sprayed alumina–titania ceramic composite to high-frequency impact loading Mater. Lett. 54 403–13
[2] Davis J R 2001 Surface Engineering for Corrosion and Wear Resistance (Delhi, India: Woodhead Publishing)
[3] Sun Y and Bell T 2002 Dry sliding wear resistance of low temperature plasma carburized austenitic stainless steel Wear 253 689–93
[4] Fan L, Tang F, Chen G, Reis S T and Koenigstein M L 2018 Corrosion resistances of steel pipe coated with two types of enamel by two coating processes J. Mater. Eng. Perform. 27 5341–9
[5] Fan L, Tang F, Reis S T, Chen G and Koenigstein M I. 2017 Corrosion resistances of steel pipes internally coated with enamel Corrosion 73 1335–45
[6] Fan L, Reis S, Chen G and Koenigstein M 2018 Corrosion resistance of pipeline steel with damaged enamel coating and cathodic protection Coatings 8 185
[7] Grewal H S, Singh H and Agrawal A 2013 Microstructural and mechanical characterization of thermal sprayed nickel–alumina composite coatings Surf. Coat. Technol. 216 78–92
[8] Qiao Y, Fischer T E and Dent A 2003 The effects of fuel chemistry and feedstock powder structure on the mechanical and tribological properties of HVOF thermal–sprayed WC-Co coatings with very fine structures Surf. Coat. Technol. 172 24–41
[9] Afzal M, Ajmal M, Khan AN, Hussain A and Akhter R 2014 Surface modification of air plasma spraying WC-12%Co cermet coating by laser melting technique Opt. Laser Technol. 56 202–6
[10] Zhou S, Huang Y, Zeng X and Hu Q 2008 Microstructure characteristics of Ni-based WC composite coatings by laser induction hybrid rapid cladding Mater. Sci. Eng. A 480 564–72
[11] Zhou S, Zeng X, Hu Q and Huang Y 2008 Analysis of crack behavior for Ni-based WC composite coatings by laser cladding and crack free realization Appl. Surf. Sci. 255 1646–53
[12] Agrawal D Microwave sintering of ceramics, composites, metals, and transparent materials J Mater Edu 19 49–58
[13] Clark D E and Sutton W H 1996 Microwave processing of materials Annu. Rev. Mater. Sci. 26 299–331
[14] Clark D E, Fozl D C and West J K 2000 Processing materials with microwave energy Mater. Sci. Eng. A 287 153–8
[15] Sharma A K and Gupta D A method of cladding/coating of metallic and nonmetallic powders on metallic substrates by microwave irradiation Indian Patent application No. 527/Del/2010
[16] Vassudev H, Singh G, Bansal A, Vardhan S and Thakur L 2019 Microwave heating and its applications in surface engineering: a review Material Research Express 6 1–20
[17] Kaushal S, Gupta D and Bhowmick H L 2017 On processing of Ni–WC based functionally graded composite clads through microwave heating Mater. Manuf. Process 33 822–88
[18] Kaushal S, Singh B, Gupta D, Bhowmick H and Jain V 2017 An approach for developing nickel–alumina powder–based metal matrix composite cladding on SS-304 substrate through microwave heating J. Comp. Materials 52 2131–8
[19] Hebbale A M and Srinath M S 2016 Microstructural investigation of Ni based cladding developed on austenitic SS-304 through microwave irradiation J. Mater. Sci. Tech. 31 1–9
[20] Pathania A, Singh S, Gupta D and Jain V 2015 Development and analysis of tribological behavior of microwave processed EWAC + 20% WC10Co2Ni composite cladding on mild steel substrate J. Manuf. Process. 37 1–9
[21] Shankar V, Rao K B S and Mannan S L 2001 Microstructure and mechanical properties of Inconel 625 superalloy J. Nucl. Mater. 288 222–32
[22] Paul C P, Ganesh P, Mishra S K, Bhargava P, Negi J and Nath A K 2007 Investigating laser rapid manufacturing for Inconel-625 components Opt. Laser Technol. 39 800–5
[23] Zupont J N D, Lippold J C and Kiser S D 2009 Welding Metallurgy and Weldability of Nickel-Base Alloys (New Jersey: Wiley)
[24] Smith G D, Tillack D J and Patel S J 2001 ed E A Loria Superalloys 718, 625, 706 and Various Derivatives (Warrendale, PA: The Minerals Metals & Materials Society) p 35–46
[25] Abiyo T E, McCartney D G and Clare A T 2015 Laser cladding of Inconel 625 wire for corrosion protection J. Mater. Process. Technol. 217 232–40
[26] Vassudev H, Singh P, Thakur L and Bansal A 2020 Mechanical and microstructural characterization of microwave post processed Alloy-718 coating Mater. Res. Express 6 1–13