Development of microwave processed Ni + 20% SiC based composite clads on AISI-304 steel

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Abstract. In the present investigation microwave hybrid heating technique was used to develop the Ni + 20% SiC based composite clads on AISI-304 substrate. The pilot experiments were carried out inside a domestic microwave oven working at 900W and 2.45GHz. The composite clads were successfully formed within 360 s of microwave exposure time. The so developed microwave processed clads were characterized through relevant techniques like XRD, SEM, EDS and Vicker’s microhardness measurement. The XRD study reveals the presence of various carbides and silicides in the composite clad region. Microstructure study results reveal the presence of hard carbide particles in the soft Ni matrix phases. The average value of microhardness in the clad region was observed as 763 ± 24HV, which was approximately 3 times that of the substrate microhardness.

1. Introduction
Metal ceramic based composite claddings/coatings are widely used on the surface of materials vulnerable to corrosion, oxidation and wear losses. These types of composite claddings/coatings constitute both tough and hard phases, which can enhance the corrosion and wear resistance of the material. Ni-based metallic materials along with various ceramic materials like tungsten carbide (WC), chromium carbide (Cr7C3), Silicon carbide etc. are mainly used for producing composite claddings [1-3]. These claddings are developed through various techniques like thermal spraying, plasma spraying, laser cladding etc. [4-6]. Laser claddings technique is widely used for developing composite claddings of various materials due to its unique properties such as the formation of an excellent metallurgical bond between the substrate and clad materials, higher cooling rate etc. [7]. However, in case of laser cladding, the heat is directly supplied on the surface of the material to be processed and hence results in large thermal gradient between the surface and the core of the material. This large thermal gradient causes the large thermal distortion and poor microstructure of the clad region. Apart from this, higher installation and running costs are associated with this process. In the recent years, the microwave cladding process is emerging as a noble and versatile surface modification process. Microwave cladding process involves some unique features such as less processing time, lower energy consumptions and volumetric heating. The volumetric heating characteristic results in uniform thermal gradient throughout the material during microwave cladding process and hence excellent microstructure is achieved. Gupta and Sharma (2012) [3] and Kaushal et al. (2017) [2] reported the composite claddings of Ni-WC8Co and Ni-Cr7C3 based materials using microwave heating at 2.45
GHz. Ni-SiC based claddings have been developed through various techniques such as thermal spraying, laser cladding etc. Less literature has been reported on the development of Ni-SiC clads through microwave cladding process. Kaushal et al. (2018) successfully developed the microwave cladding of Ni + 10% SiC based material on martensitic steel and reported that the clads were metallurgically bonded to the substrate material. It was also reported that composite claddings can be useful for anti-wear applications. In the present work, Ni-based EWAC + 20% SiC material was developed on austenitic stainless of AISI-304 grade using a domestic microwave applicator at 2.45 GHz frequency and variable power of 180-900 W. The so developed clads were further characterized using XRD, SEM/EDS, and Vicker’s micro-hardness measurements.

2. Materials and methods

2.1. Materials selection
Austenitic stainless steel (AISI-304) was used as the substrate material and was machined to the dimensions 10 mm x 10 mm x 6 mm. The commercial available EWAC powder (~98% Ni; Maker: Larsen and Toubro) of average grain size 42 ± 8 μm was mixed with 20% SiC powder (by weight%) of average grain size 30 ± 5 μm. The SEM images (Figure 1(a, b)) reveal the sharp edge morphology of SiC particles and round edge morphology of Ni based particles. The XRD spectra of both the powders are shown in figure 2 (a, b), which reveal the presence of majority SiC particles and small traces of SiO₂ in SiC based powder and enriched Ni particles in EWAC powder. Further, the chemical compositions of raw powders and substrate materials are shown in table 1.

![Figure 1. SEM morphology of (a) SiC powder; (b) EWAC powder.](image1)

![Figure 2. XRD spectra of (a) SiC powder; (b) EWAC powder.](image2)

| Material | Elements | Cr | Ni | Fe | P | C | Mn | Si | O |
|----------|----------|----|----|----|---|---|----|----|---|
| SiC powder | -       | 0.003 | 0.091 | - | 32.13 | - | Bal. | 8.11 |
| EWAC powder | 0.17 | Bal. | - | - | 0.2 | - | 2.8 | - |
| AISI-304 | 18.7 | 10 | Bal. | 0.45 | 0.08 | 2 | 1 | - |

![Table 1. Chemical composition of raw materials.](table1)
2.2. Development and characterizations of claddings

Cladding development through microwave heating is a very complex phenomenon. Prior to the deposition of the composite clad, the Ni + 20% SiC powder layer was preheated at 180°C in the microwave oven under convection mode. The preheated powder layer was placed manually on AISI-304 substrate by maintaining the approximate thickness of 1.1 mm. The substrate was placed on the refractory brick. The interaction behavior of microwaves is not same for each material. Microwave material interaction depends upon the skin depth of the material, which is defined as the depth from the surface into the material up to which the microwave power intensity falls by 36.8% of the surface value. In other words, the skin depth of the material is directly proportional to its microwave absorption capacity. The materials with lower skin depth have lower microwave absorbing capacity. However, the skin depth of the given material can be raised by increasing its initial temperature up to a critical value. The concept of microwave hybrid heating (MHH) was used by Gupta and Sharma (2014) [9] to achieve this critical temperature value. In case of MHH the susceptor particles (have good microwave absorbing characteristics) are placed on composite powder layer. An alumina separator sheet is placed between powder layer and susceptor particles to avoid any contamination. The whole arrangement was then placed inside the domestic microwave oven and the setup was turned on. Initially, the powder layer does not absorb the microwave radiations due to the lesser skin depths of the constituent powders. However, the susceptor particles start interacting with microwaves instantaneously and start converting microwave energy into heat energy. This heat energy is transferred to the composite powder layer through conventional modes of heat transfer. After getting heat from the susceptor particles, the skin depth of the composite powder layers rises up and the powder layers start absorbing microwave energy. Hence the melting of the powder layer is started and the cladding is achieved within 360 s of exposure time. The detailed principle of MHH is illustrated in figure 3. The different microwave cladding process parameters are tabulated in table 2.

To determine the behavior and various properties of the so developed composite clads, the characterizations of these clads were carried out using relevant techniques. Prior to the characterization, the microwave processed Ni + 20% SiC clad was sectioned across its thickness using the low-speed diamond saw. Polishing of the sectioned specimens was carried out through standard metallographic techniques. The X-ray diffraction spectrometer was used to analyze the different phases present in the composite clad region at the scan rate of 1° min⁻¹ and a scan range of 20° to 100°. The Microstructural characterization of the clad region was carried out using scanning electron microscopy (SEM) equipped with energy dispersive spectroscopy (EDS). The microhardness of the clad region was evaluated using Vicker’s microhardness measurement at 300 g of load and 20 s of dwell time.

![Figure 3. The principle of microwave hybrid heating.](image-url)
Table 2. Process parameters used for development of microwave composite cladding.

| Parameters                     | Description                                      |
|--------------------------------|--------------------------------------------------|
| Applicator                     | Multimode (Maker: LG, Model: Charcoal)           |
| Exposure power and Frequency   | 900 W and 2.45 GHz                                |
| Exposure time                  | 360 s                                            |
| Powder                         | Ni based + 20% SiC                                |
| Preheating powder temperature  | 180°C                                            |
| Separator                      | 99.1% pure alumina sheet                          |
| Susceptor                      | Charcoal powder                                  |

3. Results and discussion

3.1. Phase study analysis

A typical XRD spectrum of microwave processed Ni + 20% SiC clad is shown in figure 4. The presence of various phases such as Ni₃Si₂ (2θ: 29.49°, 44.68°), SiC (2θ: 35.74°, 75.45°), Ni (2θ: 39.66°, 45.06°), Cr₂₃C₆ (2θ: 48.30°, 92.12°) and NiSi (2θ: 52.07°) were observed in the composite clad region during microwave hybrid heating. During microwave hybrid heating, the SiC dissociates into Si and C at a higher temperature [8]. This free Si reacts with Ni element present in the EWAC powder to form Ni₃Si₂ and NiSi. The presence of Cr₂₃C₆ in the clad region may be due to the partial dilution of Cr elements from the substrate to the clad region and hence confirms the metallurgical bonding between the substrate and clad region. Some of the SiC is present in its original form corresponding to (2θ: 35.74°, 75.45°). The presence of SiC, Cr₂₃C₆, Ni₃Si₂ in the clad region results in higher microhardness of the composite clad.

![Figure 4. XRD spectrum of microwave processed Ni + 20% SiC clad.](image)

3.2. Microstructural study

To know the quality of the microwave processed composite clad, the microstructural analysis was carried out. It was observed in figure 5 (a), that the composite clad of approximate 1.02 mm thickness was developed using MHH technique. The clad was free from any type of visible cracks and porosity. The wavy interface between the substrate and clad region can be clearly seen. This wavy interface formation was due to the different thermal properties of constituent powders and substrate material, which results into the setup of convection currents at the interface region during microwave heating and hence causes the wavy interface. The same wavy interface was reported by Gupta and Sharma (2010). Further, the zoomed image of the clad region was shown in figure 5 (b), which reveals the randomly dispersed carbide particles in the form of flakes inside the soft Ni matrix. To determine the presence of different elements in the composite clad regions, the EDS analysis study was carried out at point X and Y corresponding to figure 5 (b). The presence of majority Ni element with small traces of C and oxygen was observed at point X (Figure 6(a)), while the presence of enriched carbon and Si elements was found at point Y as shown in figure 6(b).
Figure 5. SEM image showing (a) transverse section of the composite clad; (b) zoomed image of the enclosed region in figure 5 (a).

Figure 6. EDS analysis (a) at point X corresponding to figure 5 (b); (b) at point Y corresponding to figure 5 (b).

3.3. Vicker’s microhardness study
The presence of various carbides and silicides phases in the composite clad region could attribute to increasing its micro-hardness value. To verify this fact, the Vicker’s microhardness indentations were taken across the composite clad region starting from the top clad surface towards the substrate region at the interval of 170 µm. Three indentations were taken at each level and the average value of three readings was taken into consideration. The average value of the microhardness in the clad region was observed 763 ± 24 HV (Figure 7). The value of microhardness at the interface region was 420 ± 104 HV. The large standard deviation at the interface region was due to the dilution of different elements from the substrate and clad region.

Figure 7. Vicker’s microhardness profile of the microwave processed Ni + 20% SiC clad.

4. Conclusions
Microwave hybrid heating technique was successfully employed for the development of Ni + 20% SiC clads on AISI-304 substrate. The various conclusions drawn from the study are as following.
• The microstructure analysis of the composite clad reveals that the composite clads of 1.02 mm thickness were free from any type of visible porosity and cracks.
• The Carbide particles were randomly dispersed in the Ni matrix in form of flakes like structure.
• The presence of Ni$_3$Si$_2$, SiC, Cr$_{23}$C$_6$, NiSi phases were observed in the composite clad region during XRD study.
• The average value of the microhardness in the clad region was observed as 763 ± 24 HV which was almost 2.5 times that of substrate microhardness.

References
[1] A. Pathania, S. Singh, D. Gupta and V. Jain: J. Manuf. Processes Vol. 20 (2015), p. 79-87.
[2] S. Kaushal, D. Gupta and H.L. Bhowmick: J. Tribol- T. ASME Vol. 139 (2017), p. 061602-1-8.
[3] D. Gupta, P.M. Bhovi, A.K. Sharma and S. Dutta: J. Manuf. Processes Vol. 14 (2012), p. 243-249.
[4] H. S. Grewal, H. Singh and A. Agrawal: Wear Vol. 301 (2013), p. 424-433.
[5] G. Xie, X. Lin, K. Wang, X. Mo. Z. Zhang and P. Lin: Corros. Sci. Vol. 49 (2007), p. 662-671.
[6] L. St-Georges: Wear Vol. 263 (2007), p. 562-566.
[7] E. M. Birger, G.V. Moskvitin, A.N. Polyakov and V.E. Arkhipov: Weld. Int. Vol. 25 (2011), p. 234-243.
[8] S. Kaushal, V. Sirohi, D. Gupta, H.L. Bhowmick and S. Singh: P. I. Mech. Eng. L-J. Mat. Vol. 232 (2019), p. 80-86.
[9] D. Gupta and A.K. Sharma: J. Manuf. Processes Vol. 16 (2014), p. 176-182.