JMEPEG (2012) 21:2165–2172
DOI: 10.1007/s11665-012-0142-2

1059-9495/$19.00

Microstructural Characterization of Cermet Cladding Developed Through Microwave Irradiation

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(Submitted February 22, 2011; in revised form December 13, 2011)

In the present work, cladding of hardfacing WC10Co2Ni powder on austenitic stainless steel has been developed through a novel processing technique. The clads were developed using microwave hybrid heating. The clad of average thickness ~2 mm has been developed through the exposure of microwave radiation at frequency 2.45 GHz and power 900 W for the duration of 360 s. The developed clads were characterized using field emission scanning electron microscope, X-ray elemental analysis, X-ray diffraction, and measurement of Vicker's microhardness. The microstructure study of the clad showed good metallurgical bonding with substrate and revealed that clads are free from any visible interface cracking. Clads were formed with partial dilution of a thin layer of the substrate. The cermet microstructure mainly consists of relatively soft metallic matrix phase and uniformly distributed hard carbide phase with skeleton-like structure. The developed clads exhibit an average microhardness of 1064 ± 99 Hv. The porosity of developed clad has been significantly less at approximately 0.89%.

Keywords cermet, coatings, electron microscopy, microwave heating, stainless steels

1. Introduction

Many engineering components fail primarily due to wear and corrosion in aggressive interacting environments. Tribological components in gas turbine plant and hydro power plant are usually subjected to such severe working conditions, and consequently fail more frequently due to wear and corrosion. Designing such components for longer life, generally includes two approaches—first, bulk material design with high wear and corrosion resistance, and second, treatment (modification) of the functional surfaces to work satisfactorily in the aggressive working environment. Wear, corrosion, and oxidation are mainly surface-related phenomena and therefore, replacement of the bulk component body by newly designed material is often not a very cost-effective solutions.

Austenitic stainless steels have excellent corrosion resistance and have great utility as engineering materials in various applications across the industries. However, stainless steels exhibit inherently poor friction and wear characteristics. Austenitic stainless steels frequently suffer from severe metallic wear, due to the formation of strong adhesion junctions between the contact surfaces and severe surface/subsurface plastic deformation (Ref 1). Use of this high strength steel is thus not recommended in many potentially high wear applications. Functional properties of stainless steels can, however, be enhanced by suitable modification of the exposed surface through different techniques like carburizing, cyaniding, nitriding, and coating/cladding, etc. (Ref 1).

The improvement in wear resistance of the functional surfaces through coating/cladding with suitable overlaying material would be one of the most straightforward and economical solution for above problem. The coating/cladding can be developed through various methods like thermal spraying, plasma spraying, physical vapor deposition, chemical vapor deposition, laser melting, etc., and most of the techniques are well maturing in terms of academic research and technological developments. Laser cladding has been one of the most popular surfacing techniques among the widely practiced anti-wear industrial solutions. The laser processing too has some limitations including high distortion, development of porosity, and interface cracking apart from associated high setup and running cost. Moreover, laser processing is not very cost-effective as a technique for cladding of large areas (Ref 2). Recently, application of microwave energy for developing wear resistant cladding on metallic surfaces has been explored successfully (Ref 3). It was claimed that the novel process possesses high potential to emerge as one of the pragmatic surfacing solutions. Reasonably higher speeds of processing and higher degree of processing uniformity are some of the significant features of this process. Further, owing to volumetric nature of heating associated with microwave processing, clad produced through microwave heating exhibit significantly lesser thermal distortion, and nearly free from solidification cracks and pores. Presence of heat-affected zone (HAZ) and microcracking are the two major considerations owing to which surface scientists are emphasizing on development of an alternative technique to laser cladding process. The microwave cladding process, on the other hand, is potentially better in these aspects.

A significantly high volume of researches in the area of microwave processing due to some major significance of process like volumetric heating (Ref 4), selective heating etc., has been mainly in the domain of sintering and joining of

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ceramic and composites. Ceramic and ceramic composites are usually not good absorbers of microwave radiations at room temperature. However, using different techniques, microwaves are made to couple with such materials and processing is carried out at a very faster rate while compared to the conventional processing methods. The properties achieved by microwave processing for many applications have been reported to be superior to those obtained by conventional thermal processing (Ref 4-11). Application of microwave energy in processing metallic material is, on the other hand, quite challenging owing to the fact that microwave absorption coefficient for metals at 2.45 GHz radiation, the most common allowable frequency for industrial use, is significantly less at room temperature (Ref 12). This makes it extremely difficult to achieve heating in metallic materials through microwave irradiation without using hybrid heating technique (Ref 13, 14).

Successful sintering of metallic materials with or without susceptor (a material that readily absorbs microwave energy) was first reported in the year 1999 (Ref 15). Later, several authors have reported sintering of metallic materials through microwave heating (Ref 16-20). Gupta and Wong (Ref 21) reported sintering of aluminum, magnesium and lead. Cho and Lee (Ref 22) reported metal recovery from stainless steel mill scale using microwave heating. Takayama et al. (Ref 23) have reported production of pig iron by microwave processing of mixed magnetite and carbon powder at frequency 2.45 and 30 GHz. Borneman and Saylor (Ref 24) later reported coating of friction reducing alloys using CuNiIn powder on Ti-6Al-4V substrate through microwave irradiation. Sharma et al. (Ref 25) have reported joining of bulk metallic materials using microwave irradiation. Investigations on development of wear resistant cladding on bulk metallic substrates, however, have hardly been reported.

In the present work, cladding of WC10Co2Ni powder on austenitic stainless steel SS-316 substrate through microwave hybrid heating (MHH) technique has been carried out successfully using a multimode domestic microwave oven at 2.45 GHz frequency and power 900 W. The clads were characterized for constituting phases and structures using X-ray diffraction (XRD), electron microscope and other methods; initial results are reported.

2. Experimental Procedure

Clads were developed with certain focused objectives in which both the process and the materials (cladding as well as the substrate) play important roles. Quality of the clad, on the other hand, is influenced by a number of parameters as illustrated in Fig. 1. In the present work, wear-resistant cermet clad has been developed on metallic substrate using microwave radiation as the heating source. Processing of metallic materials in bulk form through microwave heating is very unusual. Partial dilution of the bulk metallic substrates was, however, achieved in the present work. The following sections briefly describe the development and characterization of the clads.

2.1 Materials Details

Hardness as well as toughness both are important for higher wear performance of a mating surface in sliding type of tribocontact. Tungsten carbide has good hardness and wear resistance, but poor toughness. However, tungsten carbide provides better wear resistance while bonded/dispersed in a

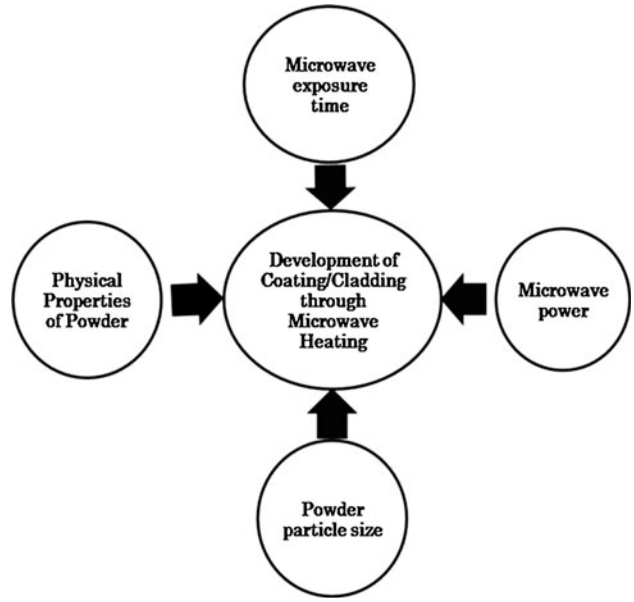

Fig. 1 Parameters affecting the microwave cladding process

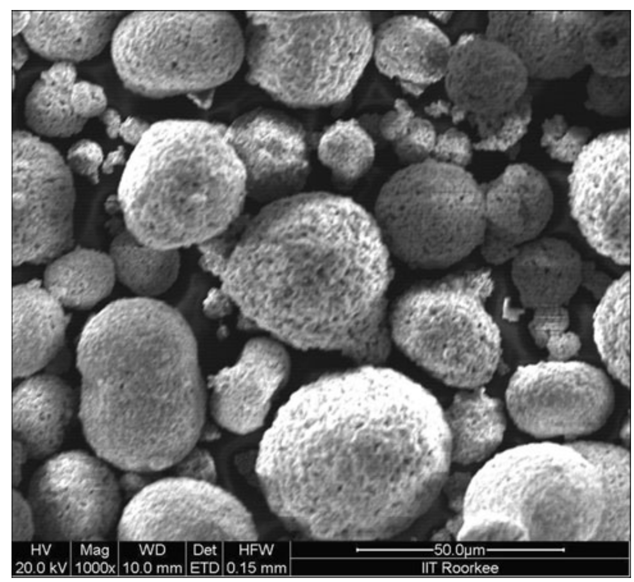

Fig. 2 Morphology of raw cladding powder (WC10Co2Ni)

tough phase. Thus, WC-Co-Ni can be one of the best systems for a combination of high hardness and toughness. In this system, cobalt (Co) acts as a binder, which is responsible for densification through wetting, spreading, and agglomerations during liquid phase sintering (Ref 26). Therefore, WC10Co2Ni system has been chosen as the clad material for the present study. In order to develop clad on austenitic stainless steel, WC10Co2Ni powder having average particle size of 40 μm was used. Typical cauliflower morphology of raw WC10Co2Ni powder used for deposition is illustrated in Fig. 2. Spherical morphology of the raw powder is seen clearly. The apparent rough and porous morphology provides higher surface area and hence better uniformity in heating through microwave absorption. The XRD spectrum as shown in Fig. 3 of the powder confirms the presence of tungsten carbide (WC) along with cobalt and nickel phase. Austenitic steel (SS-316) plates

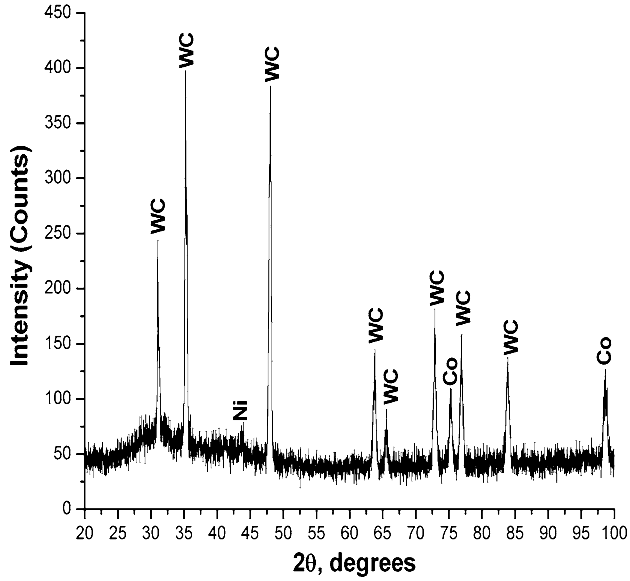

Fig. 3 XRD spectrum of the raw powder (WC10Co2Ni) obtained by the Cu Kα radiation

Table 1 Elemental weight composition of the substrate material (SS-316)

SS-316	C	Si	Mn	Ni	Cr	Mo	Fe	Others (P, S, Cu, Zn)
wt.%	0.08	0.75	2.0	8.0	17.0	2.0	Balance	1.0

machined to dimensions 35 mm × 12 mm × 6 mm were used as substrate material. Chemical composition of SS-316 used as substrate is shown in Table 1.

2.2 Development of Cladding

Preparation of raw powder and substrate is critical in development of clads. In the present work, the substrates were cleaned using alcohol in an ultrasonic bath prior to deposition. The WC10Co2Ni particles of average size 40 μm were pre heated at 100 °C for 24 h in a conventional muffle furnace (maximum temperature range 300 °C). Preheating removes possible moisture in the powder. The powder was preplaced manually on SS-316 substrate maintaining an approximately uniform thickness of 2 mm.

Processing of materials through microwave at frequency 2.45 GHz is material dependent. The development of cladding is highly dependent on physical properties of powder, powder particle size, and microwave processing parameters which is shown in Fig. 1. With the increase in surface area as the powder particle size decreases, absorption of microwave and hence melting of the particles becomes faster.

Further, the skin depth of the major constituent of the hardfacing powder tungsten (~4.7 μm at 2.45 GHz) (Ref 16) is less than the average particle size of the hardfacing powder (WC10Co2Ni) used in the trials. Hence, the WC10Co2Ni particles cannot directly interact with microwave radiation at room temperature, instead, will tend to reflect microwaves. In order to overcome the problem of microwave being reflected by WC10Co2Ni powder, clads were developed by MHH technique using suitable susceptor.

MHH technique utilizes a susceptor in the microwave cavity. The susceptor couples with microwaves at room temperature and gets heated up rapidly which, in turn, elevates the temperature of the WC10Co2Ni particles enabling them to couple with microwaves at higher temperature. The detailed discussion of microwave interaction with powder particle has been reported elsewhere (Ref 27). The metallic substrate as well as the metal-based powder particles gets heated up initially through the conventional mode(s) of heat transfer from the hot susceptor material. Subsequently, the heated metal-based materials start coupling with the microwaves and the powder particles get melted along with a thin layer of the substrate (dilution). The process gets over before a substantial melting of the substrate does take place (exposure time ~360 s in the present trials).

In order to avoid possible contamination of cladding by susceptor powder used in the MHH, a 99% pure graphite sheet was used as a separator between the susceptor and WC10Co2Ni powder as shown in the Fig. 4. The figure shows a schematic view of the MHH arrangement adopted during the trials. Details of development of cladding using MHH with suitable examples have been discussed at length by the authors (Ref 3).

In the present investigation, experimental trials was carried out in microwave multimode applicator (Make: LG, Model: Solar Dom) of frequency 2.45 GHz. The experimental trials were attempted using 600 and 900 W power while varying interaction time from 360 to 180 s in a step of 60 s for a constant thickness of preplaced powder layer. The corresponding results are presented in Table 2.

It was observed that for the current powder system, 600 W microwave power is not sufficient to cause complete melting of the powder as well as the substrate interface. However, at 900 W complete melting of the powder and partial dilution of the interface could be observed and hence, this operating conditions were maintained throughout the course of further experimentations.

2.3 Characterizations of the Clads

The developed clads were sectioned in a diamond cutter and polished with 1 μm diamond paste. The polished samples were cleaned thoroughly with acetone in an ultrasonic cleaner prior to proceeding for characterizations. The XRD patterns were obtained at room temperature in a Bruker AXS diffractometer with Cu Kα X-ray. The scan rate used was 1° min^{-1} and the scan range was from 20° to 80°.

The analysis of clad microstructures and chemical composition of the clads were carried out using a field emission scanning electron microscope (FE-SEM) at an acceleration voltage of 20 kV equipped with an energy dispersive X-ray detector (Make: FEI, Model: Quanta 200 FEG). The X-ray elemental composition analysis of clad specimens was carried out progressively at an interval of 120 μm distance from the substrate towards the top of the clad section. Microhardness measurement along the thickness of the clad and substrate was accomplished by a Vicker's microhardness tester (Mini load, Leitz, Germany) at the load of 50 g applied for 30 s.

3. Results and Discussion

It has been already reported that processing of bulk metallic substances by microwave radiation is so far not a common

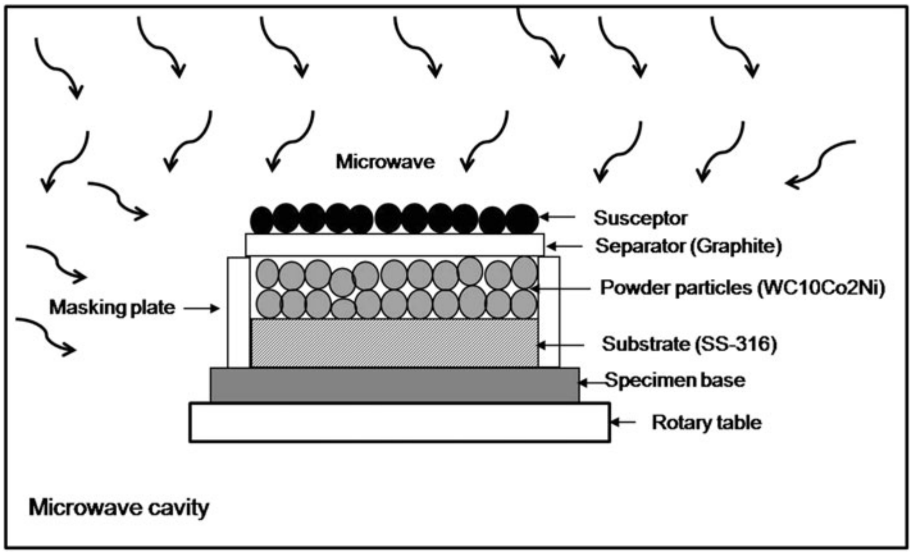

Fig. 4 Schematic of the microwave hybrid heating set-up used for claddings

Table 2 Microwave processing parameters and their effect on cladding

Trial	Processing time (s)	Microwave power (W)	Results	Possible inference
1	180	600	No melting resulting no cladding	Insufficient interaction
2	240	600	Partial melting of the powder particles and poor bonding with substrate	Insufficient interaction time, insufficient power
3	300	600	Partial melting of the powder particles and poor bonding with substrate and no evidence of partial substrate melting	
4	360	600	Partial melting of the powder particles and poor bonding with substrate	Insufficient power to cause melting of cermets as well as thin layer of substrate.
5	180	900	No melting resulting no cladding	Insufficient interaction time
6	240	900	Partial melting of the powder particles and poor bonding with substrate	
7	300	900	Partial melting of the powder particles and marginal bonding with substrate	Insufficient interaction time to cause full melting of cermets powder with no dilution
8	360	900	Cladding with good metallurgical bonding	Sufficient microwave power and interaction to cause complete melting of cermets powder with partial dilution of substrate

application of microwave heating (Ref 13, 15). Most of the ceramics and ceramic composites are also transparent to microwave radiations in room temperature. Thus, a combination of metal-ceramic system needs hybrid mode of heating for effective material processing. In the present work, microwave cladding of WC10Co2Ni powder were successfully carried out on austenitic stainless steel (SS-316) substrate using a multimode domestic microwave oven. The clads were subsequently characterized by subjecting them to different tests. Results are discussed in the following sections with suitable illustrations.

3.1 X-Ray Diffraction Studies

A typical XRD spectrum of the WC10Co2Ni clad developed by hybrid microwave heating is presented in Fig. 5. In the XRD pattern, presence of tungsten carbide (W_2C) and free tungsten (W) would be clearly identified. It is attributed to possible decomposition of WC from the starting powder into free-W and W_2C at high temperature during MHH as can possibly be represented in the relations (1) and (2):

$$WC \rightarrow W + C \quad \text{(Eq 1)}$$

$$WC + W \rightarrow W_2C \quad \text{(Eq 2)}$$

This free carbon might react with atmospheric oxygen to form CO which escapes subsequently during solidification (slow cooling rate) leading to a significantly less porosity and crack free clad. Highly reactive free carbon also reacts with both powder and substrate materials and form metallic complex carbides (Co_2C at $2\theta \sim 28.83°$, $Cr_{23}C_6$ at $2\theta \sim 59.74°$) and $Co_3W_9C_4$ corresponding at $2\theta \sim 28.83°$ which can possibly be represented in the relations (3) and (4).

$$9W + 3Co + 4C \rightarrow Co_3W_9C_4 \quad \text{(Eq 3)}$$

$$6Fe\ (\text{substrate}) + 6W + C \rightarrow Fe_6W_6C \quad \text{(Eq 4)}$$

The diffraction peaks in Fig. 5 show the presence of complex carbides like $Co_3W_9C_4$, Fe_6W_6C corresponding to 2θ of 28.83° and 42.54°, respectively, and few relatively stable

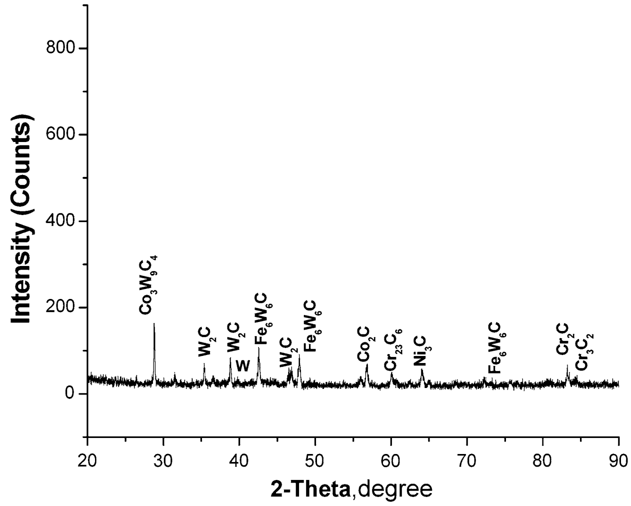

Fig. 5 A typical XRD spectrum of the WC10Co2Ni cladding (radiation: Cu Kα)

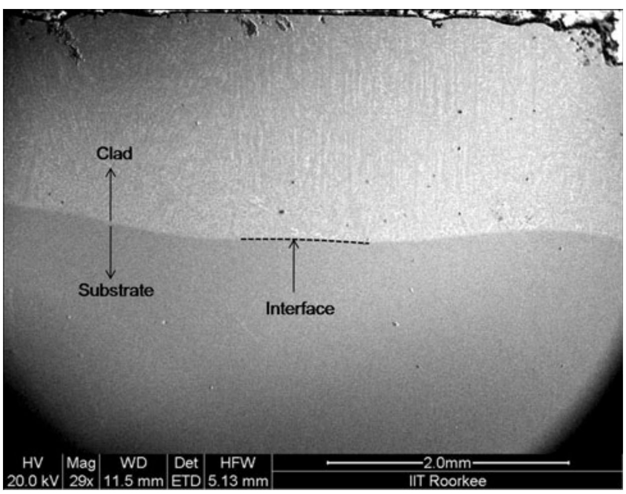

Fig. 6 BSE image of transverse WC10Co2Ni clad surface

carbides like Co_2C, $Cr_{23}C_6$, Ni_3C, Cr_2C, Cr_3C_2. The presence of Fe, Cr, and Ni carbides are due to dilution of cladding material with substrate. Intermetallics are not observed in the XRD pattern, which are usually found in the deposition process like laser cladding (rapid solidification) (Ref 28).

3.2 Observations on Cladding Microstructure

The back scattered electron (BSE) image of a typical transverse section of the WC10Co2Ni clad is illustrated in Fig. 6. The cladding with an average thickness of ~2 mm shows good metallurgical bonding with substrate through partial mutual diffusion of elements like iron (Fe), chromium (Cr) from the substrate to clad and heavier tungsten from clad to substrate. The porosity of developed clad has been measured by linear point count method; results show significantly less porosity of 0.89%. The observed porosity is, in general, very low while compared to many widely practiced surface treatment processes. This low porosity in microwave clads is attributed to the reduced thermal gradient in the process and consequently low solidification rate. The microstructure presented in Fig. 6 further reveals that the developed clad is free from interfacial cracking despite of the significance mismatch of coefficient of thermal expansion of clad material and substrate SS-316. The crack free clad microstructure is also indicative of uniform heating associated with microwave heating.

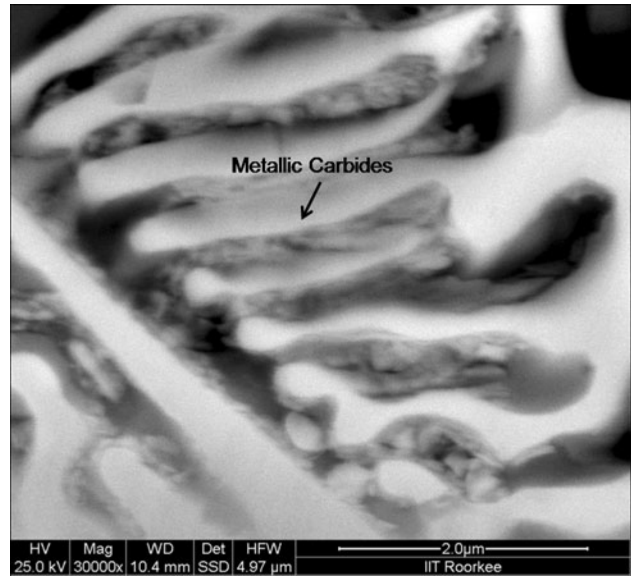

Fig. 7 Skeleton-like structure in WC10Co2Ni clad

Once the temperature of the entire powder layer rises to the melting temperature during irradiation, subsequently a very thin layer of the substrate interface reaches the melting state. This is followed by nucleation of austenite, which grows at the boundary of the liquid solution along with impoverished tungsten and carbon. Austenite, thus, forms binary carbides of (tungsten and iron) $_6$C having skeleton like structure as shown in Fig. 7, which may precipitate from the liquid solution during solidification of Fe-W-C alloys (Ref 29).

Typical microstructure of microwave clad is illustrated in Fig. 8. Microstructure of clad observed like those of reinforced composites with a dominating skeleton structure (reinforcement) in a metallic matrix. The compositions of matrix marked ‘1’ and skeleton marked ‘2’ as shown in Fig. 8 were further analyzed through point X-ray elemental composition. The corresponding elemental distributions are summarized in Table 3. The X-ray elemental composition study exhibits that the matrix consists of the major elements Fe, Cr, Ni, Co with the dominating presence of iron. The elemental distribution of the skeleton structure, on the other hand, consists of dominating W and Fe elements with corresponding contributions of approximately 61 and 24%. The EDS spectra of the points ‘1’ and point ‘2’ are shown in Fig. 9(a) and (b), respectively. Presence of carbon in the microstructure is clearly seen. Thus, it is likely that the matrix phase consists of the relatively soft metallic materials that exist in the starting powder (Ni and Co) and getting diluted from the metallic substrates (Fe and Cr). The skeleton structures, on the other hand, are the harder metallic (M) carbides (M_xC, M_xM_yC) as observed in the XRD spectrum (Fig. 5). Thus, there exists a strong possibility of exhibiting higher wear resistance by the clads with well distributed hard metallic carbides (reinforcements) in a tough metallic matrix.

3.3 Microhardness Study

Hardness of a material is one of the most important factors, which influence wear performance of the material. Generally, increasing the hardness of components can enhance the wear resistance ability although the effect of hardness is not straightforward. The Vicker's microhardness of clads over the cross-section were evaluated. The distance between two indentations was kept 120 μm with an additional indentation at the fusion line or interface and the substrate. The distribution of microhardness is shown in Fig. 10. The average microhardness of the clad section is 1064 ± 99 Hv. The distribution of microhardness in the clad section is, however, not observed to be uniform. This is attributed to the difference in the hardness of the metal-based matrix phase and the hard carbide based ceramic reinforcement (skeleton structure). As the measurements were carried out through micro indentation at 50 g load, it is quite possible to get isolated microhardness value of the matrix as well as the skeleton structure.

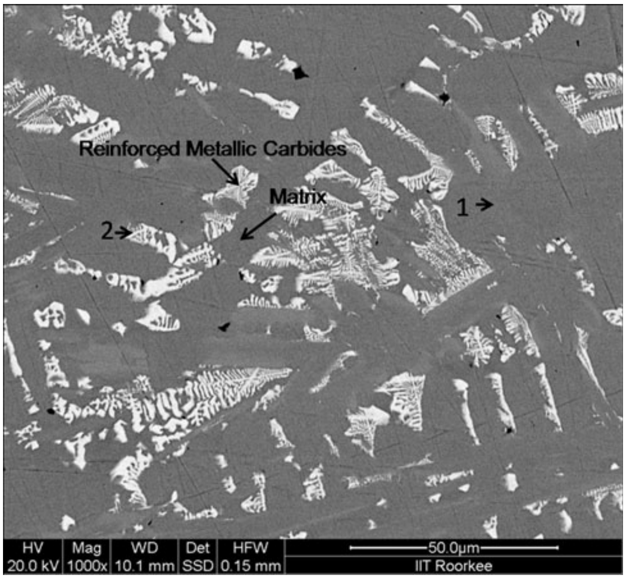

Fig. 8 Back-scattered electron image of transverse clad surface showing skeleton structure

Table 3 Results of X-ray elemental analysis

Element	wt.% at point '1'	wt.% at point '2'
CK	04.04	04.08
OK	02.00	00.69
CoK	08.73	01.55
WM	13.28	61.10
CrK	18.17	05.15
FeK	47.87	24.34
NiK	05.91	03.08

The typical indentation geometries in microwave clad is shown in Fig. 11. The indentation (A) as shown in Fig. 11, is located in between the soft matrix and skeleton. The geometry of indentation shows, diagonals has been depressed and extended towards apex of the pyramid by virtue of which it has grown bigger. The indentations (B) and (C) on the skeleton of the mainly tungsten based element are lower in size compared to the indentation (A) and subsequently high microhardness results.

An increasing tendency in hardness away from the substrate is seen. The microhardness from top of the clad to the half of clad is approximately uniform (≈1100 Hv), which, however, reduces and approaches ≈800 Hv at the clad-substrate interface. The observed higher microhardness in the clad could be attributed to the presence of different hard phase like Fe_6W_6C, $Cr_{23}C_6$, Co_3W_3C, etc., as observed in the XRD spectrum in Fig. 5. However, as observed from the Fig. 10, the drop in clad hardness occurs due to the increased metallic dilution as the interface approaches. The microhardness at the interface was found to be about ≈800 Hv. The higher standard deviation in the measurements can be attributed to the indentations being carried out at the hard skeleton structure as well as at the tough matrix phase as observed in the microstructure in Fig. 11.

4. Conclusions

A novel technique for the development of cladding has been illustrated and the characterization of the developed WC10Co2-Ni clads on SS-316 substrate has been carried out. The major conclusions drawn from the above studies are:

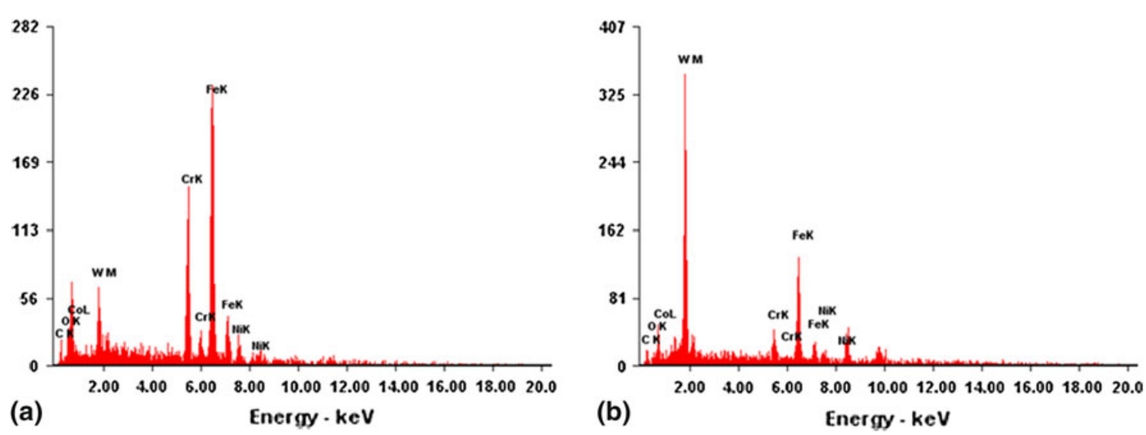

Fig. 9 Typical EDS spectra of: (a) matrix, and (b) reinforcement of the clad

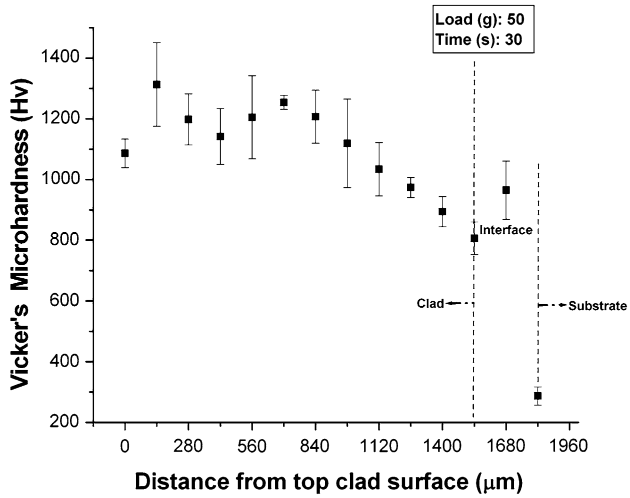

Fig. 10 Vicker's microhardness distribution in a typical section of WC10Co2Ni clad

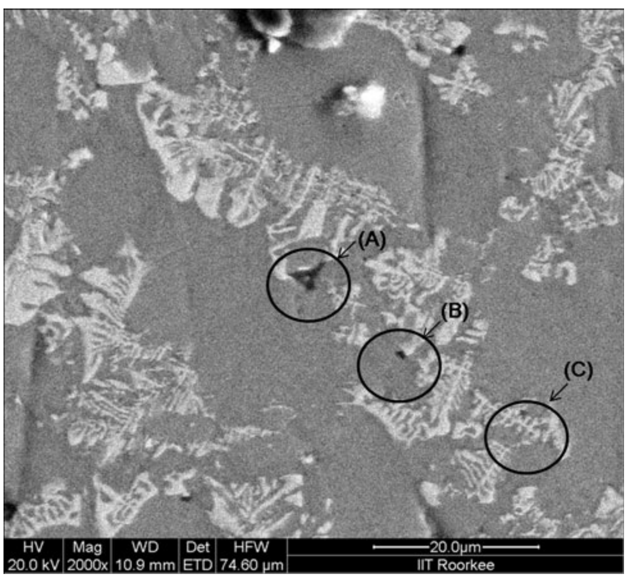

Fig. 11 Micrograph showing the Vicker's indentations in a WC10Co2Ni clad section

(1) A cermet (WC-Co-Ni based) clad of average thickness 2 mm could be developed by the exposure of microwave radiation at 2.45 GHz and power 900 W for a duration of 360 s.
(2) The microwave cladding is capable of producing uniform treatment on target bulk metallic surfaces owing to the volumetric heating associated with microwave heating.
(3) The microwave clad is free from interfacial cracking due to volumetric heating during microwave irradiation.
(4) The microwave clads exhibit significantly less porosity which is in the order of 0.89% owing to low solidification rate associated with microwave treatment.
(5) The clad is well metallurgically bonded with substrate in term of dilution and partial mutual diffusion of elements.
(6) The characteristic skeleton like structure is formed consisting of carbides of Fe-W in the microwave clads during MHH.
(7) The typical skeleton-like microstructure significantly contributes towards enhancement in microhardness of the modified surfaces (clads).
(8) The average microhardness of developed clad is $\sim 1064 \pm 99$ Hv. Microhardness at the top clad surface zone is higher; which, however, reduces towards the interface zone owing to the increased dilution with the metallic substrate.
(9) The developed technology has potential for applications in surface treatment of materials to induce specific functionality (for example, higher wear resistance).

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