SURFACE MODIFICATION OF MARTENISTIC STAINLESS STEEL THROUGH MICROWAVE HYBRID HEATING

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in
Production Engineering

by
Vinay Sirohi

Registration No. 801382028

MECHANICAL ENGINEERING DEPARTMENT
THAPAR UNIVERSITY, PATIALA

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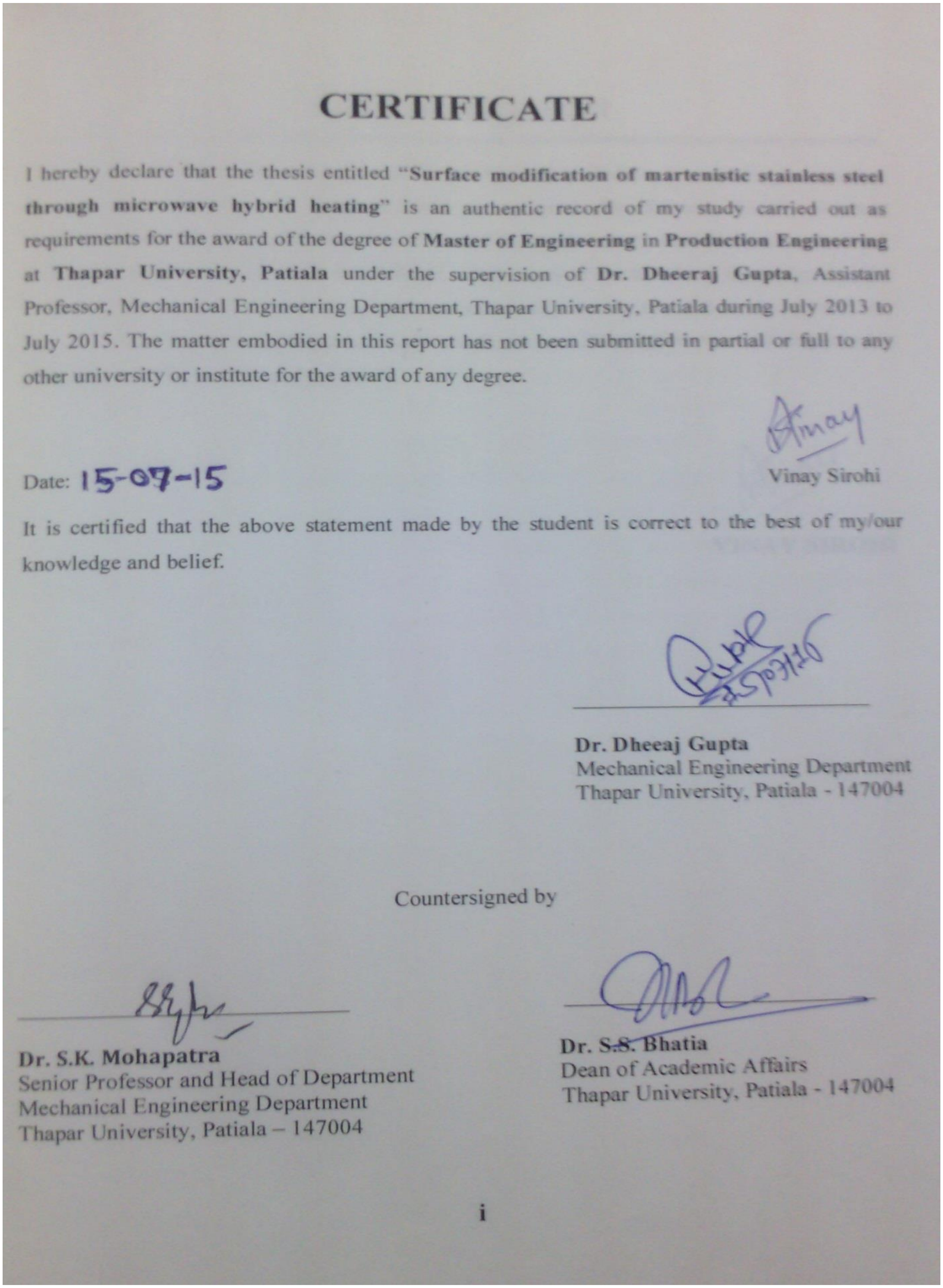


CERTIFICATE

I hereby declare that the thesis entitled "**Surface modification of martenistic stainless steel through microwave hybrid heating**" is an authentic record of my study carried out as requirements for the award of the degree of **Master of Engineering** in **Production Engineering** at **Thapar University, Patiala** under the supervision of **Dr. Dheeraj Gupta**, Assistant Professor, Mechanical Engineering Department, Thapar University, Patiala during July 2013 to July 2015. The matter embodied in this report has not been submitted in partial or full to any other university or institute for the award of any degree.

Vinay

Date: 15-07-15

Vinay Sirohi

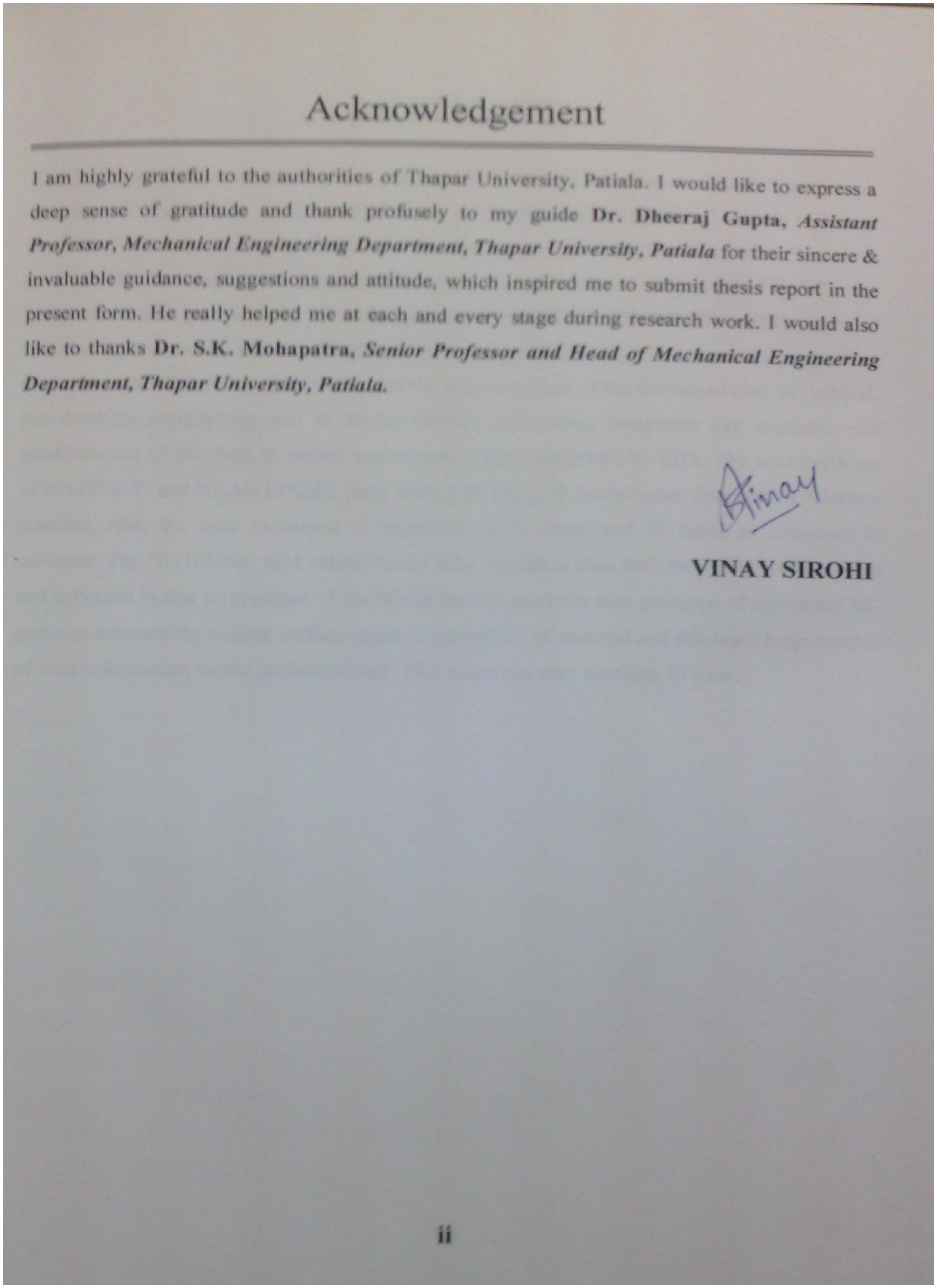
It is certified that the above statement made by the student is correct to the best of my/our knowledge and belief.

Dr. Dheeaj Gupta
Mechanical Engineering Department
Thapar University, Patiala - 147004

Countersigned by

Dr. S.K. Mohapatra
Senior Professor and Head of Department
Mechanical Engineering Department
Thapar University, Patiala – 147004

Dr. S.S. Bhatia
Dean of Academic Affairs
Thapar University, Patiala - 147004



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Vinay

VINAY SIROHI

ABSTRACT

Surface modification of martensitic stainless steel through microwave hybrid heating was investigated. The present work mainly focuses on development of hardfaced carbide layer and intermetallic matrix composite (IMC) with reinforcement, and their characterization. The clads were developed using nickel based powder with 10 wt.% silicon carbide (Ni+10%SiC) and nickel aluminium powder with 10 wt.% silicon carbide (Ni-Al+10%SiC). The XRD results of two clad layers confirm phases of high carbide $Cr_{26}C_6$ and NiAl–Ni_3Al intermetallic composites. Characterization of clad surface results that the microstructure of the developed clad Ni+10%SiC has dendritic morphology and in Ni-Al+10%SiC clad shows composite like structure with reinforcement of SiC both in melted and unmelted form confirmed by EDS. The microhardness of Ni+10%SiC and Ni-Al+10%SiC clads were 2.86 and 2.76 times higher than that of substrate material. Also the wear resistance is improved by 71 times and 52 times as compared to substrate. The Ni+10%SiC clad exhibit better wear resistance than both the Ni-Al+10%SiC clad and substrate is due to presence of hardfaced carbide particles also presence of un-melted SiC particles between the mating surface results in ploughing of material and this leads large number of craters formation on the surface of clad. The craters are very sensitive to wear.

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Abbreviations

IMC	Intermetallic Matrix Composite
EDS	Energy Dispersive X-Ray Spectroscopy.
EM	Electromagnetic Spectrum.
HAZ	Heat Affected Zone.
MHH	Microwave Hybrid Heating.
SEM	Scanning Electron Microscopy.
XRD	X-Ray differactometer.
SS	Stainless Steel

Chapter 1

Introduction

1.1 Stainless Steel

1.1.1 General Introduction

Stainless Steel (SS) is an alloy of iron having minimum of 10.50% chromium content by mass [1]. It doesn't easily corrode and get rusted. Even though the name, it is not totally mark-proof it gets marks with water considerably under high-salinity or poor environments or low-oxygen. There are number of grades of SS as shown in Table 1.1 which is used in various fields like domestic, transport, architectural/civil engineering, food and drink, chemical/pharmaceutical, medical etc. used to suit different working environments. The alloy must endure different properties to satisfy the working environments.

Table 1.1: Different grades of stainless steel and their applications [2]

Types	Examples	Applications
Ferritic	410S, 430, 446	• Refrigeration cabinets • water treatment plant etc.
Austenitic	304, 316	• automotive trim • cookware.
Duplex	1.4462	• tubes and pipes • heat exchangers • pressure vessels etc.
Martensitic	420, 431	• Nuclear reactor • turbine blades • multi-purpose tools etc.
Precipitation Hardening	17/4PH	• flat springs and rings • aerospace components etc.

1.1.2 Stainless Steel 420 (SS-420)

Stainless steel 420 grade have chromium (Cr) content less than 12% and is high carbon percentage steel. It can be hardened like other SS, with help of heat treatment. It also has excellent properties like resistance to corrosion and in annealed state it shows very good ductility. In between all the grades of stainless steel, grade 420 has highest hardness. The ranges of element composition are shown in Table 1.2.

Table 1.2: Ranges of element composition of SS-420 [3]

Grade		**C**	**Mn**	**Si**	**P**	**S**	**Cr**
420	min.	0.15	-	-	-	-	12.0
	max.	-	1	1	0.040	0.03	14.0

Mostly for engineering applications like nuclear reactor, multipurpose tools, turbine blades etc. stainless steel 420 is used since it has better hardness, corrosion resistance etc. as compared to other grades of stainless steel. But to further improve its function and serviceability surface engineering techniques like cladding, thermal spraying, carburizing etc. are applied on it.

1.2 Surface Engineering

Surface engineering is a multidisciplinary activity that uses a voluminous range of technologies. The aim of surface engineering technology is designing and modifying the properties of engineering components [4]. It is used to alter the properties of the surfaces of components, so that we can improve their function and serviceability. Hence this technology is used to optimize the surface properties. The desired properties or characteristics of surface-engineered components needed are shown in Fig1.1.

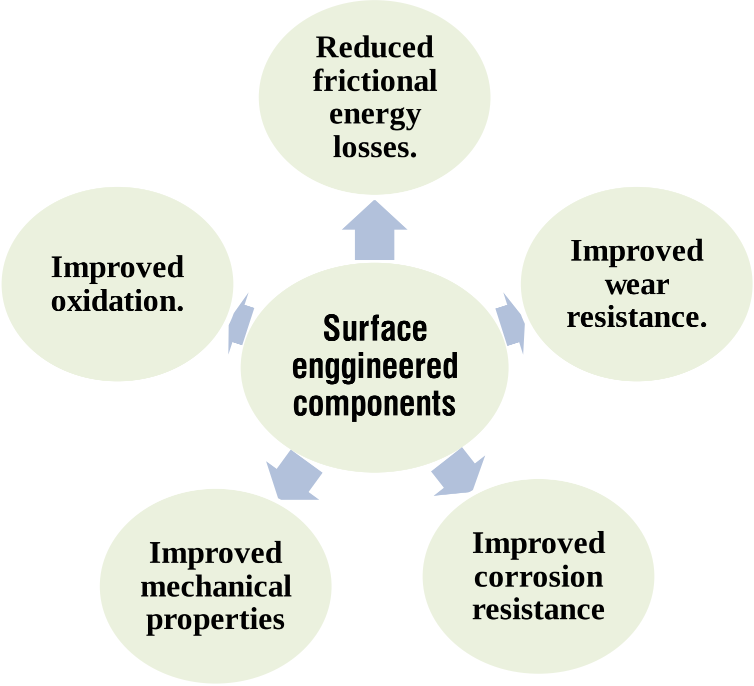

Figure 1.1: The desired properties or characteristics of surface-engineered components

Surface engineering can be done by three methods that are surface metallurgical modification, surface modification by changing surface chemistry and surface coating. Processes used for surface engineering includes chemical vapour depositioning (CVD), explosion cladding, physical vapour depositioning (PVD), plasma and thermal spraying, cladding and electroplating etc [5]. Surface modification processes are applied to improve the physical or mechanical or both properties of the components like surface wear, control friction and corrosion resistance. Some of surface modification methods are hardening of material by laser or electron beam, flame, diffusion treatments, e.g. nitriding & carburizing etc. [6].

When there is relative motion between the two surfaces which is in contact, the stresses between the two surfaces increases due to the relatively low percentage of the load-supporting area. This possibly can even lead to failure as a result of friction and wear. In today's high stress applications of modern engines, there are certain areas where due to the pressure between two moving metal surfaces, forces away all the lubricating oil and leads heat to build up. In the extreme cases where the pressure and friction are very much high, this heat is sufficient to instantaneously weld the two parts together before their movement broke them apart [7]. These constants welding and breaking process which is occurring at the atomic scale results in drag and wear, which act destructive. If the gears are the components of critical machinery like spacecraft and if only the gear part fails, it leads to the failure of entire million dollar spacecraft. So it is highly desirable for engineering component surfaces to fight friction and reduce wear.

1.2.1 Surface Modification and Surface Coating Techniques

A variety of surface modification methods exists ranging from simple heat treatment to the relatively sophisticated diffusion treatment methods. Some of the surface modification methods are shown in Fig.1.2 [8]. Surface modification technique to be used for developing a substrate depends upon the purpose for which it is coated. Coating material used and other parameters influence the surface coating techniques.

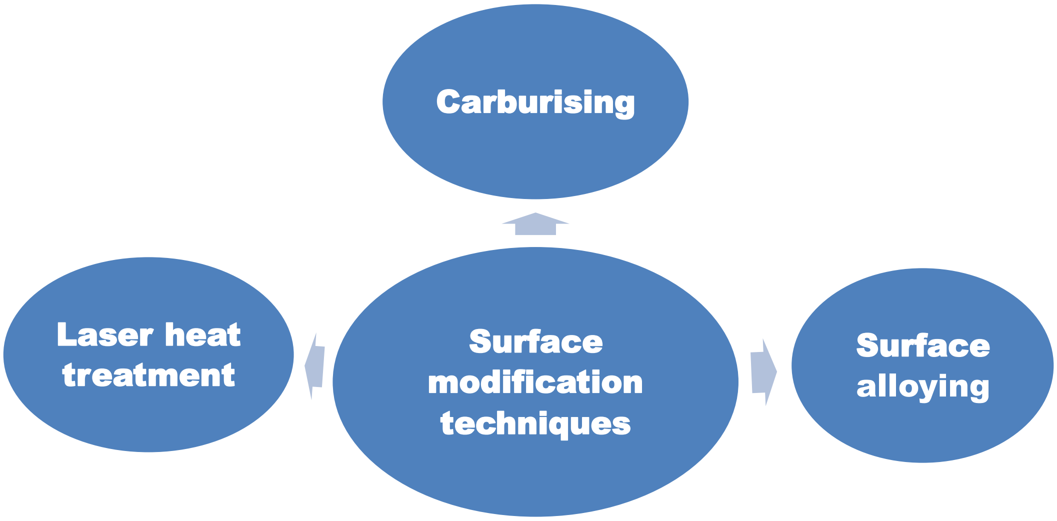

Figure 1.2: Some common surface modification techniques

Surface coating processes involve depositing a layer onto a substrate; the layer can be of molten material, semi-molten material or some chemical material. Instead of redesigning the composition of the bulk material, with the help of surface coating the material can be modified and reinforced to perform the surface functions. Some popular surface coating techniques used in industry are shown in Fig.1.3 [9].

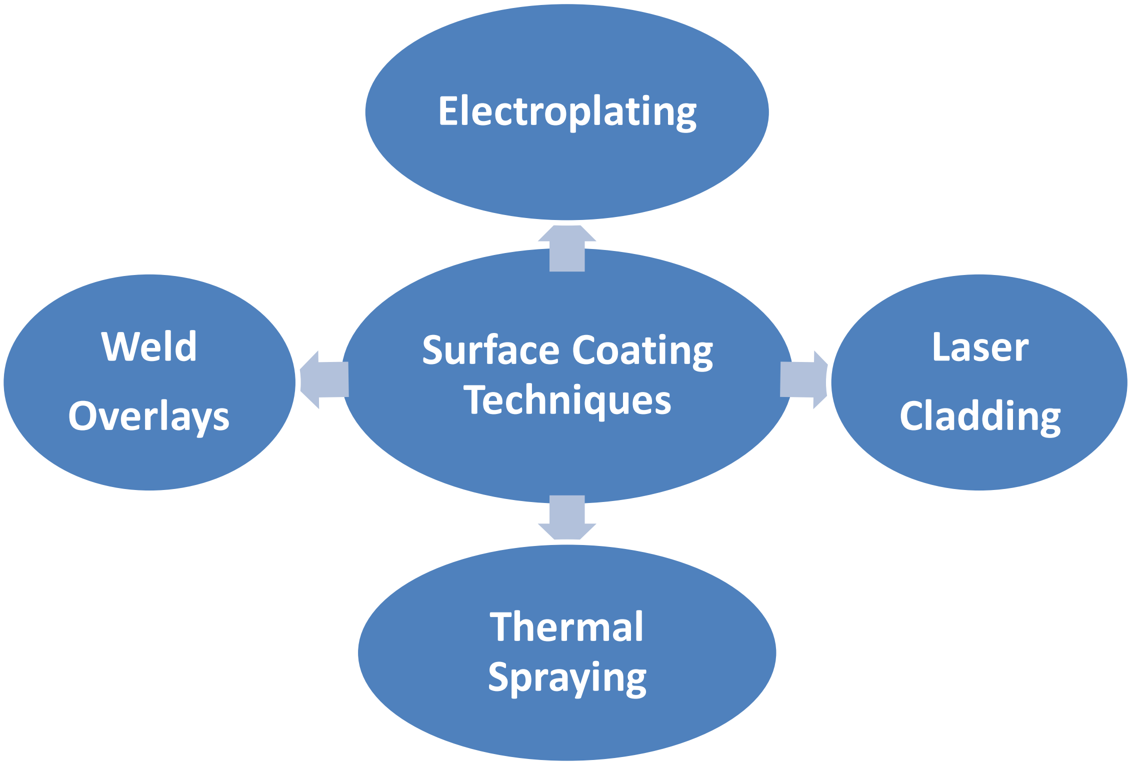

Figure 1.3: Some common surface coating techniques

Surface coating processes involve depositing a layer onto a substrate; the layer can be of molten material, semi-molten material or some chemical material [10]. Instead of redesigning the composition of the bulk material, with the help of surface coating the material can be modified and reinforced to perform the surface functions. Some common problems depending on the applied techniques are the occurrence of porosity, applied clad layer poor bonding with base material, the thermal distortion of the specimen and also the dilution / mixing of clad layer to base material [11].

One of the techniques that overcome these problems is microwave cladding.

1.2.2 Application of Surface Engineering Techniques

The applications of surface engineering have been extensively investigated. They include aeronautic and transport industries, chemical and petroleum industries, sports technology and the electronics industries. Recently, in some specialized areas the surface engineering technologies have been utilized. Such applications include thermal sprayed coatings in the sports industry, dentistry, cancer therapy, biomedical/orthopaedics (e.g. hydroxyapatite), art industry (e.g. glass colouring and enamelling) [12]. To modify the properties of the different components a wide range of methods provided by the surface coatings. A typical coating includes pure metals and alloys, carbides, nitrides, diamond like carbon (DLC), thermal barrier coatings and decorative coatings. Modern cutting applications can’t be fulfilled without protecting the cutting tools with a thin wear-resistant coating. These modern applications include machining of high hardness (Rockwell>60C) materials, high speed cutting tools and difficult to cut materials such as AlSi alloy, titanium, or other non-ferrous abrasive. The surface coatings deposited on the different tool surfaces normally have a several microns of thickness. These coatings enhance the cutting edge wear resistance and reduce friction and diffusion [13].

1.3 Wear and Corrosion

It is highly desirable for engineering component surfaces to fight in harsh conditions and owing to which reduced wear rate. Surface engineering techniques solve many failure problems (friction, corrosion, and wear) and most responsible factors that causes catastrophic failure of engineering components. The degradation of the components due to corrosion has been shown in Fig. 1.4.

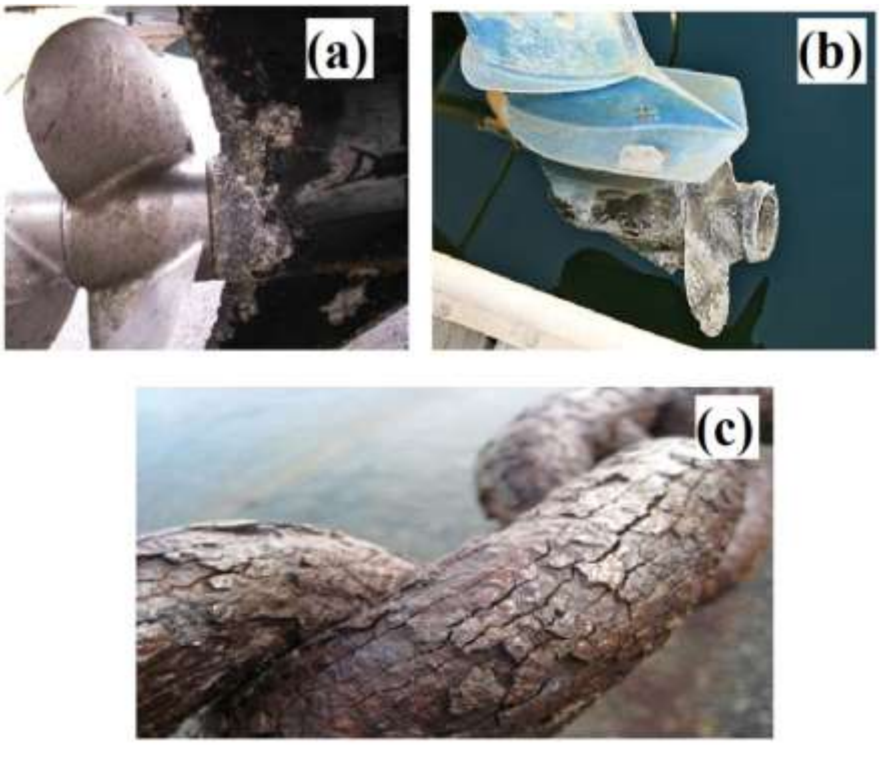

Figure 1.4: Sea water damage due to corrosion of different components, (a) wear on 3-blade fixed propeller (b) heavy rust on the links of a chain near the Golden Gate Bridge in San Francisco (*http://en.wikipedia.org/wiki/Rust*)

1.4 Microwave Material Processing

Percy Spencer, from Howland, Maine in 1945 discovered accidentally the high-power microwave beam specific heating effect. He noticed that a candy bar which is in his pocket was melted by microwaves from active radar started. Various materials like metals, ceramics etc. are processed then and still new possibilities are investigated for new materials to be processed by microwave [14]. The microwave energy is used for processing various materials such as metals, ceramics and also composites. It offers many advantages over the various conventional heating methods. Although, in the processing of the metallic materials the adaptability of microwaves energy is very difficult considering to fact that for metals at room temperature, the microwaves absorption coefficient at 2.45 GHz radiation is considerably very less and the likely thermal instabilities in process can potentially leads to the phenomenon of temperature runaway [15].

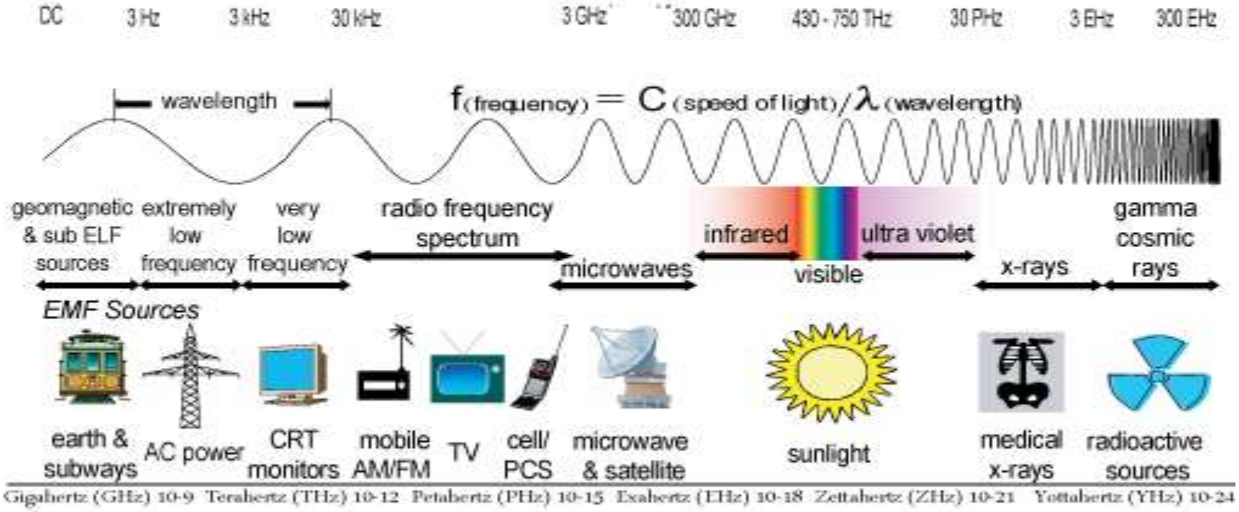

Figure 1.5: EMF radiation spectrum (*http://www.emf-safety.com*)

The Electro magnetic field (EMF) radiation spectrum is shown in Fig.1.5. So without hybrid heating it is extremely difficult to use microwaves for heating metallic materials. Advantages of microwave processing over many conventional heating methods includes the uniqueness in microstructure and the properties, product yield is well improved, savings in energy, manufacturing cost reduction and new materials synthesis.

Recently diversity of microwave heating has been explored in terms of deposition of hardfaced materials on poor tribo characteristic materials and the developed process known as “Microwave

Cladding”. Microwave cladding can be defined as “a process in which electromagnetic wave is used for fusion of a material with some different metallurgical properties onto a substrate, so as to achieve a strong metallurgical bonding” [16]. Properties of the clad have to be maintained by controlling the dilution level of the substrate/clad. Cladding process have wider applications which includes the gas turbine blades corrosion resistance enhancement , diesel engine exhaust valves wear resistance improvement and the dies and inserts repair [17].

Characteristic for microwave cladding is the conflict, on one side attaining no mixing between the clad material and substrate and on other side achieving a very good metallurgical bonding. This means for achieving an acceptable degree of mixing, the heat input with the help of electromagnetic wave (time) must be very well-controlled.

Chapter 2

Literature Review

2.1 Literature Review

The literature review on different surface engineering techniques has been carried and briefly discussed in the present chapter.

Lo et al. (2003)[1] investigated the improvement of wear resistance of AISI 316 stainless steel by laser surface alloying using fine WC powder. Materials used are Austenitic SS 316 as a substrate and slurry was prepared by mixing fine WC powder and a binder. Laser surface alloying was performed using a 2.5 kW Nd:YAG laser, with argon flowing at a rate of 20 l/min as the shielding gas. Fine WC powder was placed on SS 316 and using laser radiation substrate top layer was melted and melt pool formed. Into the melt pool WC powder got dissolved and then solidification of an alloyed layer takes place rapidly in the melt pool. This results in the formation of complex metal carbides ($M_{23}C_6$, M_7C_3, M_6C_2, W_2C & WC) that is verified by XRD.

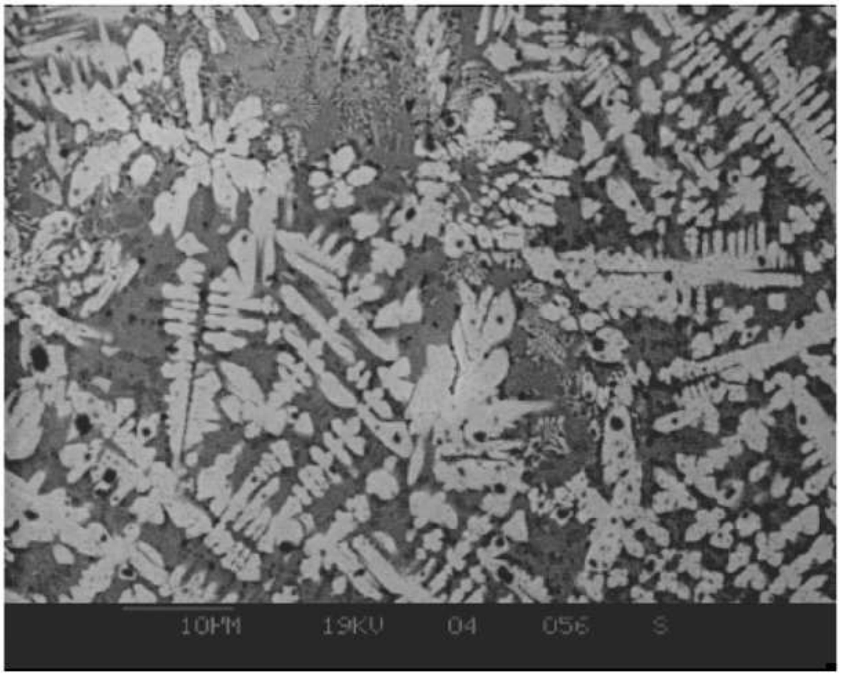

Figure 2.1: SEM of laser surface-alloyed sample WC-SS316 [1]

Alloyed layer has dendrite structure as shown in Fig.2.1. High carbide content is also verified by EDS. As result of dissolution of WC in melt pool there is high concentration of carbon. Dendrite structure was seen in SEM, this is due to precipitation of metal carbides. There is improvement by 30 times of laser surface-alloyed specimen in wear resistance.

Viswanathan et al. (2009) [2] investigated development of cladding on AISI 316L stainless steel of WC-iron silicide (Fe_5Si_3) composite. AISI 316L stainless steel plate (dimension 100×80×5mm)was taken as a substrate material. First using polyvinyl alcohol glue with different compositions of WC-Si-Fe powders slurry was prepared and then material coated on surface of substrate of about 0.5mm average thickness. After drying at room temperature the coated specimen were laser treated with CO_2 laser. For prevention of oxidation of melt pool argon was used as shielding. It is concluded that at laser energy density is 22.5 J/mm^2 and 40WC-40Si-20Fe (wt %) composition, almost uniform hardness distributions averages about 883 Hv. In the clad layer no cracks and pores are observed and also as compared to the substrate the clad have higher wear resistance.

Gupta and Sharma (2011)[3] investigated failure of engineering components due to wear and corrosion, and provided that modification can be done in material by nitriding, carburizing, coating, cladding and other different techniques. In all these the most effective technique found is cladding. The cladding on austenite stainless steel which has good corrosion resistance property has been done by microwave processing. Through microwave hybrid heating (MHH) technique as shown in Fig.2.5 the cladding on austenitic stainless steel SS-316 substrate has been carried out using $WC_{10}Co_2Ni$ powder. $WC_{10}CO_2Ni$ powder of grain size 40 µm was used. The characterization of clad was done through Vicker's microhardness, field emission scanning electron microscope (FE-SEM), X-ray diffraction (XRD) and energy dispersive X-ray (EDS). XRD confirms the presence of WC with cobalt and nickel phase. A domestic microwave oven at a frequency 2.45 GHz and 600 – 900 W power is used in this paper for carrying experimentations.

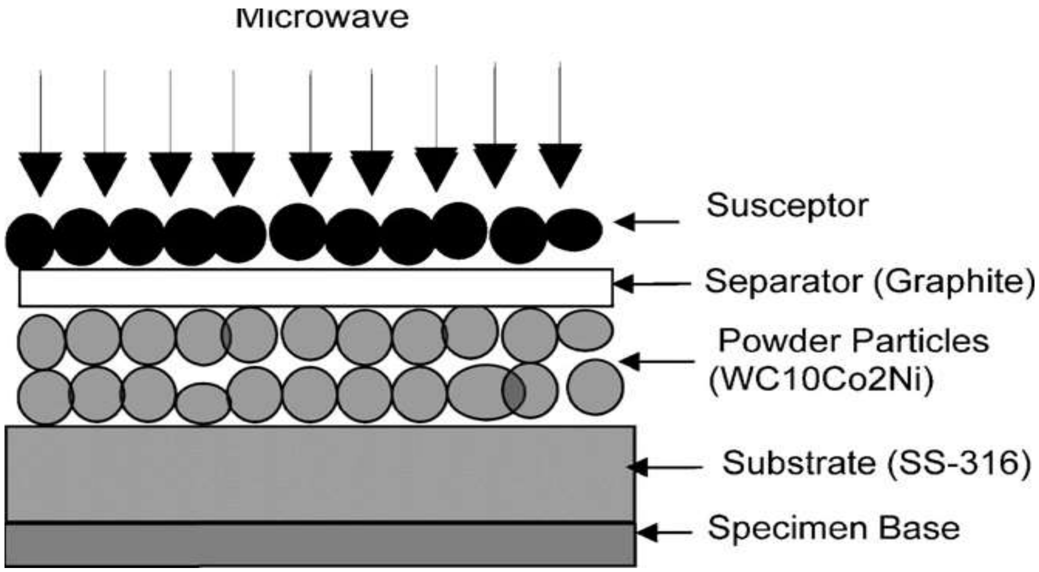

Figure 2.2: Schematic of MHH setup used for cladding [3]

A 99% pure graphite sheet was used to avoid contamination of cladding by susceptor powder used in MHH. Graphite sheet acts as a separator between the $WC_{10}CO_2Ni$ powder and susceptor as shown in Fig.2.2. There is good metallurgical bonding between clad and substrate by mutual diffusion of elements. Clads had a 0.89% porosity and free from cracks. As compared to the SS-316 steel material the microwave clads exhibit wear resistance by 84 times corresponding to 0.5 m/s sliding speed.

Kacar et al. (2004) [4] investigated the explosion cladding of 316L stainless steel. The flyer and parent plates are 316L-type austenitic stainless steel and DIN-P355GH-grade steel, respectively. The arrangement is shown in Fig.2.3.

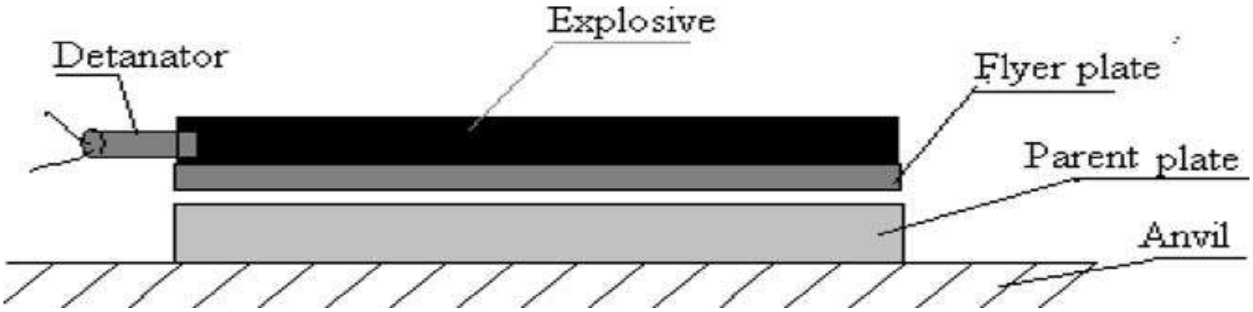

Figure 2.3: Parallel arrangement of experimental setup for explosive cladding [4]

It was found that at cladded metals interface sections there were local melting zones and the hardness also increased while it does not change considerably in either metal within 100µm distance from the interface. This was due to the high level of plastic deformation adjacent to the welded interface. Due to the high plastic deformation the ductility of the explosively cladded metals decreased. Also due to high velocity explosion wave the welded interface had a wavy appearance. Grains were generally elongated closure to the welded interface and parallel to the explosion direction, indicating a high degree of plastic deformation. Due to the excessive plastic deformation done by the explosion wave the microhardness increased 1.5 times near the welded interface.

D.Zhang et al. (2005) [7] investigated laser cladding with Ni-Cr_3C_2 and Ni-WC of martensitic stainless steel for improving wear performance. The substrate material used is martensitic stainless steel. Ni-based alloy powders were used for clad and in scanning electron microscopy (SEM) it appeared to be spherical and the particles were 10–30 µm in size. 20–50 µm was the particle size of Cr_3C_2 powder and WC powder was 1.5–5 µm. The SEM morphology is shown in Fig.2.4.

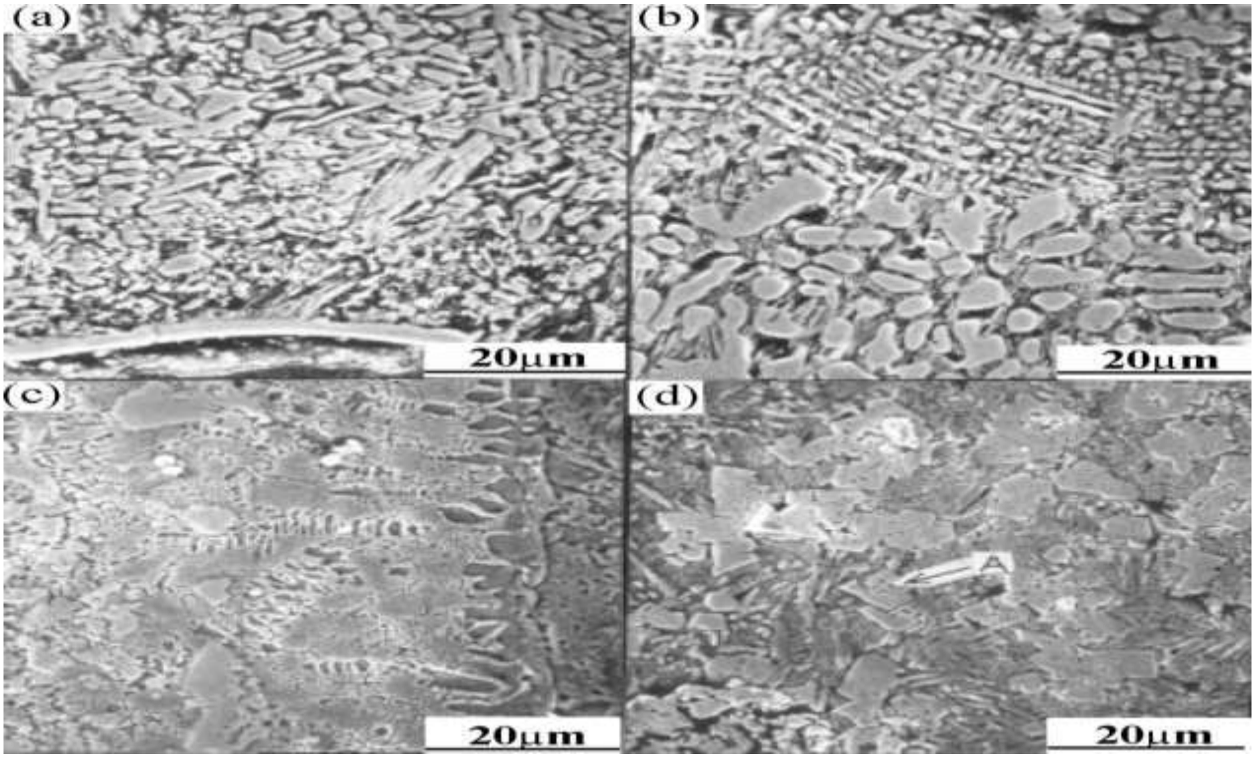

Figure 2.4: SEM micrographs showing typical microstructure of the cross-section specimens [7]

The surface alloy layer of Ni+50%Cr_3C_2 is composed mainly of M_7C_3 (M:Fe,Cr) verified by XRD patterns. Since Cr_3C_2 particles completely dissolved in melt pool this led to increased content of C and Cr in the liquid alloy and hence formation of M_7C_3 phase during rapid solidification takes place. In Ni+50%WC surface alloy near the bonding zone there remain many incompletely dissolved WC particles as compared to that of the Cr_3C_2 surface alloy coating. This is due to the addition of WC (2730°C) particles having high melting point and high hardness and the melting point of Cr_3C_2 (1895°C) is less as compared; it leads to many unmelted WC particles remained in the solidification structure in precipitated form. In case of Ni–Cr_3C_2 microhardness maximum value is about 480Hv near the surface region, and the whole clad layer ranges from 380 to 480 HV; and in case of Ni–WC microhardness maximum value is about 790Hv near the surface region, and the whole clad layer ranges from 680 to 790 Hv. in comparison with substrate the microhardness increases by 1.5 times and 2.4 times average in Ni–Cr_3C_2 and Ni-WC coatings respectively. Due to different features of solidified structure of two coatings they have such hardness differences. As compared with the stainless steel substrate the wear rate of surface alloy coatings Ni-Cr_3C_2 and Ni-WC decreased approx. 30% and 60% respectively.

M.Duraiselvam et al. (2006) [9] investigated laser-clad of AISI 420 martenistic stainless steel with nickel aluminide intermetallic matrix composites (IMC) with TiC reinforcement. The AISI 420 Martensitic stainless steel was used as a substrate. For IMC coating first the powder mixture was prepared with 80 wt.% Ni and 20 wt.% Al and a clad layer is developed; then addition of 10 wt.% TiC for reinforcement. The laser machine is operated at 1.5KW laser power and 100mm/min scanning speed produce specimen Ni-Al-TiC-1 and Ni-Al-TiC-2 when operated at 2KW and 150mm/min. The microstructure in Fig.2.5 showed a strong metallurgical bonding of the clad with good homogeneity and uniformity. The phases mainly consisted in IMC are of NiAl, Ni_3Al and/or Fe_3Al as showed in the specimen XRD spectrum. At room temperature the ductility of nickel aluminide is improved due to the formation of Fe_3Al. This may be caused due to the dilution of Fe from the substrate in specimen Ni–Al–TiC-1 throughout clad matrix the TiC particles in unmelted and partially melted form was mostly distributed. But, the amount of unmelted TiC particles in the specimen Ni–Al–TiC-2 was reduced significantly. There is decrease in resistance against wear in Ni–Al–TiC-1 specimen due to the high content of unmelted TiC particles. Reinforced matrix microstructure is also a very important decisive factor indicated by Ni–Al–TiC-1 poor performance relative to Ni–Al–TiC-2. Hence it is not necessary that only ceramic reinforcement may always improve the erosion characteristics.

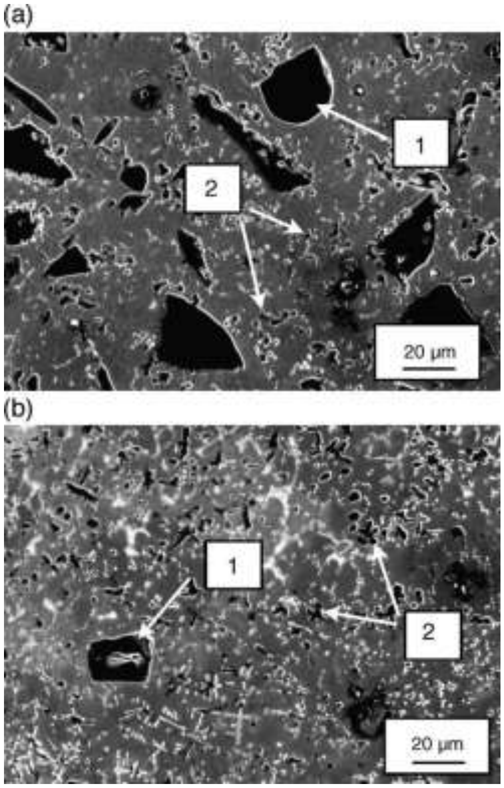

Figure 2.5: Cross-section of nickel aluminide IMC coatings, (a) Ni–Al–TiC-1; (b) Ni–Al–TiC-2 (1 — unmelted TiC; 2 — precipitated TiC) [9]

With the help of pre- and post-heat treatment the pores and cracks in the IMC coatings were completely eliminated. Ni–Al–TiC-1 and Ni–Al–TiC-1coatings has wear resistance 3.3 and 3.6 times as compared to substrate. There was no such correlation found between hardness and wear resistance hence it is concluded that hardness alone cannot be considered as an indicator for wear resistance.

Gupta and Sharma (2011) [10] investigated microwave cladding on austenite stainless steel (SS-316) for enhancing of surface properties. Using microwave irradiation the cladding of nickel based powder (EWAC) was developed. Nickel based material exhibit excellent wear and corrosion resistance. Cladding was developed on S-316 substrate using a domestic microwave oven system with frequency of 2.45GHz and 1KW power. The powder was preplaced manually on SS-316 substrate maintaining an approximate thickness (1 mm). **U**sing techniques like XRD pattern, FE-SEM, energy dispersive, energy dispersive X-ray (EDS) and Vicker's microhardness the clad were characterized. By partial mutual diffusion of elements, there was strong metallurgical bond formed of clad with the austenitic stainless steel substrate. The microstructure of the developed clad has cellular morphology with no transition of cell to dendrites. Formation of nickel silicide (NiSi) and iron nickel ($FeNi_3$) intermetallic phases reported during the microwave irradiation process. Microwave clad had 304±48 Hv average microhardness, which is almost twice that of the substrate. Due to the presence of different phases like chromium carbide in the microstructure there is variation in the microhardness.

Gupta et al. (2014)[11] investigated microwave cladding approach using WC10Co2Ni powder on SS-304 through microwave hybrid heating, the partial dilution of the substrate (austenitic steel) with the cladding material made a metallurgical bonding between the substrate and deposits. Austenitic steels are known for their corrosion resistance properties, this is the main reason of their high demand in industries. But, when the point comes on wear, these materials show poor tribological properties, to enhance this property surface engineering in the form of microwave processing is a well-established technology over the other surface engineering techniques like nitriding, cyaniding and carburising etc. The basic working principle with experimental set up is shown in Fig 2.6.

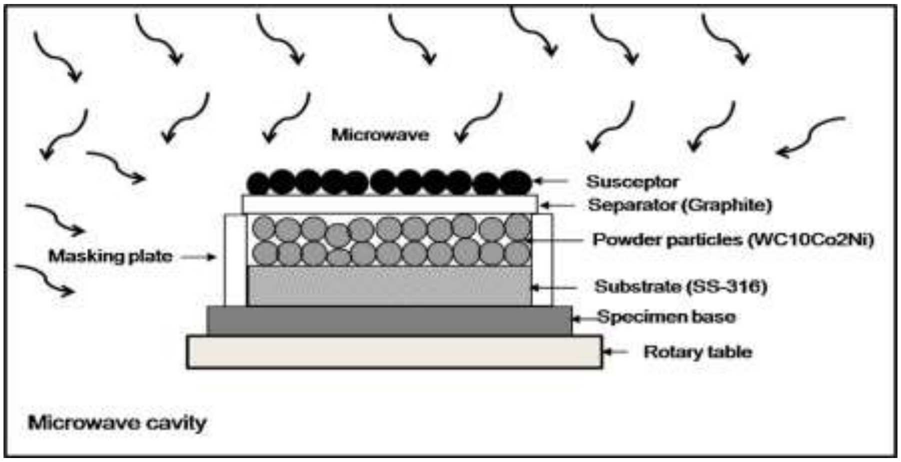

Figure 2.6: Experimental setup for Cladding/Coating through microwave processing. [11]

Because of its excellent feature of minimal dilution and strong metallurgical bonding between the clad layer and substrate material it leads to improved mechanical properties. In this study, the ease of processing and benefits over the laser cladding have also been discussed, which leads to lower setup cost, maintaining cost as well as lower operating cost and higher deposition efficiency. The cracking tendency generally induced in laser cladding processes due to high cooling rate, localised thermal distortion and residual stresses are induced on the substrate; these things can be avoided upto an extent due to furnace cooling. The cladding produced through microwave processing showed an excellent resistance to abrasion and erosion wear due to formation of carbides of tungsten and nickel.

Gupta and Sharma (2012) [12] investigated the composite clad layer on austenitic steel substrate using carbide reinforcement. A domestic multimode microwave oven with frequency of 2.45 GHz at 900 W power was used to develop the tungsten based reinforcement layer. The composite cladding having WC10Co2Ni as the reinforcement and Nickel based material (EWAC) was used as a matrix material. Because of the mechanical properties of nickel such as toughness, corrosion resistance and high oxidation etc. was chosen as a matrix material and on the other hand, WC (ceramic) having high hardness and wear resistance was selected as reinforcement material. The 80% Ni based powder and 20% WC10Co2Ni powder by weight ratio were mixed proportionally in a mixing device.

It was concluded that there is a possibility of metal-ceramic composite cladding layer on a steel substrate using microwave hybrid heating. In the microstructural analysis, it was confirmed that there is a good metallurgical bonding at the interface due to partial melting of the substrate and uniform dispersement of reinforcement on matrix. There was no crack formation even after the ceramic addition. The solidification of the composite layer was in cellular structure, which is supposed to because of volumetric heating associated with hybrid heating. As for as Vicker’s hardness of the clad is concerned, it was achieved upto 416± 20 Hv, which is significantly higher than the soft metallic substrate SS-316 (=200 Hv). Clads exhibits good stiffness and good adhesion with the substrate.

Sharma et al. (2012) [13] investigated cladding through microwave radiation. The resistant to wear of composite (Ni based + 20% $Cr_{23}C_6$ powder) cladding has been developed on substrate austenitic stainless steel (SS-316). Cladding had been developed by conducing the experiments in domestic microwave oven , by the exposure of microwave radiation at frequency 2.45 GHz for duration of 360 s. the composite cladding had shown good metallurgical bonding with the substrate by partial dilution. The back scattered electron image of clad cross section showed the reinforced chromium carbide ($Cr_{23}C_6$) particles are uniformly distributed and well embedded in the Ni based matrix. The clad is metallurgical bonded with the substrate by partial mutual diffusion of elements. The clad is free from visible solidification cracking and possesses significantly lesser porosity which is of the order of 0.90%. The $Cr_{23}C_6$ particles were reinforced in a tougher and ductile metallic (mainly nickel) matrix. The average Vicker's microhardness of developed clad was observed 425 ± 140 Hv. The developed microwave clad SEM micrographs showing typical microstructure of the cross-section specimens.

Sharma et al. (2010) [14] investigated clad development on austenitic stainless steel through microwave processing and studied its effects on the microstructure and flexural strength of cladding developed. It was metal-ceramic composite cladding developed on the surface of substrate. Austenitic steel was taken as a substrate material with Ni based EWAC having 20% WC10Co2Ni as a reinforcing material. Nickel was chosen because of having high toughness property, high oxidation and corrosion resistance at elevated temperatures and WC helps to increase the hardness and wear resistance. Thus, a cladding produced with this composition is supposed to have tensile and compressive stress bearing capability. Through microwave processing, a defect free cladding with 0.89% porosity was developed. This reinforced cladding on a substrate helped to increase the hardness of SS from 200HV to 416±20HV and flexural strength of clad to 629±8N.

Viswanathan et al. (2007) [15] investigated the coating of Si_3N_4 and Ti on AISI 316L stainless steel with laser irradiation. Using different laser-processing parameters and compositions of Si_3N_4 and Ti, the AISI 316L was treated to improve its surface properties. SEM verified Ti-based silicides reinforcement. There is improvement in surface hardness (250-1000Hv) through laser-treatment, also variations in the surface morphology (smooth and bowl like). The presence of pores and cracks depends upon different laser-processing parameters and compositions of Si_3N_4 and Ti. It was concluded that using composition 75–25 wt% of Si_3N_4–Ti and the laser-processing parameters are 1.5KW laser power and 1.0m/min scan speed, there is smooth surface morphology with improvement in hardness of coating about 800Hv. The coating region is free from cracks and pores. Between coating zone and substrate a good interfacial bond is observed and has retained austenitic structure reinforced with titanium silicide (Ti_5Si_3), which decrease the corrosion and enhance wear resistance.

Riveiro et al. (2013) [18] investigated optimization of laser cladding for Al coating production. The production of aluminum based coatings on a stainless steel (AISI 304) substrate by laser cladding, using a high power diode laser was experimentally studied. The costs distribution during laser cladding of aluminum using a diode laser is shown in Fig.2.6. It was observed that the filler material and gas has maximum investment.

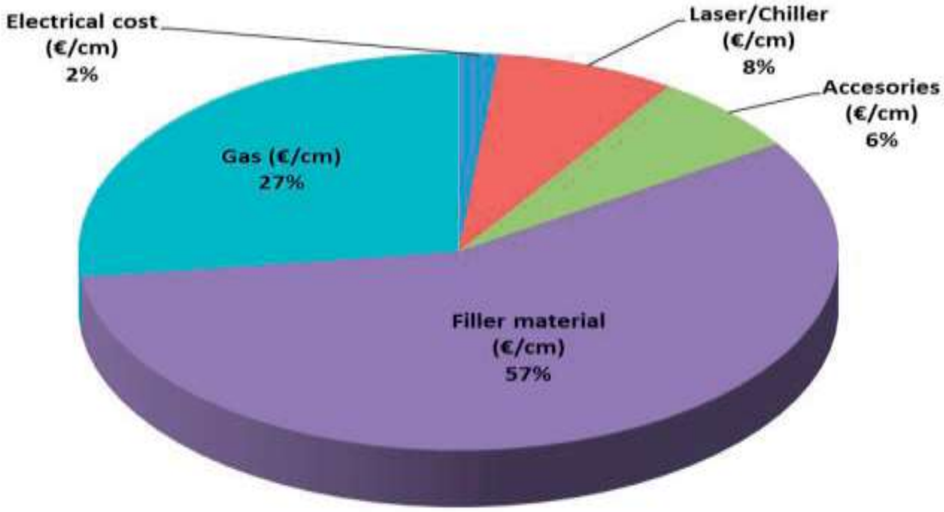

Figure 2.7: Distribution of costs during laser cladding of aluminum using a diode laser [18]

In this cladding technique a carrier gas was used for blowing particles of the precursor material over a substrate. With regard to the laser beam and powder flow the substrate is moved. A stationary high-power laser beam heats up the precursor material and creates a molten pool on the substrate where the particles impinge. On the other hand, a shielding inert gas is injected into the interaction zone in order to avoid the oxidation of the molten material. It is concluded that the influence of each parameter on the total cost of the process reveals the filler material as the most influential parameter.

Mordike et al. (2006) [19] investigated plasma-sprayed NiTi coating cavitation erosion. NiTi with exceptionally long incubation periods (passed cavitation time without measurable mass loss) shows high resistance to cavitation erosion as well as to wear. The surfaces of stainless steel (316L) substrates were sand blasted to increase the sticking ability of the coatings. Coating powder material consisted of an alloy Ni: 50.6/Ti: 49.4 (at.%). To achieve Ni-Ti coating on 316L steel substrate vacuum plasma spraying was applied. Distilled water was used for the cavitation erosion test using a vibratory cavitation device. The main phase was identified with a near equiatomic composition as NiTi. For the large white areas characterizing the Ni_3Ti phase and a Ni content of about 75 at.% was measured. For the dark gray phase it was determined that the Ni:Ti ratio is 1:2. A slightly increased oxygen peak provides evidence in favor of the oxide $Ni_2Ti_4O_x$ which cannot be quantified. A metastable precipitation of the Ni_4Ti_3 phase typically formed. During the first 10 h of exposure to cavitation coating showed mass loss this is due to the removal of low adherence particles in the pores. Coating showed localized damages in pores by growth and coalescence after the cavitation test.

Islak et al. (2005) [21] investigated the low temperature plasma nitro-carburising (LTPN) of AISI 316 austenitic stainless steel. Materials System used are Austenitic SS 316 as a substrate and nitrogen, hydrogen and methane gases. Both nitrogen and carbon are employed as alloying species in the process of plasma surface engineering. The process produces a precipitation free hard layer since austenitic stainless steel introduced with nitrogen and carbon alloying elements. The process was carried out at low temperature to obtain a precipitation-free hardened layer on 316 stainless surfaces with excellent corrosion resistance, thick layer and high hardness. The process improves the hardness and wear resistance of austenitic stainless steels without a compromise in their corrosion resistance.

2.2 Summary of given Literature

1. Several authors worked in different fields to improve the surface quality of different materials like stainless steel, mild steel, aluminum, etc. since all these materials are part of our daily life utilizations. Surface engineering techniques used to develop a wide range of functional properties at the required surfaces. **(Lo et al.-2003, Kacar et al.-2004, Gupta and Sharma-2011)**

2. Several authors have investigated the improvement in mechanical properties and surface behavior resulting from changes in content in the cladding material and substrate. The hardness, microstructure, wear resistance, corrosion resistance have been reported to show significant changes with different cladding techniques. Authors have worked to find the specific levels of cladding which could provide an optimum combination of the properties. **(Xunhong et al.- 2013, Gupta and Sharma-2012)**

3. A number of methods are used for surface engineering like electroplating, plasma nitro-carburizing, laser cladding, microwave processing, explosion cladding**,** laser welding or welding using advanced heat sources like plasma, laser, pulsed arc etc. **(Cheng et al.-2005, Viswanathan et al.-2009, Gupta et al.-2012)**

Chapter 3

Research gaps and Problem Formulation

3.1 Introduction

This chapter covers the detailed objectives of the proposed research work. It presents the overall objective of the proposed research, methodology and experimental procedure to be adopted. The objectives of research work are to be set by keeping in mind the research gap in literature.

3.2 Research Gap

The study on the surface behaviour of the materials is one of the distinguished fields of research. The researchers/ academician have explored and worked on various methods of surface improvement techniques to achieve goals like good tribolological behaviour of surfaces with enhanced physical, chemical and mechanical properties.

The present literature survey reported gaps and limitations on hardfaced materials deposition techniques which are presented as follows:

1) The process like laser cladding and thermal spraying leads to severe cracks and porosity with thermal stresses.
2) In literature review it has been found that there is a lack of cladding process to overcome the limitations of the existing well matured processes.
3) Microwave cladding technique has the potential to overcome the limitation but very limited work has been carried out in this area.
4) No work has been carried out on enhancement of tribo charactristics of martensitic stainless steel through microwave cladding. Very less work has been carried out on deposition of composite metallic materials through microwave cladding.

3.3 Establishment of the objectives

The literature review reveals that the problems in conventional thermal processing methods include overheating the surface since heating of material takes place from outer surface to the interior and it is more time consuming. In conventional thermal processing methods like in case of laser cladding, the laser cladded surfaces generally have crack during solidification this is due to high thermal stresses. These methods have high residual stresses and also have thermal distortion. These methods have less control over dilution level. So there is a need of development of some new techniques or/and modify the conventional thermal processing methods to get better results.

In material processing techniques the microwave processing has unexpectedly rise as one of the fastest. The microwave processing of materials is very much different as compared to the conventional thermal processing methods. In microwave processing uniform bulk heating is done by generating heat at molecular level, where as in the conventional heating methods heating of material takes place from the surface to the interior. In the conventional heating methods there is higher thermal gradient than microwave processing, which might result in poor microstructure. Bulk or volumetric heating is the main characteristic of microwave processing which as compared to other thermal processes reduces the likeliness of developing residual stresses, porosity, cracks, and thermal distortion on the base material. The main problem basically arise with microwave heating of metals at 2.45 GHz radiation is that at room temperature there is very low microwave absorption coefficient for metals. Also thermal instabilities, which can possibly cause phenomenon of thermal runaway. The objective of the present work has been decided under the mentioned limitations of microwave processing.

The main objective of proposed research work is surface modification or wear resistance enhancement of martensitic stainless steel by depositing a suitable hardfaced clad materials layer through microwave heating.

Chapter 4

Material Selection

4.1 Introduction

In the literature a complete overview is reported for different combinations of the clad powder materials and the base or substrate materials. Selection of material is done on the basis of objectives decided to be achieved. It is basically based on the desirable properties of any component under its functional environment. Selection of clad powder is very important aspect so as to achieve a defect free and durable clad layer.

Since the chemical composition and microstructure of the clad layer strongly depend on the cooling rates during solidification and degree of mixing; to the microwave cladding process the parameters controlling these mechanisms are much important. Steps for selection of parameters and properties are shown in Fig.4.1.

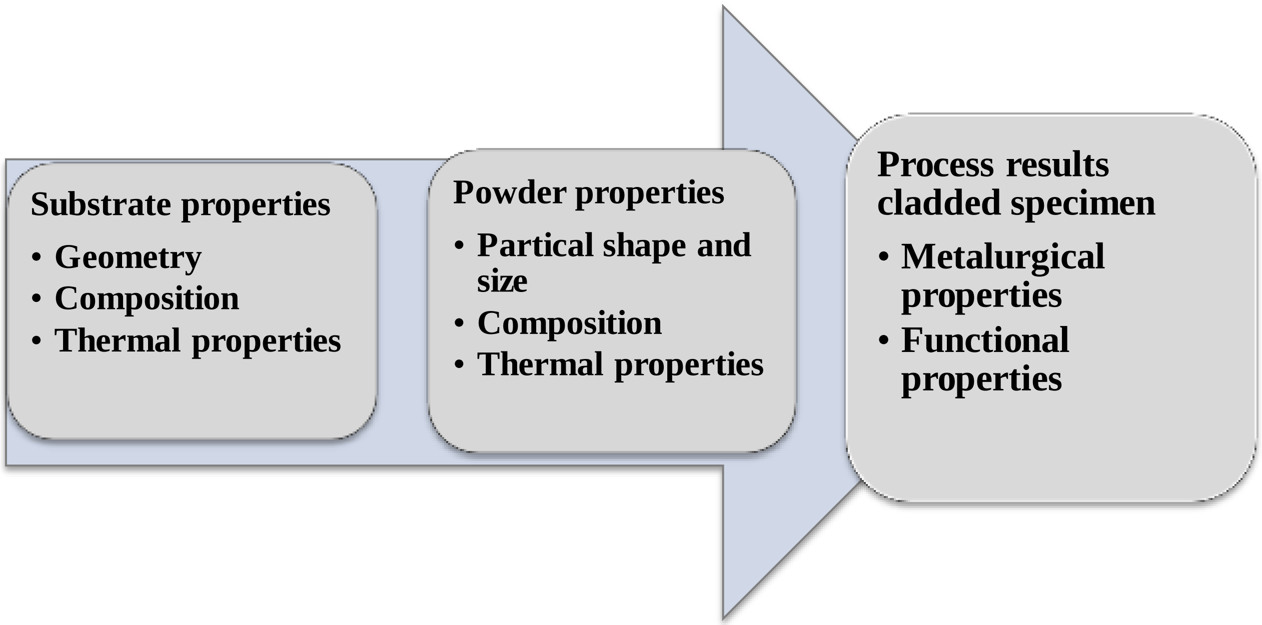

Figure 4.1: Steps for selection of parameter and properties for microwave processing

Material properties that affect these mechanisms are their thermal conductivity and their melting point, among others. The main reason for cracks formation in the clad layer is due to the thermal stress. These thermal stresses are the result of difference between thermal expansion coefficients and high thermal gradient that got built up while cooling stage. The presences of brittle and hard particles, such as carbides in clad layers are liable to cracking.

4.2 Properties of the Clad Layer

Metallic hardfacing powders for cladding are available commercially in wide varieties. The properties of clad can be basically classified in four groups as shown in Table 4.1. On the basis of these desired clad layer properties clad powder selection is done in the present work. Some properties of the clad may be inter-dependent. The hardness, the bonding between clad material and substrate, the microstructure, also the cracks number and cracks depth and their direction etc. affect the wear mechanism.

Table 4.1: Clad Layer Properties

Clad layer Qualitative properties	**Clad layer mechanical properties**	**Clad layer geometrical properties**	**Clad layer metallurgical properties**
• Cracking in clad • Porosity in clad	• Hardness distribution in clad • residual stress in clad • wear resistance of clad • tensile strength of clad	• dimensions of clad • dilution of clad • roughness of clad	• microstructure of clad • dilution of clad • grain size • homogeneity in clad • corrosion resistance of clad

Practically, it is very much difficult to create a clad layer which can meet all the functional requirements. Basically between several properties a balance must to be found.

4.3 Selection of substrate and clad powder

In present research work the martensitic stainless steel (SS-420) has been consider for enhancement of wear properties. The martensitic stainless steel is widely used in hydro power plant and machineries. The frequent failures of the components are very common and owing to which system shut down will be occurred. Hence, surface modification is the best solution to overcome these issues. The clad powder materials like nickel based, silicon carbide and aluminum has been selected for the present work. The average particle size of powders was 50µm and morphology is shown in Fig.4.2. SEM images shows that nickel powder is spherical in nature whereas alumina and silicon carbide is in the form of irregular particulates. Nickel base powder is best suited for elevated temperatures and aggressive atmosphere. The Ni-based powder has a good oxidation resistance and high temperature corrosion resistance. Silicon carbide powder is very hard in nature and aluminum powder can impart good atmospheric corrosion resistance.

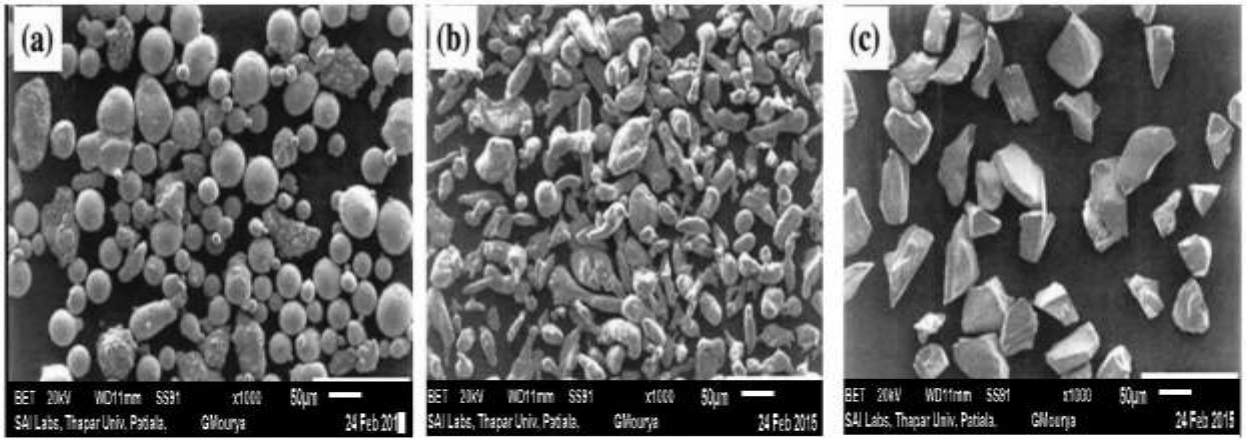

Figure 4.2: Morphology of raw cladding powders (a)Ni based, (b) Al powder and(c) SiC powder

With the addition of silicon carbide the wear resistance and hardness are improved due to formation of hard carbides. Silicon improves the wetting behavior hence helps to achieve a very smooth surface. Mostly for engineering applications like nuclear reactor, multipurpose tools, turbine blades etc. stainless steel 420 is used since it has better hardness, corrosion resistance etc. as compared to other grades of stainless steel.

Chapter 5

Methodology

5.1 Introduction

This chapter presents the step wise procedure which is used to accomplish the research objectives, The plan of work was started with the selection of materials through literature review and then development of clads. The detailed methodology for the proposed work comprises of two main phases:

Phase 1

In the first phase the selection of substrate material and powder for cladding was carried out such that their compatibility has no issue and satisfies the proposed objectives. Materials selected were such that the desired properties would be achieved. In this phase the developments of clads were carried out on substrate steel through microwave hybrid heating technique.

Phase 2

In the second phase of work, characterizations in terms of microstructural analysis, mechanical testing and tribological properties were analysed on the microwave developed clads. The developed clads were cut through the cross section and were polished using fine grades of emery paper. Available techniques of X-ray diffraction, Scanning Electron Microscopy, Energy Dispersive Spectroscopy were utilized for studying the phases of developed clads and study of microstructures. Micro hardness was carried out in order to study the effect of cladding powder and microwave processing. Dry sliding wear test were conducted at varying sliding speeds and sliding distances. Further, normal load was varied to study the effects of load on wear phenomenon of developed clad.

5.2 Experimental Procedure

By using the microwave heating the development of cladding is a challenging task. It is very difficult to develop a clad considering to fact that metals at room temperature reflects the microwaves due to lower absorption coefficient at 2.45 GHz frequency. To overcome this problem, microwave hybrid heating technique was utilized. In the present work, wear resistant nickel based claddings are developed on SS-420 by using domestic microwave oven. The following sections completely describe the procedure for development and characterization of composites. The plan for clad development and characterisation is shown in flow chart represented by Fig.5.1.

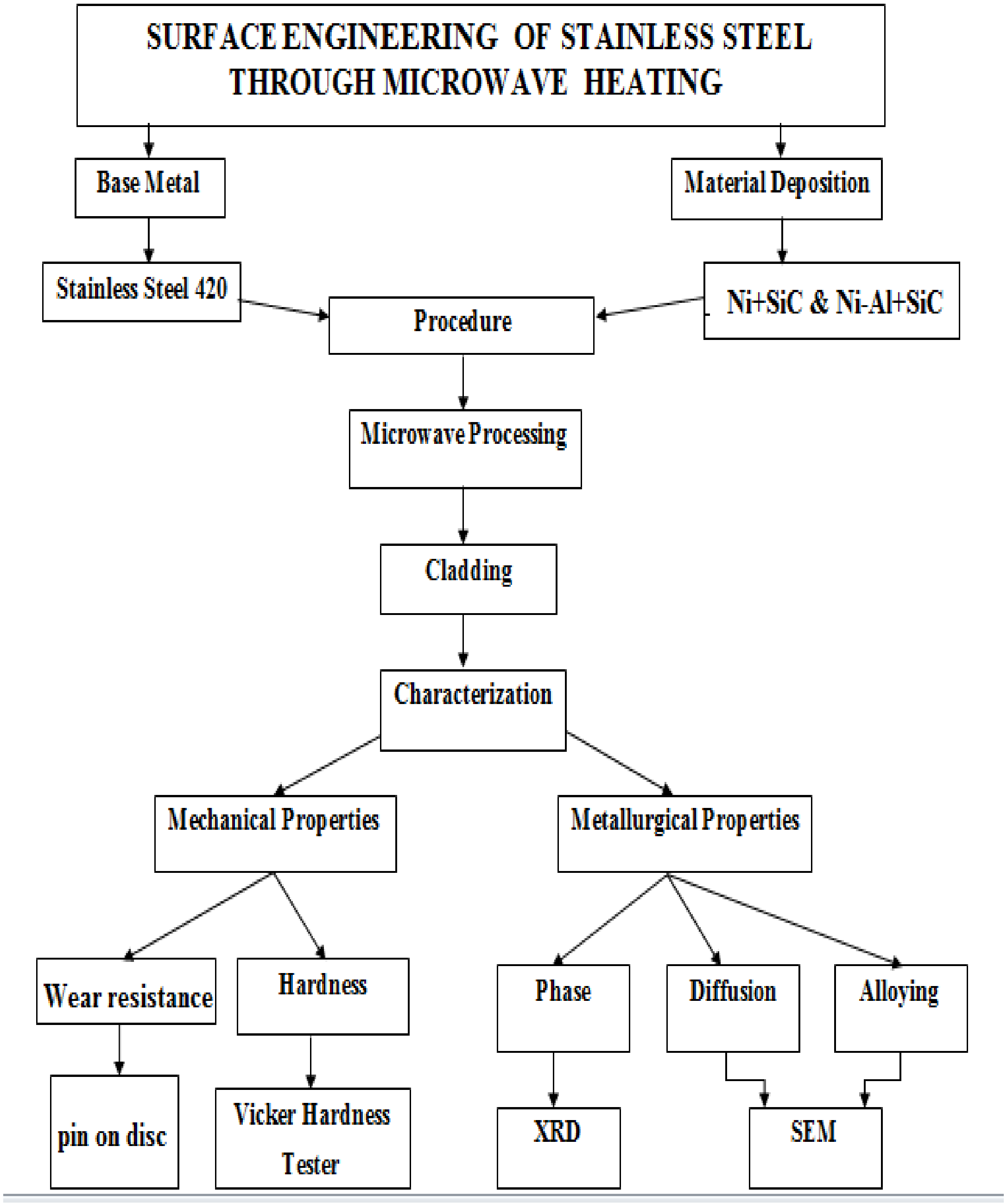

Figure 5.1: Flowchart showing the plan for clad development and characterisation

5.2.1 Microwave Hybrid Heating (MHH)

In the processing of the metallic materials the adaptability of microwaves energy is very difficult considering to fact that for metals at room temperature, the microwaves absorption coefficient at 2.45 GHz radiation is considerably very less. So a passive heating is done through material having considerable microwave absorbing source called susceptor. The schematic principle for microwave hybrid heating setup is shown in Fig.5.2

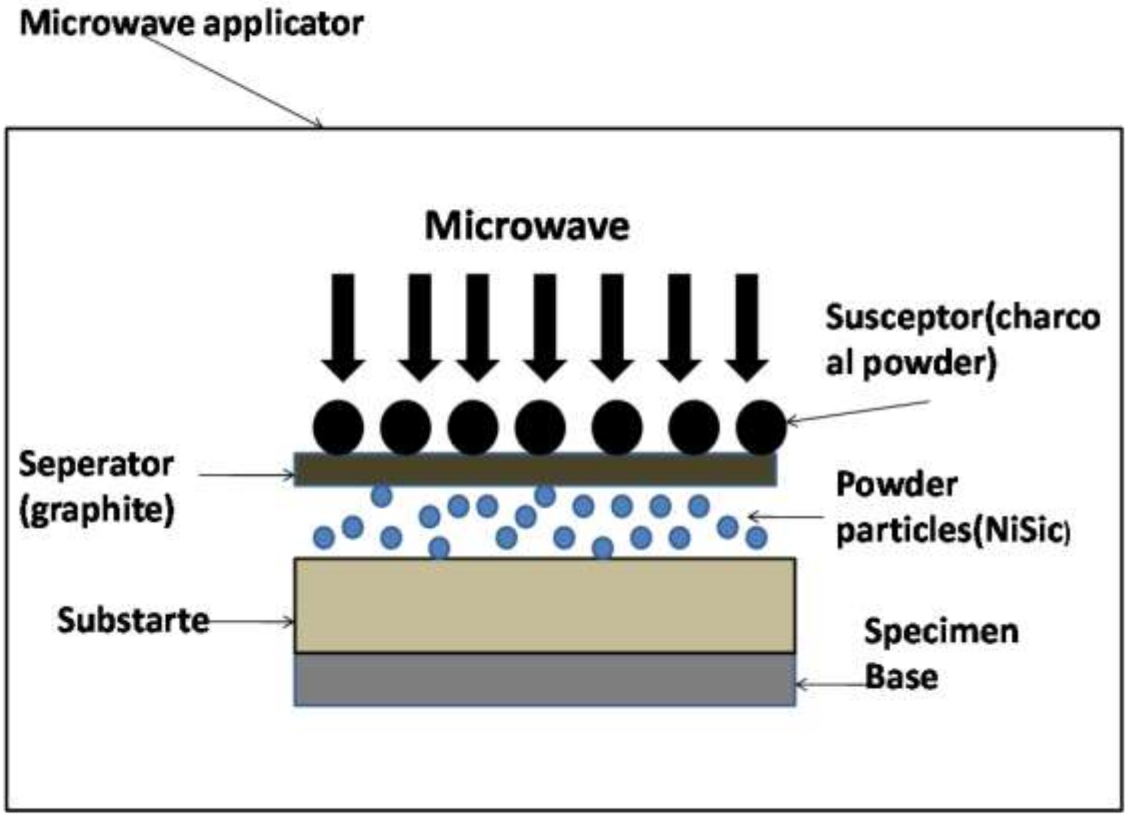

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Figure 5.2: Microwave hybrid heating setup

On martensitic stainless steel 420 substrate cladding was done by microwave processing. Clad powders used are nickel based powder with 10% silicon carbide (Ni+10%SiC). In case of intermetallic matrix composite cladding first a clad is developed of 80% nickel and 20% aluminum on martensitic steel and then again cladding is done with Ni-Al+10% silicon carbide reinforcement (Ni-Al+10%SiC). Steps followed for the clad development process are:

I. First preheat the clad powder in microwave under convection mode. Since preheating reduces the moisture from the powder. Hence there is reduction of porosity in clad layer.

II. Clad powder is then manually dispersed on the SS-420 that is cladding powder which is properly mixed is spread over the substrate. Another important parameter in any cladding process is mixing and spreading of powder on substrate. If this is not mixed properly it leads to cracks, poor bonding and porosity in clad surface.
III. This preplaced clad powder substrate is then placed on the ceramic brick.
IV. Then a graphite sheet is cut, having exactly same dimension of the substrate. This graphite sheet is then placed on the top of preplaced cladding powder substrate. It is used to separate the clad powder from charcoal and provide conventional heating of powder.
V. The specimen is the covered by susceptor material.
VI. Microwave power was set on 900W and for a time period of 300 seconds.

In microwave processing, electromagnetic wave is introduced directly to the material. As electromagnetic wave is introduced the charged molecules get align itself with the field and as the polarity of wave changes with each wave cycle from positive to negative, this causes rapid and continuous orientation of the molecules that result into heating of material by collision between molecules. From the literature it also followed that the time applied for microwave cladding also affect strongly the properties of clad layers.

Formation of a melt pool starts in clad material. Then the melt pool propagates from the interface and extended to the substrate, continued heating ensures that and fusion bond achieved is strong. To prevent substrate deep melting the heat input (time of processing) must be well controlled to achieve a well strong fusion bond.

5.2.2 Polishing

Polishing machine is shown in Fig.5.3. The first step after clad preparation is to polish it for carrying out different studies rotating wheels. There are two rotating wheels, one wheel is covered using soft cloth which soaks the polishing medium and the second wheel is used for mounting the ambry paper of different grades. For getting mirror like, polished and scratch free specimen, it is held on the rotating disc.

Figure 5.3: Polishing Machine (Courtesy: CNC Lab, Thapar University)

The cloth used for polishing is washed with water before starting of polishing; this is done to avoid any chances of contaminants introduced which may also cause deep scratches on surfaces.

5.2.3 Hardness Testing Machine

The hardness of the specimen is used to measure Vickers hardness tester as shown in Fig.5.4. Test samples must be prepared carefully and properly by grinding and polishing. In Vickers micro-hardness test procedure an indenter creates the indentation with a range of loads. By measuring the indentation the hardness value is calculated. After the indentation is completed, the two diagonals are measured and the average value is considered.

Figure 5.4: Vickers Micro-hardness Tester (Courtesy: A.M. Lab, Thapar University)

Vickers Hardness Number (VHN/ VPN) is obtained by dividing the applied load in kilogram-force by surface area of indentation in materials. The indenter used for indentation can be used for all materials irrespective of hardness of materials.

5.2.4 Scanning Electron Microscope (SEM)

By using SEM the scanning of specimen is done with high-energy beam of electrons. Some signals produced by interaction of the electron and atom of specimen that contain information about the characteristics of the specimen's surface morphology, composition and other properties. It can produce the specimen's surface images which are having very high-resolution, revealing less than one nano meter in size details.

Figure 5.5: Scanning Electron Microscope (Photo courtesy: SAI Labs, Patiala)

SEM as shown in Fig.5.5 (Make: JEOL JSM-6510LV, Oxford Instruments) is available at SAI Labs, Thapar Technology Campus, Patiala is used for analysis. It is a high-performance and low vacuum SEM for fast characterization and imaging of fine structures having magnification from 5X to 300,000X.

5.2.5 X-Ray Diffraction (XRD) Machine

X-ray diffraction technique is one of the most powerful tools for qualitative and quantitative analysis of materials. The XRD machine will be used for studying phase type, the volume fraction of phase, grain size measurement, and other minute details. X-ray diffraction shows unique fingerprints associated with the crystal structure. It is a non-destructive analytical technique.

Figure 5.6: X-ray Diffractometer (Photo courtesy: SAI Labs, Patiala)

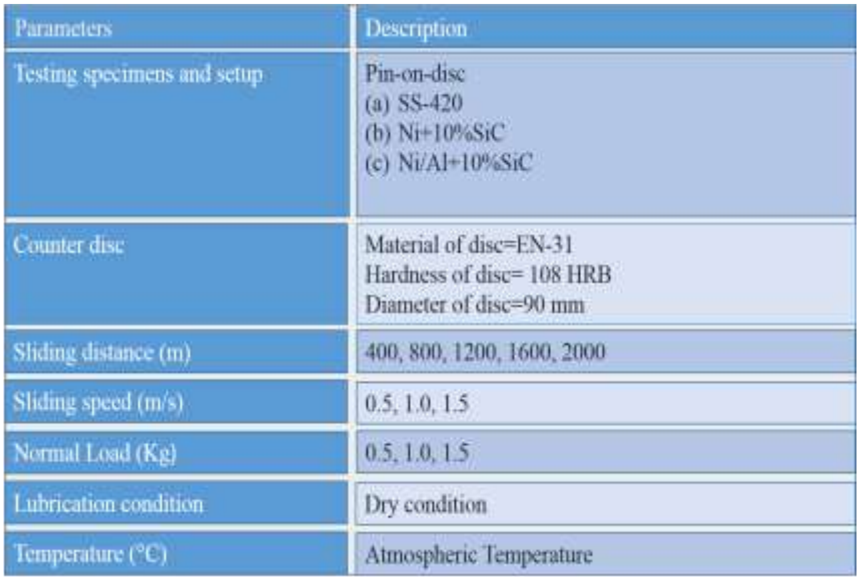
X-ray diffractometer as shown in Fig.5.6 (Make: X'Pert PRO, PANalytical) in SAI Labs, Thapar Technology Campus, Patiala is used for obtaining the quality diffraction data.

5.2.6 Wear Study (Pin-On-Disc Tribometer)

A pin on disc tribometer (Ducom India) as shown in Fig.5.7 used to calculate the dry wear characterstics of the clad. Clad specimen having dimensions (8mm × 8mm × 6mm) and samples clamped in the square clamp (V-shaped) against the rotating disc. The rotating disc was prepared from EN31 steel. The rotating disc as well as samples was cleaned with acetone prior to start the testing.

Figure 5.7: Tribometer setup for wear study (Courtesy: Machine tool Lab, TU)

Table 5.1: Details of Sliding wear parameters



Parameters	Description
Testing specimens and setup	Pin-on-disc (a) SS-420 (b) Ni+10%SiC (c) Ni/Al+10%SiC
Counter disc	Material of disc=EN-31 Hardness of disc= 108 HRB Diameter of disc=90 mm
Sliding distance (m)	400, 800, 1200, 1600, 2000
Sliding speed (m/s)	0.5, 1.0, 1.5
Normal Load (Kg)	0.5, 1.0, 1.5
Lubrication condition	Dry condition
Temperature (°C)	Atmospheric Temperature

The standard test conditions and all related parameters are shown in the Table 5.1.Wear study is to be carried out at different speeds and by varying the loads. Wear behavior is to be studied for sliding distances (400m, 800m, 1200m, 1600m and 2000m) and with three different loads (0.5 Kg, 1Kg and 1.5 Kg) and sliding speed (0.5 m/s, 1m/s and 1.5 m/s) without any lubrication. Before measuring the weight of samples, they were cleaned with the acetone and dry air. By using electronic balance with ±0.1 mg accuracy weight is to be measured after every 400 m of sliding distance.

Chapter 6

Results and Discussion

6.1 Introduction

Microwave claddings of Ni+10%SiC and Ni-Al+10%SiC were successfully developed on martensitic stainless steel by using microwave hybrid heating. The following section describes the characterizations of the developed clad**.**

6.2 X-ray Dffraction Studies

X-ray diffractometry (XRD) is used to identify the phases developed in the clad region due to intense heating which leads to the formation of intermetallic. A typical XRD spectrum and their relevant study for the claddings developed through microwave hybrid heating are presented below.

6.2.1 XRD Study of Ni+10%SiC Cladding

From Fig.6.1, Presence of free silicon (Si) is clearly identified in Ni+10%SiC cladding. It is attributed to possible decomposition at higher temperature during MHH from starting powder to

$$SiC \longrightarrow Si+C \qquad (1)$$

This carbon reacts with atmospheric oxygen results into the formation of CO, which during solidification (slow cooling rate) escapes subsequently leading to crack free cladding and less porosity. On the other hand, XRD spectrum of the developed clad also shows the presence of Ni_3Si, CrNi, $Cr_{26}C_6$, Fe_2Si and Ni_3Fe.

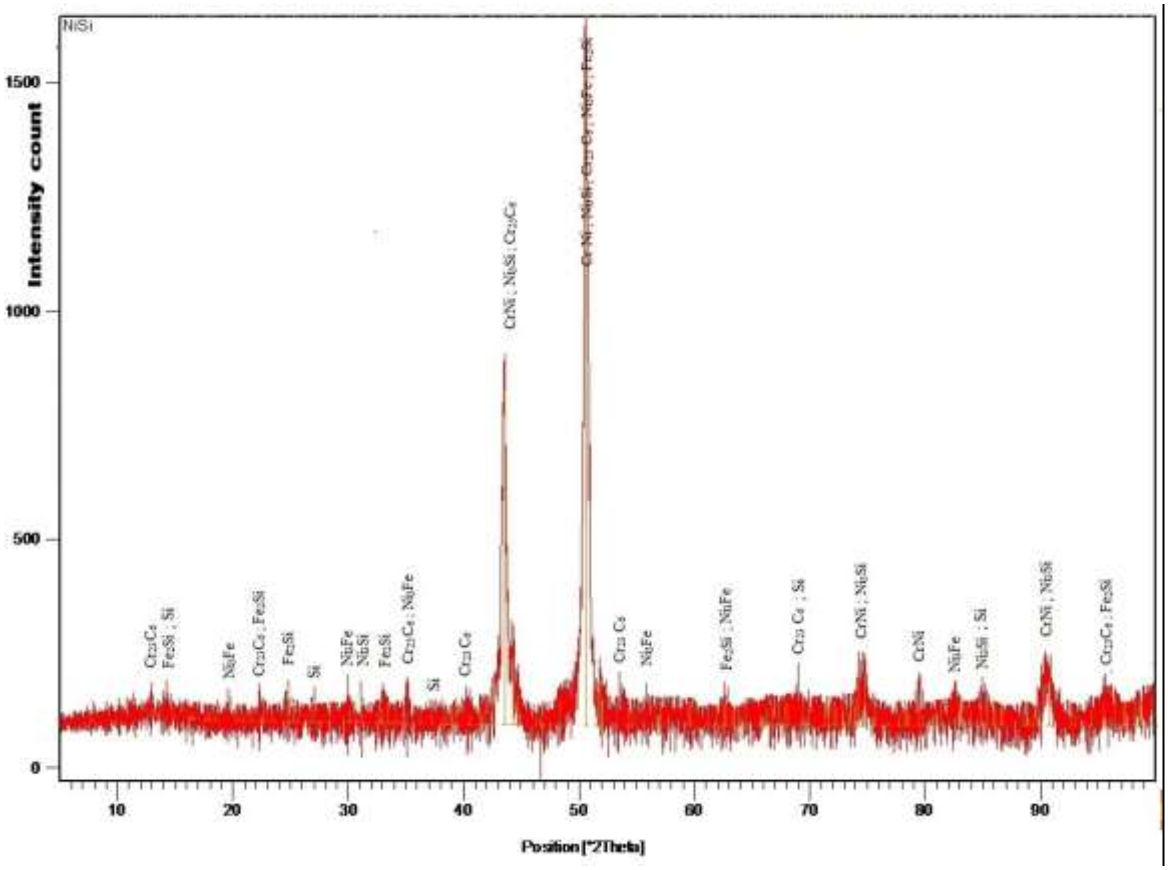


Figure 6.1: A typical XRD spectrum of the Ni+10%SiC cladding

During the microwave interaction the partial mutual diffusion of Fe and Cr could be attributed from the substrate to clad. Chromium carbide formation takes place due to the presence of free carbon in the nickel-silicon carbide starting powder which reacts with the chromium during the irradiation and constitutes the major portion in the clad. Also elemental dilution of iron from the substrate results into the formation of Fe_2Si (intermetallic).

6.2.2 XRD Study of Ni-Al+10%SiC Cladding

The XRD spectrum of the cladding shown in Fig.6.2 illustrates that the intermetallic matrix composite coating mainly consisted of Ni_3Al, NiAl, Ni_3Si, Fe_3Al and SiC phases. The development of Fe_3Al is associated with the dilution of Fe from the substrate material.

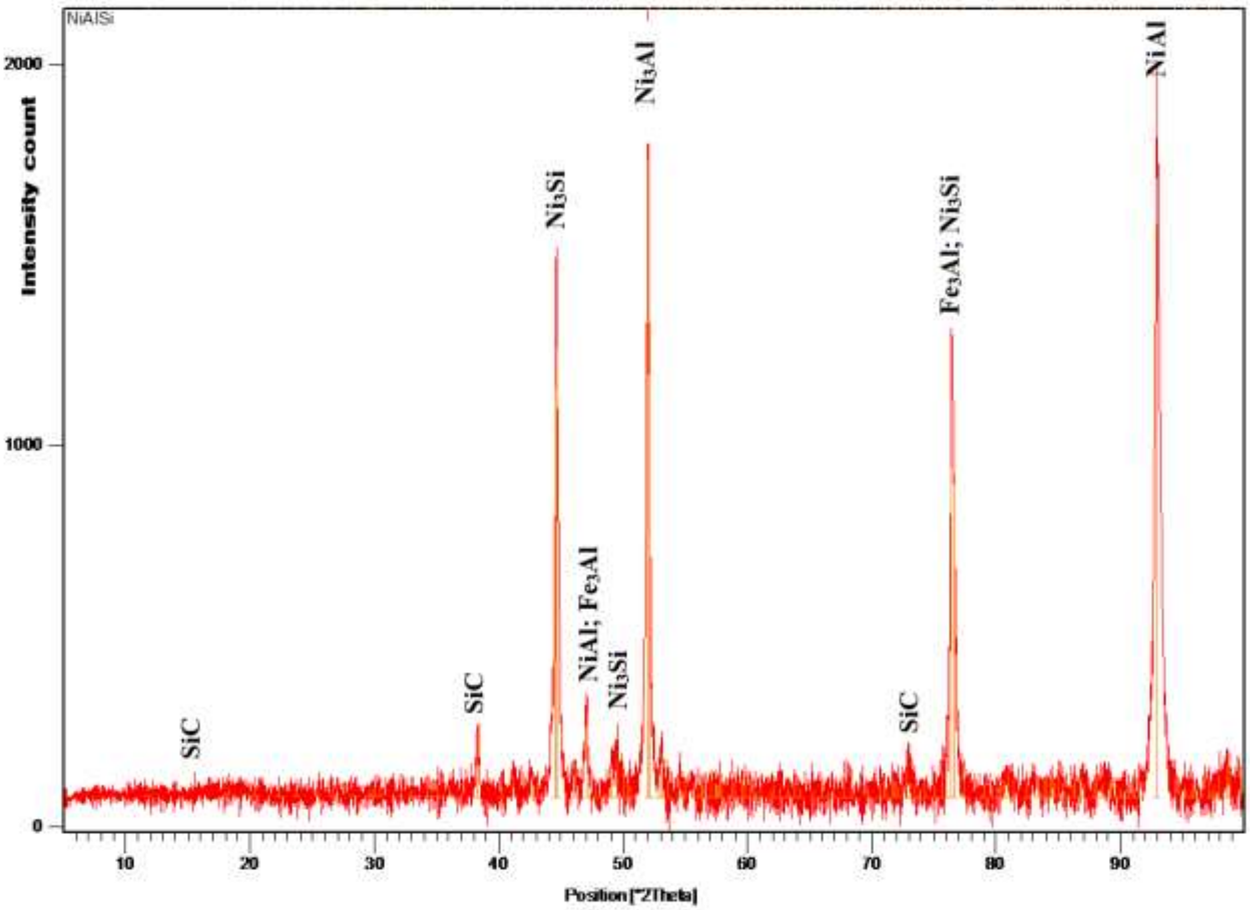


Figure 6.2: A typical XRD spectrum of the Ni-Al+10%SiC cladding

The development of Fe_3Al is associated with the dilution of Fe from the substrate material. Here also the silicon carbide dissociation takes place. Some of the SiC particles may not get dissociate and remains in same form. These SiC particles may be in both precipitated and melted form

6.3 Cladding Microstructure Observations

Microstructure observations of claddings specimen are carried out using scanning electron microscope (SEM) analysis. In SEM analysis scanning of specimen is done with a high-energy beam of electrons. Before SEM analysis specimens were sectioned using diamond cutter and mirror polished by using emery papers of 180,300,600,1000,2000,3000 grits finally followed by diamond paste polishing. SEM images of the developed clad is shown below.

6.3.1 Ni+10%SiC Cladding Microstructure Study

The SEM image of a typical transverse section of the Ni+10%SiC cladding is illustrated in Fig.6.3(a). The clad of thickness ~ 1.25 mm exhibits good metallurgical bonding with the substrate by partial mutual diffusion of elements.

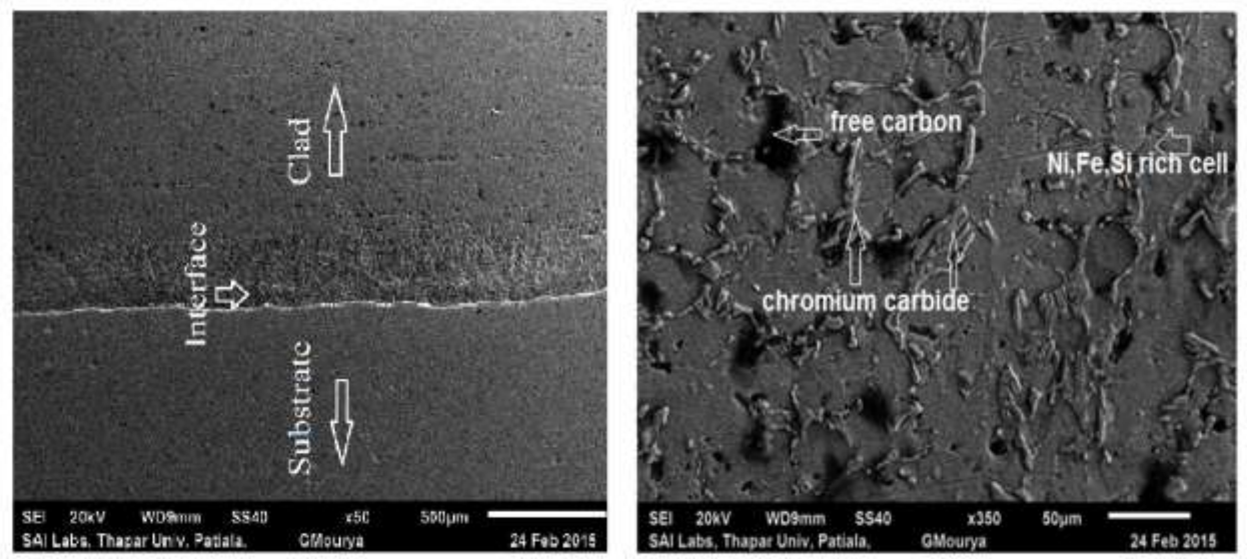


Figure 6.3(a): SEM morphology of Ni+10%SiC clad section (at 500µm and 50µm) showing carbide precipitation at cell boundaries with free carbon

The results of EDS analysis is shown in Fig.6.3 (b), which confirm the presence of high chromium and carbon in form of chromium carbide in Ni, Fe, and Si rich cell. Typical grain structure with dendritic grains is clearly seen. The microstructure shown in Fig.6.3(a) further depicts crack free clad, which indicates uniform volumetric heating associated with microwave heating. The linear point count method was used to measure the porosity of developed clad which showed the significant less porosity of 0.90% which is significantly less while compared to the thermal spray processes.

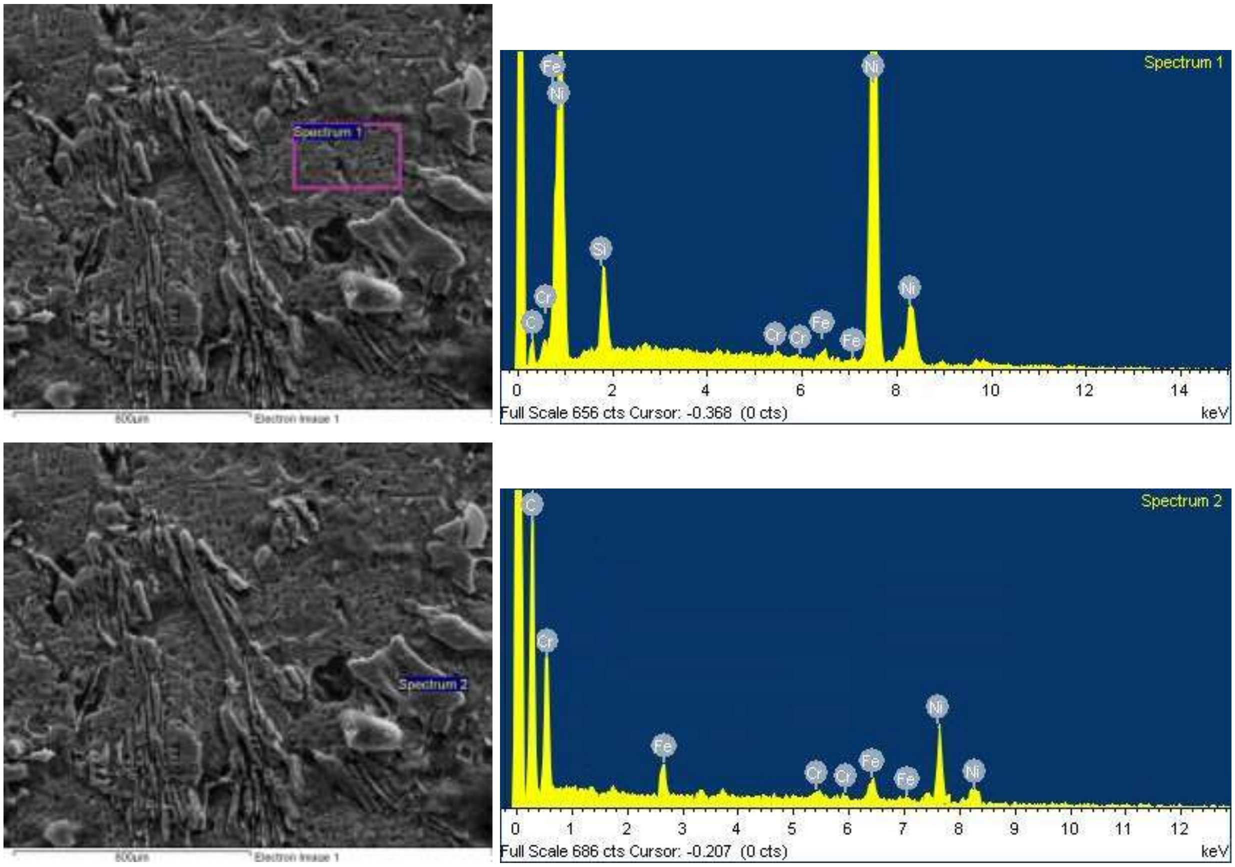


Figure 6.3(b): EDS of Ni+10%SiC clad section

During microwave hybrid heating gases may diluted in the molten metal. But during solidification of metal solubility of gases may reduce, hence gases may come out from the solidifying clads in the form of bubbles. As the gaseous bubbles velocity is higher than the clad solidifying velocity, it results into porosity free clad.

Further the SEM micrograph (Fig.6.3a) of clad cross-section shows the entire clad exhibits dendritic structure. There is no transition of dendritic to cellular structure which attributes the volumetric nature of heating.

6.3.2 Ni-Al+10%SiC Cladding Microstructure Study

Intermetallic matrix composite (IMC) coating has been observed with SiC reinforcement as shown in Fig.6.4 (a). In the developed clad specimen, the SiC particles distributed throughout in the nickel aluminide matrix mostly in melted and partially unmelted form this is verified by EDS as shown in Fig.6.4 (b).

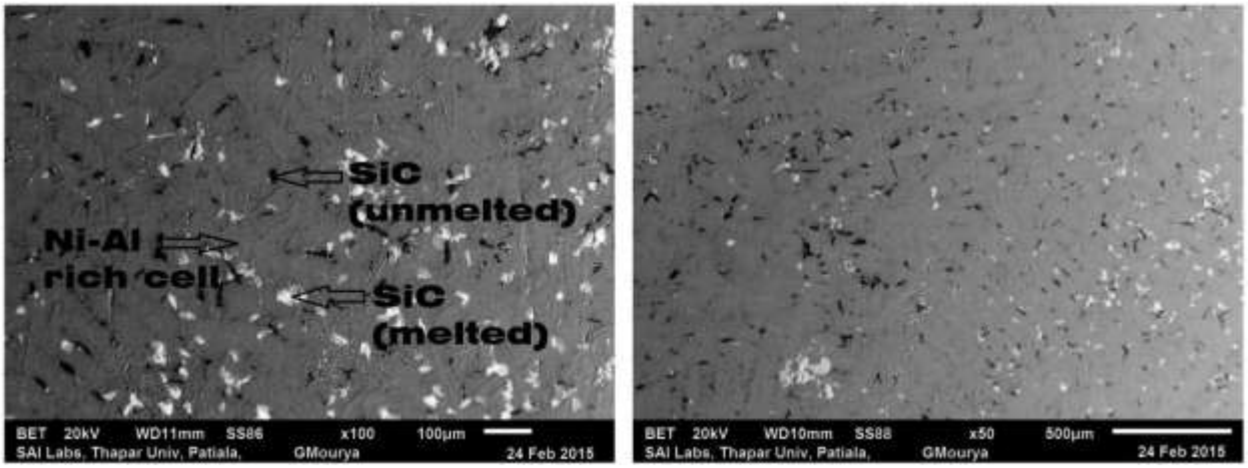


Figure 6.4(a): SEM morphology of Ni-Al+10%SiC clad section (at 100µm and 500 µm) showing Ni-Al rich cell with both melted and unmelted or precipitated silicon carbide.

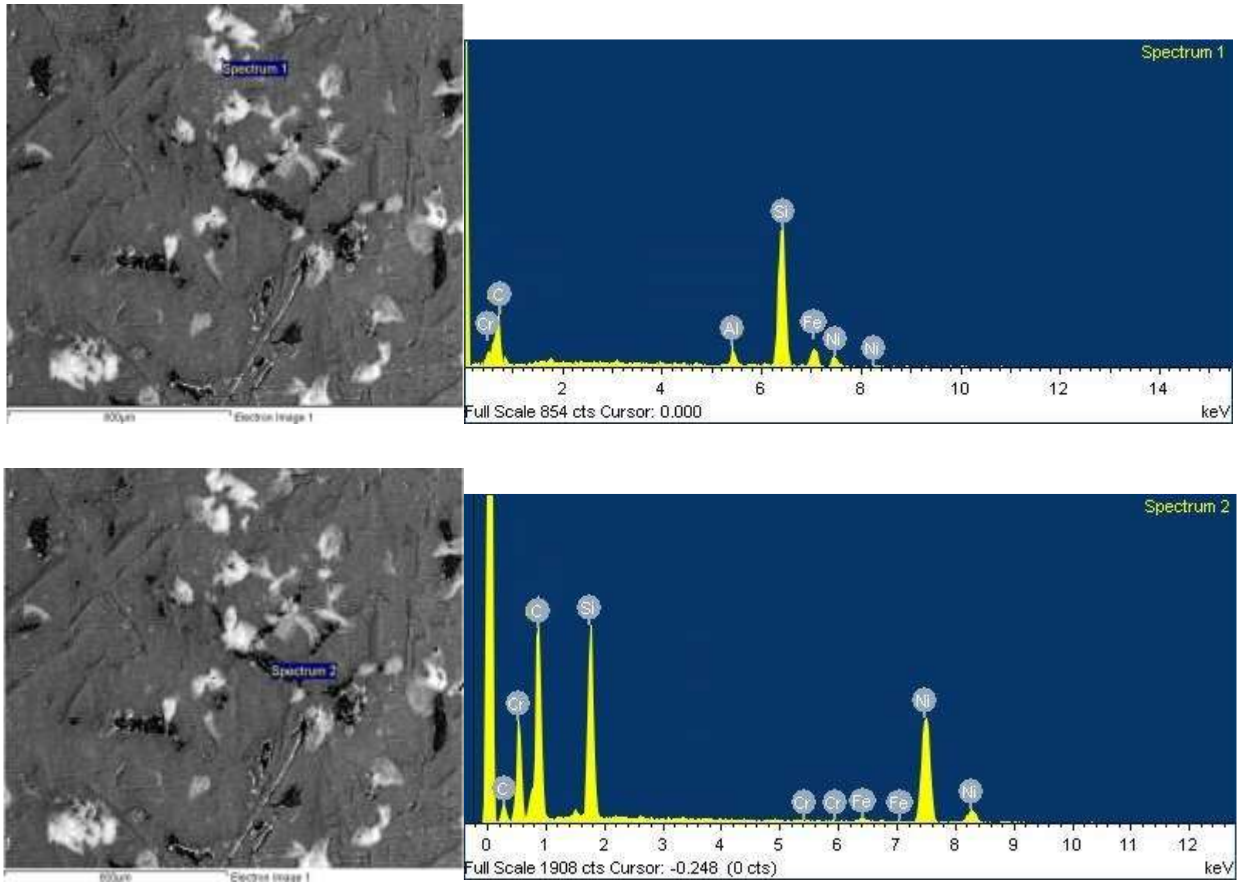


Figure 6.4(b): EDS of Ni-Al+10%SiC clad at three different points

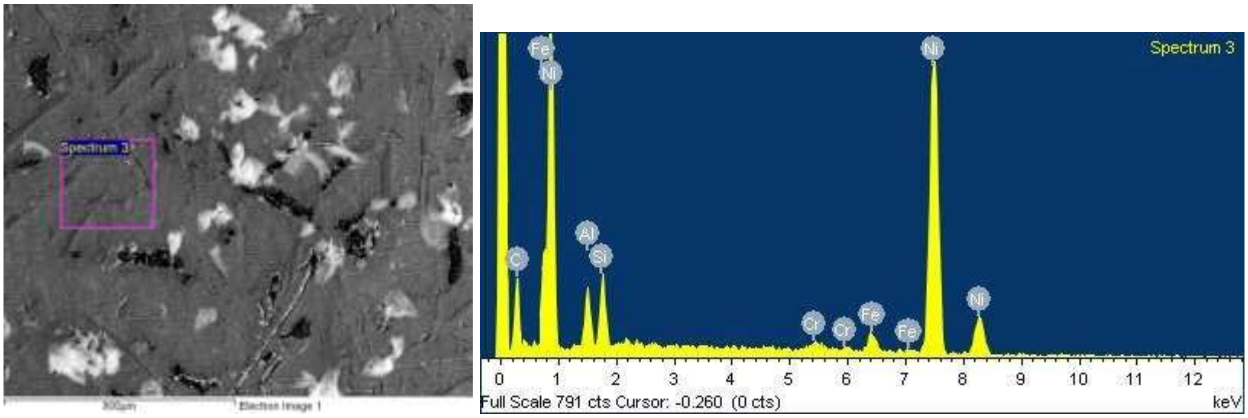


Figure 6.4(b): EDS of Ni-Al+10%SiC clad at three different points

This is due to the addition of SiC (2730°C) particles having high melting point and high hardness and the melting point of Ni-Al matrix (~1500 °C) is less as compared; it leads to many unmelted SiC particles remained in the solidification structure in precipitated form. Performance of coating is definitely got affected due to these few unmelted particles.

The distribution of SiC unmelted particles in matrix result in decrease in wear resistance due to poor bonding with the matrix. During the microwave interaction the partial mutual diffusion of Fe and Cr might have taken place which lead to partial melting of substrate and formation of metallurgical bonding was favored. No crack in IMC has been found. A good uniformity and homogeneity noticed in the SEM images.

6.4 Elemental Study of Cladding

The distribution of different elements was analyzed through energy dispersive X-ray elemental spectroscopy from the interface towards the clad.

6.4.1 Elemental Study of Ni+10%SiC Clad

Elemental study results are illustrated by showing the various elemental distributions at different location from the interface to the clad.

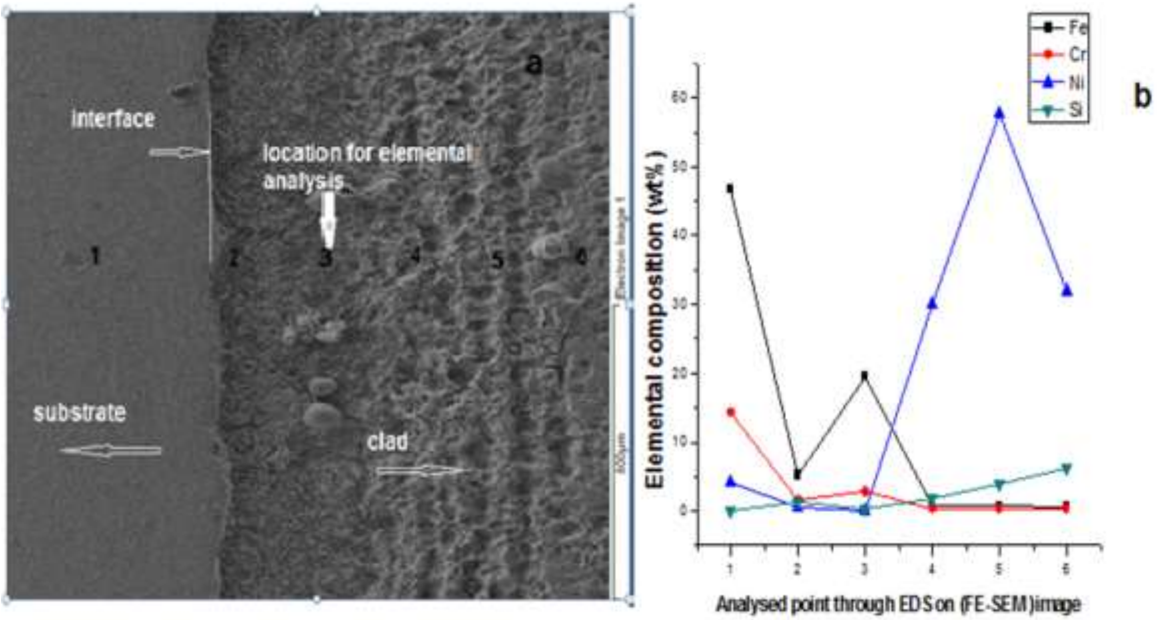


Figure 6.5: Elemental distribution of Ni+10%SiC clad from interface to clad (a) locations for analysis and (b) elemental distribution

As shown in Fig.6.5 there is decrease in percentage of iron content from interface towards clad, while there is slight increase in percentage of chromium contents. As the preplaced Ni+10%SiC powder does not contain any iron content and chromium content is also very less. Thus the presence of these constituents in the clad evidences to the substrate dilution. The percentage of chromium was decreased significantly from interface towards the clad. Formation of intermetallic such as Fe_2Si and Fe_3Ni resulted from the presence of Si, Fe and Ni. Further higher level of chromium is resulted into the formation of chromium carbide in the form of $Cr_{23}C_6$. Fig.6.3 represents the traces of free carbon present. As carbon has more affinity towards chromium results into formation of carbide during solidification.

6.4.2 Elemental Study of Ni-Al+10%SiC Clad

The location of points and at respective point the elemental composition is shown in Fig.6.6. As shown in Fig.6.6(a) there is maximum iron present at substrate. As we move to interface the content of iron decrease due to dilution of clad in substrate. This dilution in substrate increases the nickel content at interface. Elemental composition of nickel is high as shown in Fig.6.6(b) so the clad matrix is nickel rich and this nickel is present mainly in Ni_3Al, NiAl and Ni_3Si phase form.

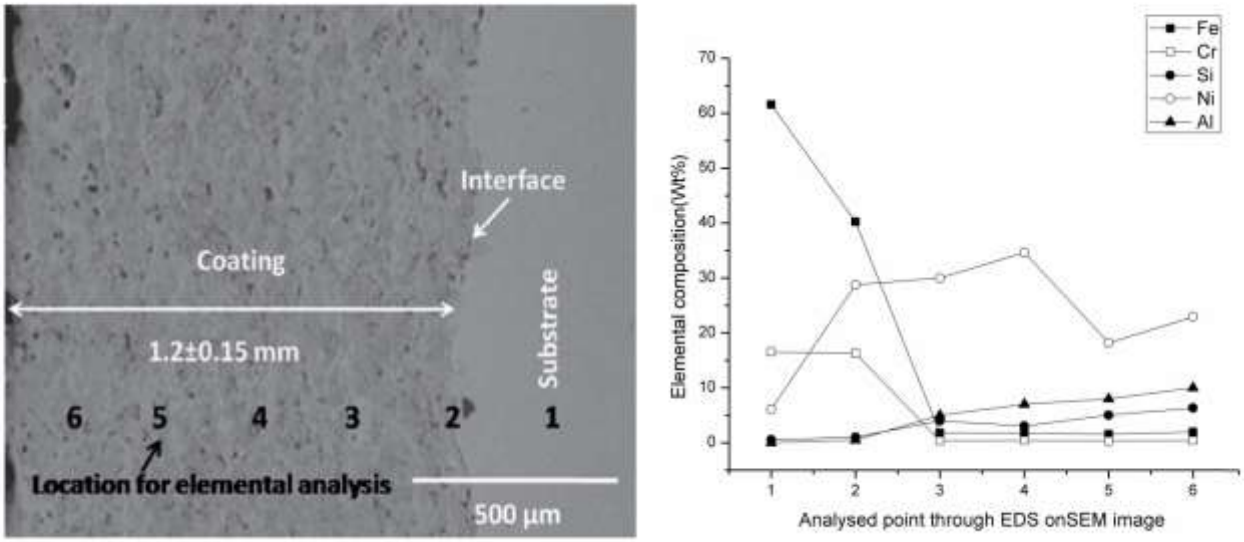


Figure 6.6: Elemental distribution of Ni-Al+10%SiC clad from interface to clad: (a) locations for analysis, (b) elemental distribution

After interference the content of silicon slightly increase as we move from interface to the clad this is due to presence of SiC particles as shown in Fig.6.4. Since the curve of aluminium content has not any significant changes in slope this shows that aluminium is uniformly distributed in clad matrix in different phases like Ni_3Al, NiAl and Fe_3Al.

6.5 Study of Microhardness

Wear performance of the material strongly depends upon hardness of that material. Most frequently wear resistance ability of component increases with increase in hardness of that component although this effect is not straight forward. In the present study Vicker's microhardness of clad layer has been evaluated over the cross-section.

6.5.1 Ni+10%SiC Cladding Microhardness Study

Indentations were taken at 125µm distance apart with an additional indentation at the fusion line or interface and the substarte. The microhardness distribution is shown in Fig.6.7. The average microhardness of the clad section is ~652±30Hv.

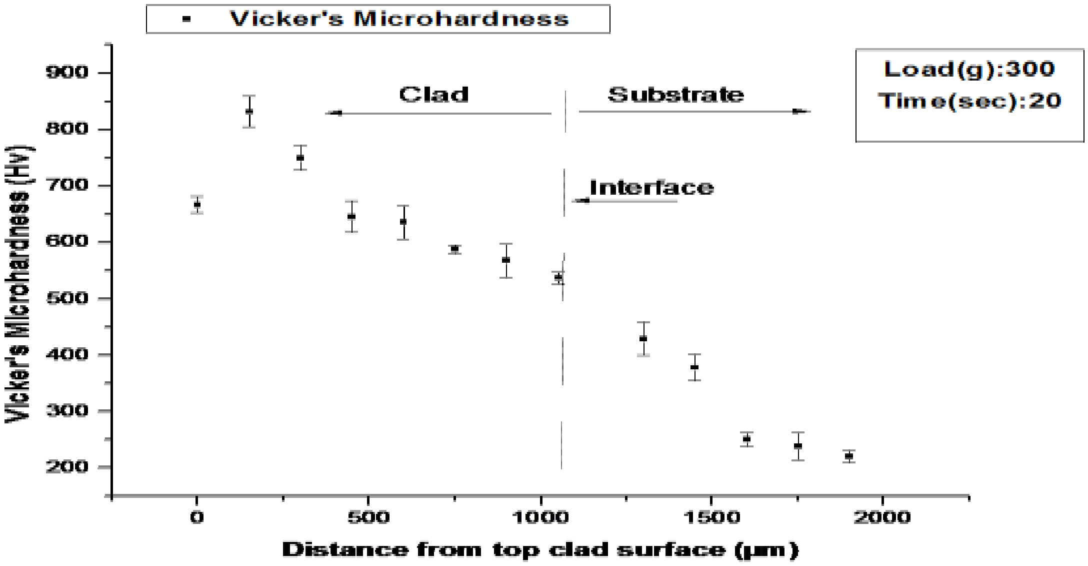


Figure 6.7: Vicker’s microhardness distribution ofNi+10%SiC clad section

The microhardness distribution in the clad section is not observed to be uniform. Microhardness from the top of clad surface to half of clad surface is almost uniform. But at the clad-substrate interface it reduces to 530Hv.The highest hardness (~859Hv) available at the clad top surface, it could be attributed to the presence of hard carbide phase. However due to increase in metallic dissolution at the clad-substrate interface, drop in hardness occurs at the interface. As the indentations being carried out at the hard skeleton structure as well as the tough matrix phase those results into higher standard deviation in the hardness measurement.

6.5.2 Ni-Al+10%SiC Cladding Microhardness Study

Indentations were taken at 150µm distance apart with an additional indentation at the fusion line or interface and the substarte. The microhardness distribution is shown in Fig.6.8. The average

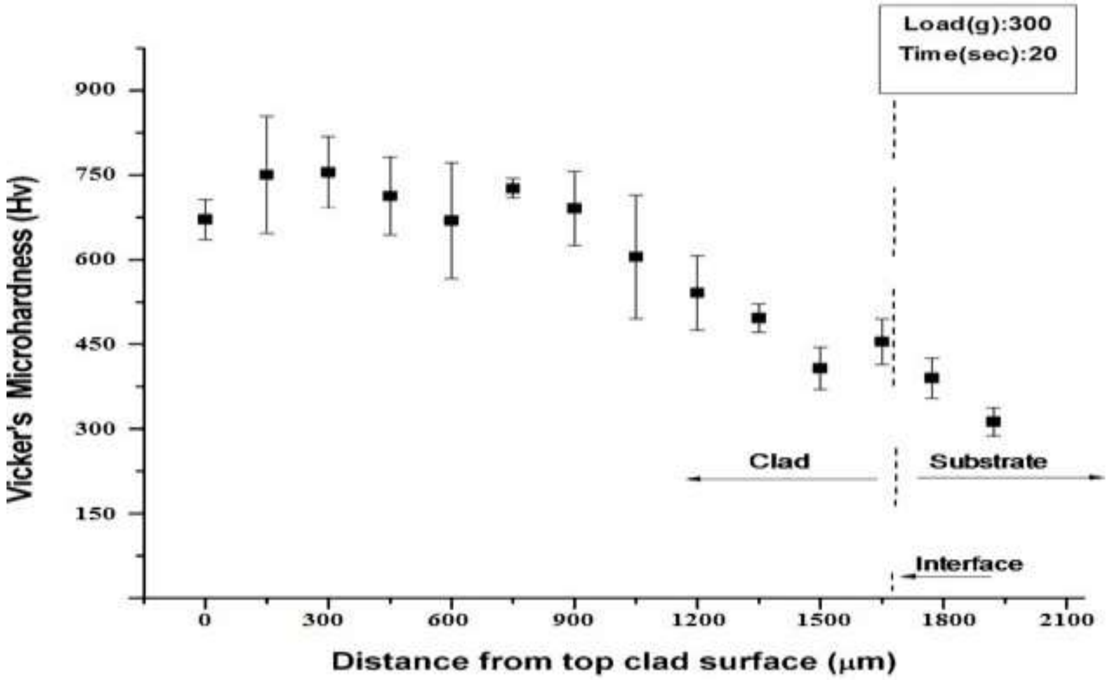


Figure 6.8: Vicker’s microhardness distribution of Ni-Al+10%SiC clad section

microhardness of the clad section is ~615±30Hv. The maximum hardness of clad is at top surface (~830Hv). Moving towards the substrate the microhardness decreases. There is little variation in trend in-between and also standard deviation in microhardness introduced may be due to presence of precipitates of SiC. At the clad-substrate interface microhardness reduces to 450Hv. The microhardness of the substrate also increases near interface due to presence of heat affected zone.

6.6 Wear study

For studying the wear rate of the clad as well as the substrate material the cumulative weight loss characteristics are presented in figures below. With the help of figures a summary of the cumulative weight loss data corresponding to 400m, 800m, 1200m, 1600m and 2000m of sliding distance at different sliding speeds is illustrated. An important parameter in the wear process is the interaction of two mating surfaces having relative sliding speeds.

In general, during initial phase there is more weight loss since the loose particles get easily wear off from the specimen. After some distance the wear loss started decreasing that is since the loose particles are removed and the hard phase of clad material come in contact with the rotating disc. Further due to formation of oxide film wear rate should decreases but there are other factors like ploughing by detached worn hard phase, plastic flow, rubbing etc. form crater and grooves that result in uneven trend of weight loss. These crater and grooves are responsible for crack formation.

6.6.1 Wear study at Load 0.5Kg

The cladding developed on SS-420 substrate is investigated at a load of 0.5Kg and at three different sliding speeds with respect to sliding distance as shown in Fig.6.9 and Fig.6.10.

For Ni+10%SiC at load 0.5Kg, the cumulative weight loss increased linearly with the sliding distance as shown in Fig.6.9(a). At sliding speed 0.5m/s cumulative weight loss suddenly decrease from 1600m to 2000m distances, as compared before. As far as cumulative weight loss is concerned to sliding speed of 1m/s, it increased linearly and at sliding speed of 1.5m/s, the cumulative weight loss increased gradually up to 800m of sliding distance. After 800m of sliding distance, the wear becomes steady and steady weight loss was noticed.

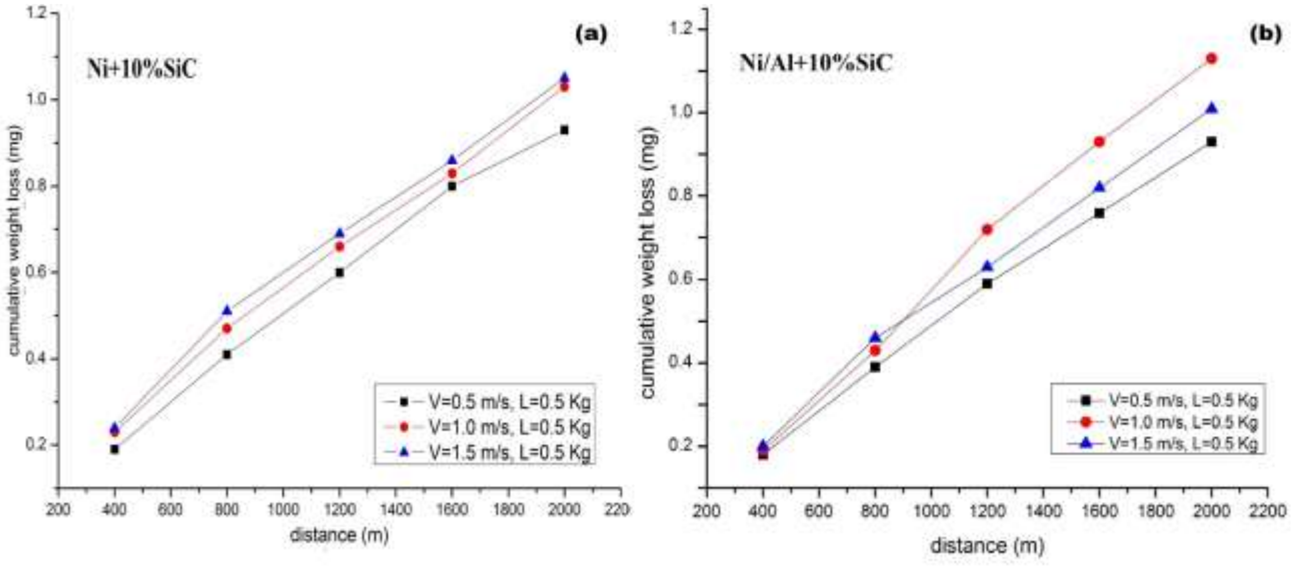


Figure 6.9: Cumulative weight loss at 0.5Kg Load (a) Ni+10%SiC and (b) Ni-Al+10%SiC

For Ni-Al+10%SiC clad as shown in Fig.6.9(b) there is linear variation of cumulative weight loss with the sliding distance. But there is sudden increase in wear at sliding speed 1m/s, which is due to abrasive wear done by separated particles of un-melted SiC particles. The un-melted SiC particles got stick between the clad specimen and rotating disc, leads to large craters on the surface of clad. Theses craters on clad surface are sensitive to wear.

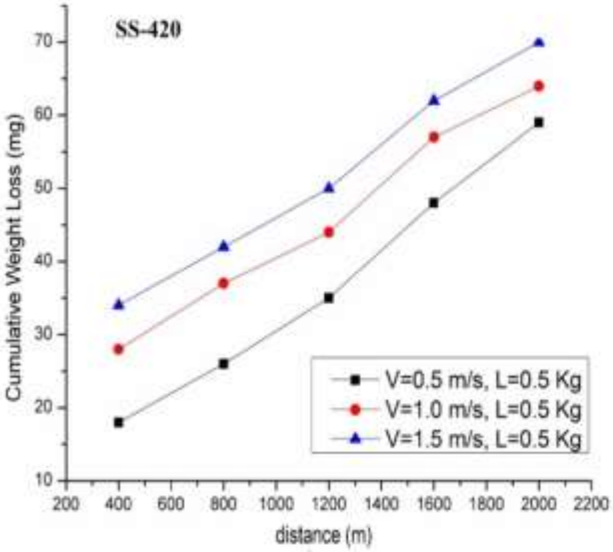


Figure 6.10: Cumulative weight loss of SS-420 at 0.5Kg Load

From Fig.6.10 it is observed that the cumulative weight loss shows linear trend for SS-420. With increase in sliding speed there is increase in weight loss. There is sudden increase in weight loss between 1200m to1600m for all consecutive sliding speeds.

6.6.2 Wear study at Load 1.0 Kg

In case of Ni+10%SiC wear rate slightly decrease at the intermediate region from 800 to 1200m sliding distance and then again increase linearly as shown in Fig.6.11(a) . It might be due to sticking of wear particles between the specimen and the rotating disc, due to that there is no direct contact between the clad and rotating disc.

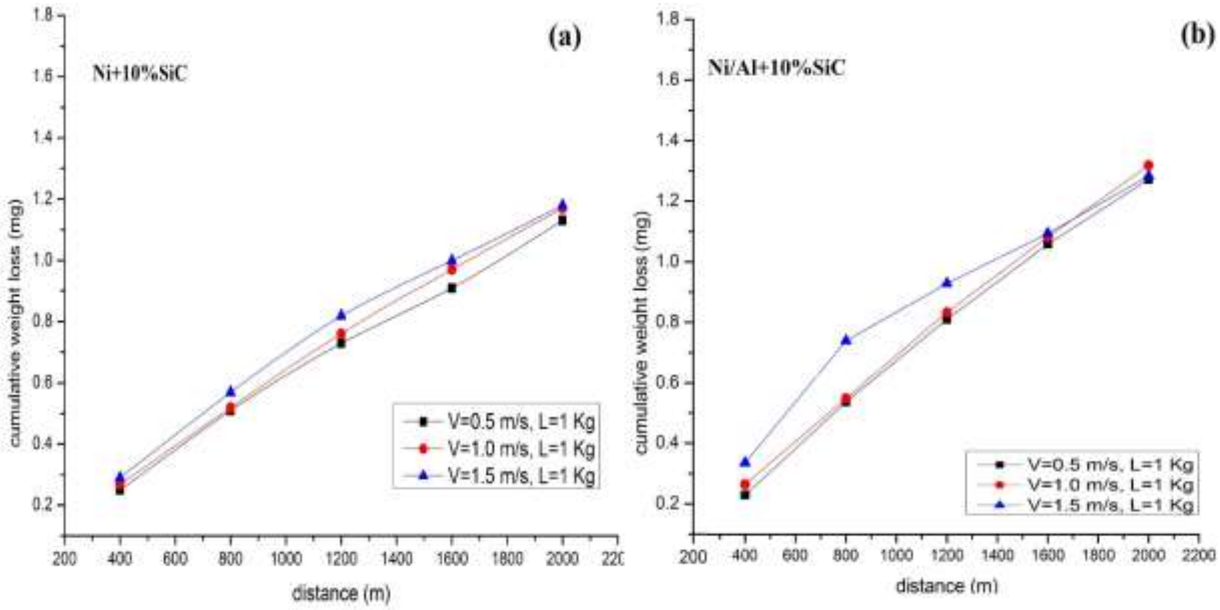


Figure 6.11: Cumulative weight loss at 1.0Kg Load (a) Ni+10%SiC and (b) Ni-Al+10%SiC

For Ni-Al+10%SiC clad as shown in Fig.6.11(b), initially at sliding speed 1.5m/s the cumulative weight loss is drastically increasing and then gradually decreasing. This may be due to removal of unmelted SiC particles that result in abrasive wear of the clad, but with further increase in sliding distance a unstable and hard oxide layer is formed that results in decrease of wear. For sliding speed 0.5m/s and 1m/s there is linearity in graph that is with increasing sliding speed the cumulative weight loss increase continuously with respect to sliding distance.

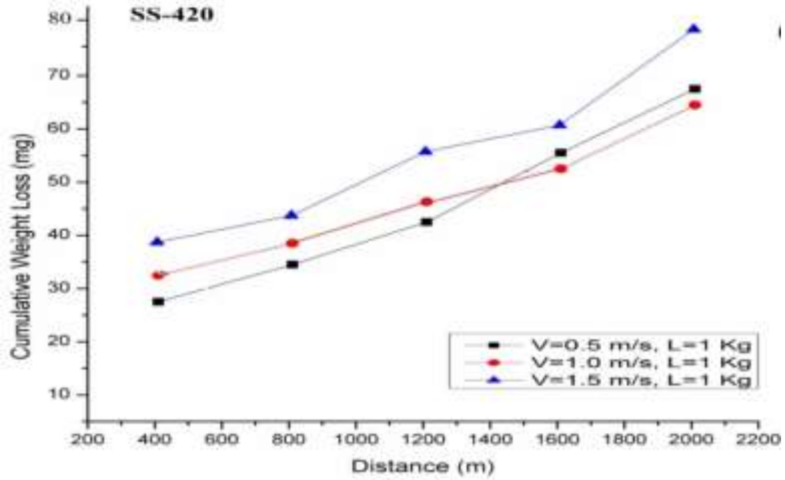


Figure 6.12: Cumulative weight loss of SS-420 at 1.0Kg Load

From Fig.6.12, it is observed that for SS-420 the wear loss increases with the increase in sliding speed till 1200 m. Then there is change in trend of wear loss between 1200m to 1600m. At approximately 1500m the wear loss at 0.5 m/s sliding speed increase than wear loss at 1m/s sliding speed.

6.6.3 Wear study at Load 1.5Kg

For Ni+10%SiC at 1.5Kg load, same trend is seen as in previous observations, the weight loss is increasing progressively with increasing sliding speed as shown in Fig.6.13(a) in case of sliding speed 1.5m/s there is increase in cumulative weight loss at last that may be due to smearing of brittle oxide film. The brittle oxide film cannot be prolonged for longer sliding distance. It is a continuous phenomenon of formation and decay of layer between specimen and rotating disc.

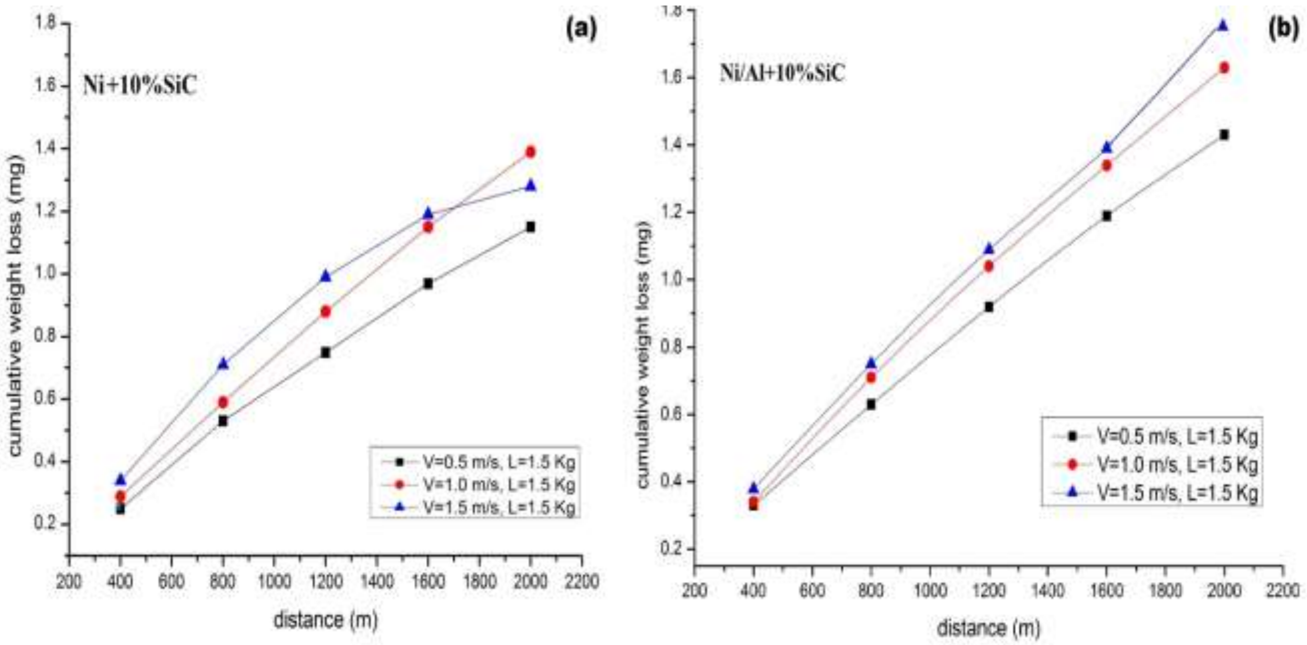


Figure 6.13: Cumulative weight loss at 1.5Kg Load (a) Ni+10%SiC and (b) Ni-Al+10%SiC

For Ni-Al+10%SiC clad there is linear trend for sliding speed 0.5m/s and 1m/s. but there is slight decrease in wear for 1.5m/s between 1200m to 1600m and after 1600m there is significant decrease in wear. Since the formation of oxide film is fast in case of higher sliding speed than that of lower sliding speed due to high temperature. So may be the oxide layer formation is started before 1600m and as thickness got increased that leads to decrease in wear.

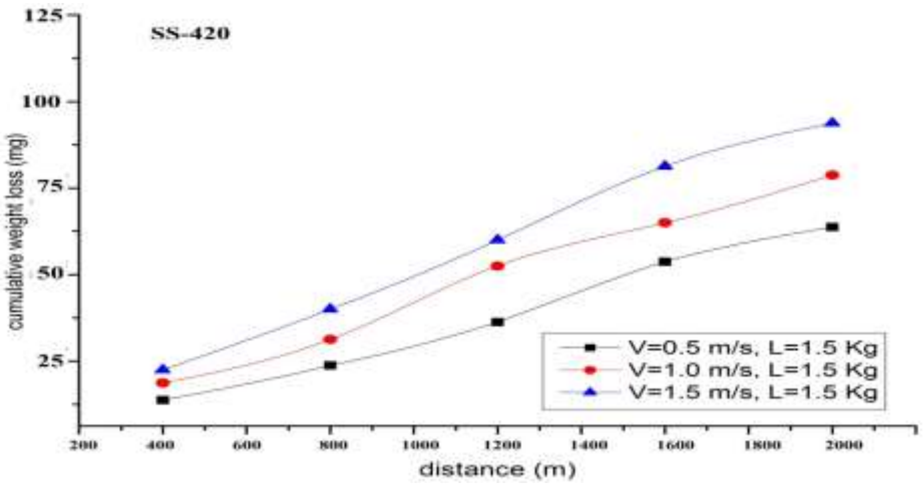


Figure 6.14: Cumulative weight loss of SS-420 at 1.5Kg Load

As shown in Fig.6.14 there is a similar trend followed by different sliding speed. The wear loss is highest in SS-420 at load 1.5kg as compared to load 0.5Kg and 1kg.

Hence the conclusion of above study is that the clad surfaces have much better wear resistance as compared to substrate. Ni+10%SiC clad have less wear as compared to Ni-Al+10%SiC clad, this is due to presence of hard carbide particles in Ni+10%SiC clad. In Ni-Al+10%SiC clad the main reason for wear loss is presence of unmelted SiC particles.

6.7 Factographic Analysis of Substrate and Clads

Images of worn sliding surfaces are shown in Fig.6.15. Due to loss of material there is formation of crater, cracks and grooves can be seen. Here a relative study of claddings with respect to the substrate material is done at sliding speed 1.5m/s, load1.5Kg and sliding distance 2000m. At higher sliding speed the duration for which two mating surfaces come in contact is less as compared to low sliding speed, and therefore the loss of material from clad specimen is severely due to plastic flow. This results in crater formation as shown in Fig.6.15 due to ploughing and then shearing of material. At lower sliding speed the duration for which two mating surfaces come in contact is more as compared to low sliding speed, and therefore loss of material from clad specimen is due to rubbing. As relative speed increases, the factor that plays dominant role is micro-cutting.

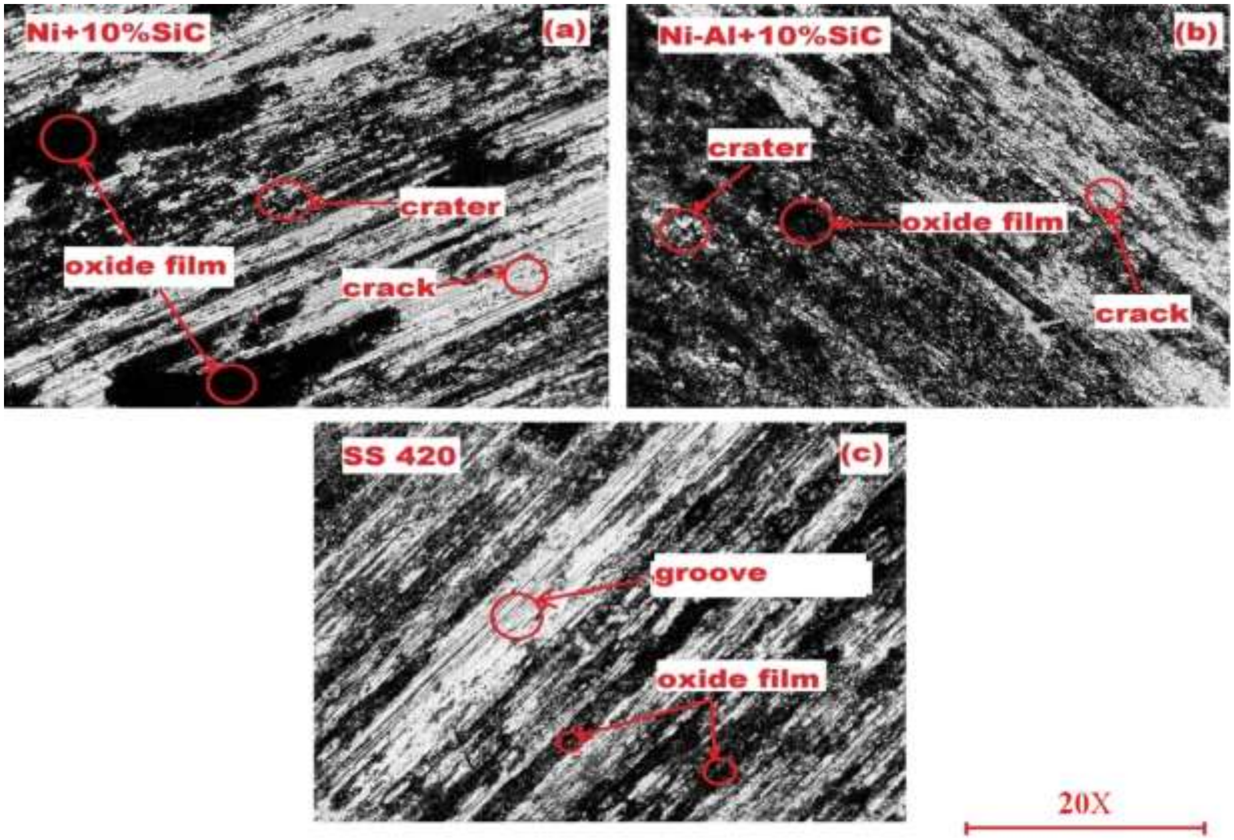


Figure 6.15: Optical microscope images of typical worn surfaces at sliding speed 1.5m/s, load 1.5 Kg and sliding distance 2000m

For Ni+10%SiC clad, it is clearly seen from Fig.6.15(a) that there is formation of oxide film on clad surface with crater formation. This formation of oxide film results into decrease in wear rate since there is no direct contact between the clad and rotating disc surface. Also there are cracks on worn surface. In case of Ni-Al+10%SiC clad also there is formation of oxide film on clad surface as shown in Fig.6.15(b). Presence of un-melted SiC particles between the mating surface results in ploughing of material and this leads large number of craters formation on the surface of clad. These craters on clad surface are very sensitive to wear. SS-420 worn surface is shown in Fig.6.15(c), depression is clearly seen due to loss of material from surface during sliding against rotating disc. Main reason for loss of material is localized welding and ploughing . Long grooves are also seen on the clad surface.

Chapter 7

Conclusion

The cladding of Ni+10%SiC and Ni-Al+10%SiC composite powders was successfully deposited on martensitic steel SS-420 using microwave hybrid heating and characterizations of microstructure of clads were carried out. Major conclusions drawn from the work are:

1) Ni+10%SiC and Ni-Al+10%SiC cladding of thickness ~1.5mm on SS-420 substrates has been effectively developed by microwave hybrid heating.
2) The developed clads shows metallurgical bonding with the martensitic steel substrate by partial mutual diffusion of elements.
3) The microstructure of the developed clad Ni+10%SiC has dendritic morphology and in Ni-Al+10%SiC clad shows composite like structure with reinforcement of SiC both in melted and unmelted form.
4) The porosity in clads is very less and no visible crack formation was observed.
5) Ni+10%SiC clad have high carbides formation and in case of Ni-Al+10%SiC clad, the matrix consists of NiAl and Ni_3Al intermetallic phases with SiC reinforcement which were confirmed by XRD and EDS.
6) The clad layers exhibit higher Vicker’s microhardness than the steel substrate which is due to the formation of carbides and intermetallic hard phases. The microhardness of Ni+10%SiC and Ni-Al+10%SiC clads were 2.86 and 2.76 times higher than that of substrate material.
7) The Ni+10%SiC and Ni-Al+10%SiC clad exhibit 71 times and 52 times higher wear resistance as compared to substrate, this is due to presence of hard carbide particles and intermetallic phases in clad.
8) With increase in load, the rate of wear increases but in case of sliding speed there is not such strong relation is visible for the wear with respect to sliding distance. This is due to different factors like formation of oxide film, which influences and reduces the wear of clads.

Visible Output

- Submitted paper in Journal of Materials: Design and Applications.

  Processing and characterization of composite cladding through microwave heating on martensitic steel.