

**Joining and Characterization of Austenitic  
Stainless Steel (SS-316) and Hastelloy  
through Conventional and Microwave  
Processing Route**

*A Dissertation submitted*  
in partial fulfillment of the requirements  
for the degree of

**Master of Engineering**  
in  
**Production & Industrial Engineering**

by  
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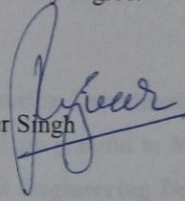
# CERTIFICATE

I hereby declare that the thesis entitled "Joining and Characterization of Austenitic Stainless Steel (SS-316) and Hastelloy through Conventional and Microwave Processing Route" is an authentic record of my work carried out as requirements for the award of the degree of **Master of Engineering in Production & Industrial Engineering** at **Thapar University, Patiala** under the supervision of **Dr. Dheeraj Gupta & Dr. Vivek Jain**, Assistant Professor, Department of Mechanical Engineering, Thapar University, Patiala during July, 2014 to December, 2014. No part of the matter embodied in this report has been submitted to any other university or institute for the award of any degree.

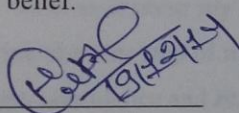
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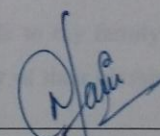
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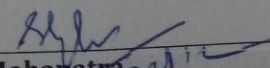


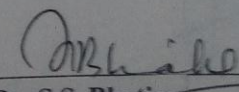
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# Abstract

The application of microwave energy in heating of materials is not new; however, newer applications are emerging in the field of material processing, which allowed microwave processing as novel processing methods. The earlier reported work was based on sintering of many ceramic materials, which are better absorbers of microwaves. The applications were further extended to metallic powders. In recent years, researchers have developed methods to process bulk metallic materials. The developments reported were in the field of joining of bulk metals and claddings on various steels. The present work is based on the comparative evaluation of properties of microwave processed joining and TIG welded joints of SS-316 and Hastelloy steels. The joints were characterized by available techniques such as XRD, EDS and SEM. Mechanical characterizations were carried in the terms of Vickers microhardness and tensile testing of joints. Results revealed that the joining of steels was successfully attempted by using nickel based powder. The characterizations of joints revealed that microwave processed joint showed cellular structure with lower defects. Joints mechanical characterizations showed that microwave processing enhanced the mechanical strength due to better metallurgical bonding and diffusion, with lower processed defects. Ultimate tensile strength was 20.7% more than TIG welded SS-316 joint and 23.25% more than TIG welded Hastelloy steel. Higher hardness was obtained in microwave processed joints due to the presence of carbon which was absorbed from graphite sheet, which led to the formation of carbides in the microstructures.

Fractographic analysis showed mixed mode fracture for microwave processed joint and ductile behavior for TIG joint. The brittle behavior was due to the presence of unmelted nickel particles in the joint region.

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# Abbreviation

ASTM	American Society for Testing and Materials.
EDS	Energy Dispersive X-Ray Spectroscopy.
EM	Electromagnetic Spectrum.
EPMA	Electron Probe Micro Analyzer.
HAZ	Heat Affected Zone.
MHH	Microwave Hybrid Heating.
MW	Microwave.
SEM	Scanning Electron Microscopy.
TIG	Tungsten Inert Gas.
UTS	Ultimate tensile strength.

# Chapter 1

## Introduction

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### 1.1 Introduction

In the environment of growing technologies and cutthroat engineering developments, the industries are looking forward for new and improved processing techniques, which can be used for a wide range of materials including ceramics, metallic, nonmetallic and composites. The thrust for lowering the environmental degradations and higher energy efficient processing methods lead researchers to focus on processing methods which consumes less power and processing time and yet produce high quality products. The unique characteristics of microwaves led the researchers to explore the potential of microwave heating in various areas. Recently, lot of research is going in the field of joining of material using microwaves and applications are still increasing. This chapter will cover the fundamentals of microwave heating of materials and applications in various fields.

#### 1.1.1 Introduction to Microwaves

The microwaves are the part of the electromagnetic (EM) spectrum in which electric and magnetic waves propagate perpendicular to each other with frequency range of 300 MHz to 300 GHz [1]. The wavelength of these waves varies from 30 cm to 1 mm in air as shown in Fig.1.1, which represents the EM spectrum. This range of frequency allows microwaves to be used in a variety of applications including communication systems, food processing, medical purposes, industrial heating, material processing etc [2]. The common frequency on which domestic microwaves in India operates is 2.45 GHz, which is primarily used for heating foodstuffs. The furnaces have been developed for material processing purposes which are used in many industrial applications and they work on higher frequencies ranging from 915 MHz to 18 GHz [3-4].

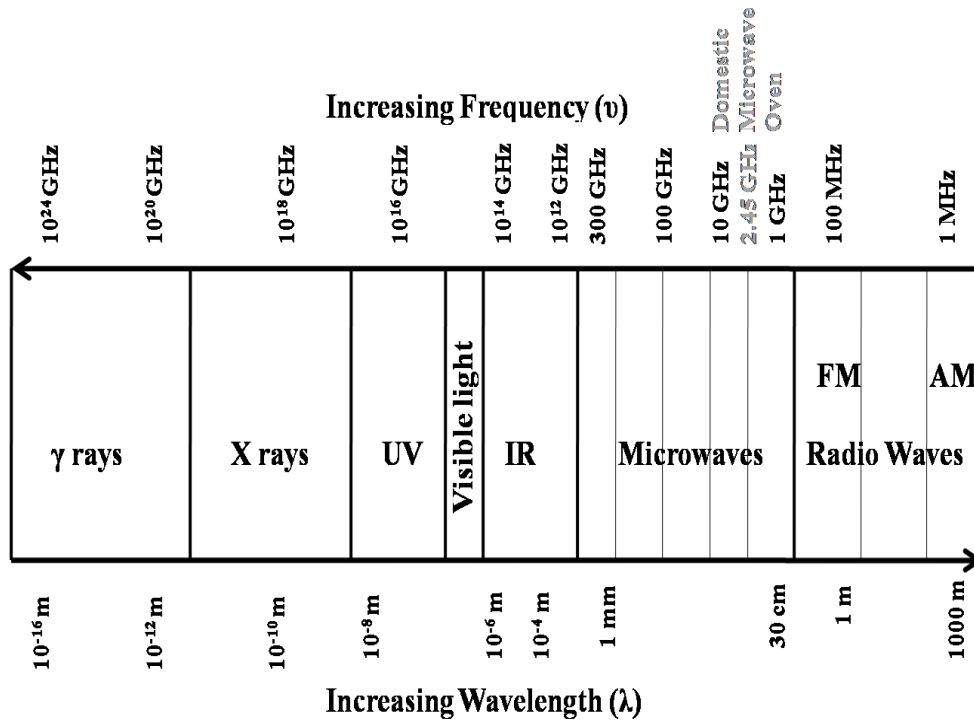


Figure 1.1: Electromagnetic spectrum showing frequencies and wavelengths of microwave band.

The primary applications of microwaves were involved in communication systems including RADAR, satellite communications and television broadcasting. The heating effects of microwaves were accidentally discovered by Spencer and in the year 1945 the first patent [5] of microwave oven for heating purposes was filled. The microwave oven over the time become one of the popular house hold item for carrying out heating of foodstuffs due to the features of higher heating rate, low processing time and lower energy consumptions. These favorable and unique characteristics of microwaves attracted many researchers towards the possibilities of processing different materials using microwave heating phenomenon.

The applications of microwaves for heating of materials were further explored by researchers [6-8] in the field of material processing in order to gain the advantages of higher heating rates coupled with lower processing time. These researches led the applications of microwaves in chemical reactions, vulcanization of rubber, processing of ceramics and metallic materials, steel making, alternative sources of energy recovery [9] etc. The major characteristics of microwave materials processing are represented by Fig.1.2, which shows that microwaves can be used for processing of a variety of materials with higher heating rates and environmentally friendly characteristics.

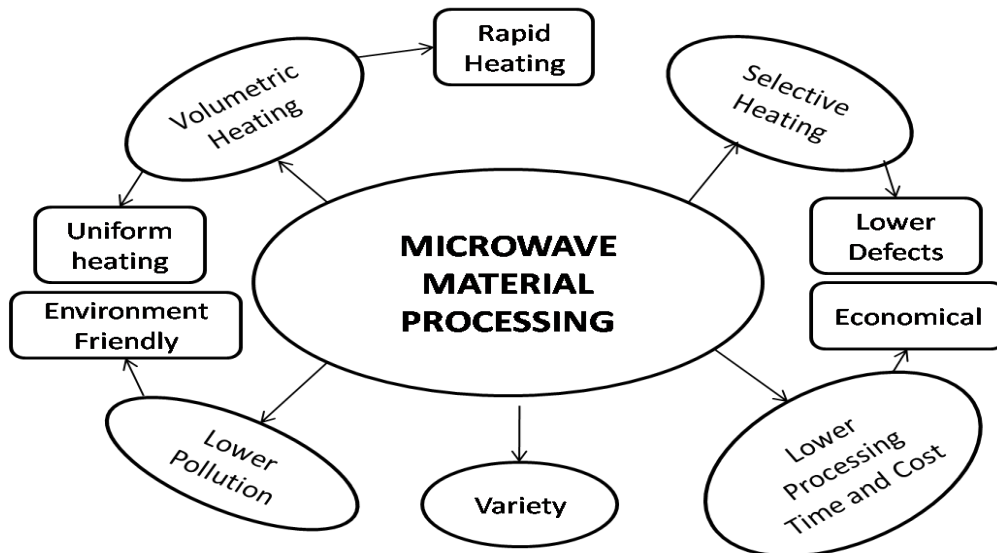


Figure 1.2: Unique characteristics of microwave materials processing.

The direct absorption of microwaves to the atomic level of microwave coupled materials leads to volumetric heating of material from within the materials, this leads to rapid heating rates with less thermal gradient inside processed materials. The rapid heating rates arises due to volumetric heating characteristic of microwaves, which leads to lower down the processing time and owing to which it consumes less energy in comparison to conventional heating systems. The mode of selective heating of material by exposing microwaves to the focused region, allows localized heating which results in lower heat affected zone (HAZ) and lower defects during processing. The work of various authors [10-12] reported huge savings in terms of processing time and power requirements during microwave processing. The microwave processing of many ceramics was achieved successfully, with enhanced diffusions, densifications and improved properties and positive results were reported by many researchers [13-15].

### 1.1.2 Theory of Microwave Heating and Microwave Hybrid Heating

The heating of materials takes place by direct absorption of microwaves throughout the volume of material. However, the effective and efficient heating of materials via microwave radiations depends upon the physical properties of the materials and these properties plays a dominant role in deciding the processability of materials by microwaves. Different materials can interact in different ways with microwaves, which is shown in Fig. 1.3 and classifies the main three groups of materials [16].

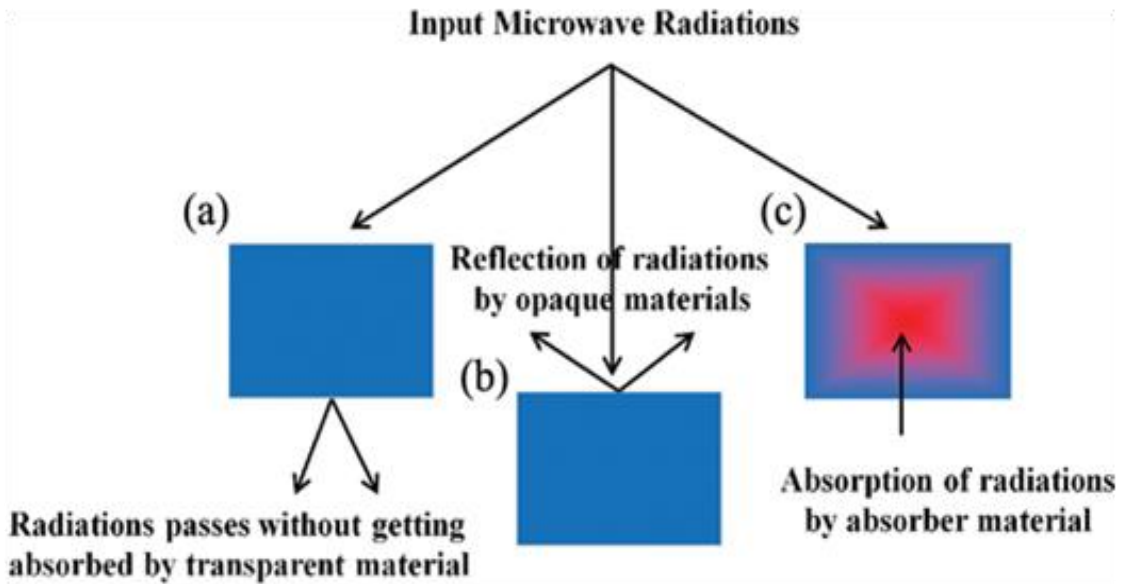


Figure 1.3: Interaction of microwaves with (a) transparent materials, (b) conducting opaque and (c) dielectric materials (Singh et al.)

The transparent materials such as glass do not absorb microwaves and readily allows them to pass through without any loss and hence no heating takes place. On the other hand bulk metallic conductor materials do not allow microwaves to pass and neither absorbs but causes reflection from surface. This leads to the plasma formation and causes surfacial heating of the body. However, third class of materials, mainly known as microwave coupled materials (dielectric material) readily absorbs the microwaves and converts these radiations into heat. This generation of heat is a complex phenomenon which occurs mainly by the dipolar loss or reorientation mechanism. When material is subjected to microwaves then electric and magnetic fields alternates 2.45 billion times in a second and this causes rotation of dipoles as shown in Fig.1.4.

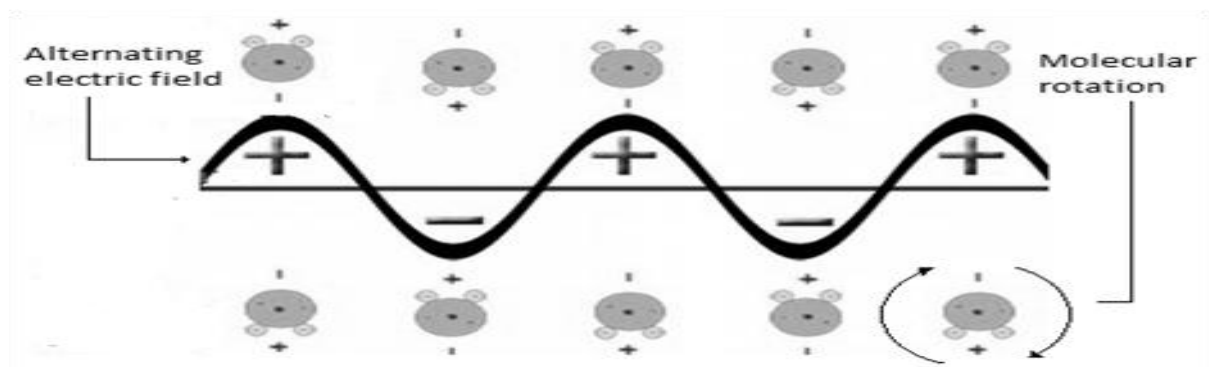


Figure 1.4: Mechanism of dipole rotation subjected to microwaves

The presence of cohesive forces between the dipoles hinders the rapid reversals and causes frictional heating. The internal resistance of material also causes resistance heating on application of alternating electric fields. This process takes place instantaneously within the whole body and leads to the volumetric heating of whole mass subjected to microwaves. The main highlight of microwave heating is that heat is produced within the material and has inverted profile i.e. from inward to outward surface; whereas in conventional heating surface is heated first and then heat is transferred inwards. The comparison of heating mechanism is shown in Fig.1.5.

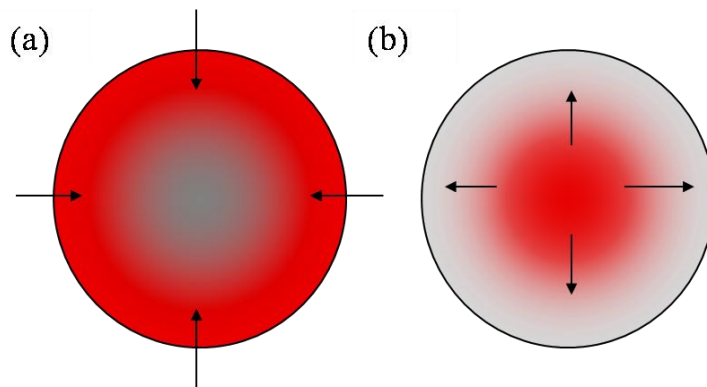


Figure 1.5: Heating mechanism for (a) conventional heating in which heat is transferred from outer to inner surface and (b) microwave heating profile from inner to outer surface (Singh et al.)

The main properties of materials responsible for absorption of microwaves are complex relative permittivity and loss tangent represented by Eq.(1.1)-(1.2) [16].

$$\epsilon = \epsilon_0(\epsilon' - j\epsilon'') = \epsilon_0\epsilon'(1 - j\tan\delta) \quad (1.1)$$

$$\tan\delta = \frac{\epsilon''}{\epsilon'} \quad (1.2)$$

where:

$\epsilon$ = electric constant in medium

$\epsilon_0$ = electrical permittivity in space

$\epsilon'$ = electrical permittivity in medium

$\epsilon''$ = dielectric loss factor

$j$ = electrical polarisability

$\delta$  = dielectric loss tangent

The dielectric loss factor decides the ability of a material to convert microwave energy into heat energy and dielectric constant measures the polarisability of material. The increase in temperature due to absorption of microwaves is governed by Eq.(1.3) [16].

$$\frac{\Delta T}{\Delta t} = \frac{2\pi f \epsilon_0 \epsilon'' |E|^2}{\rho C_p} \quad (1.3)$$

The heating rates in microwave processing are accelerated by a combination of heat generated by various processes such as polarization process, conductive and radiative heat losses from the materials. The dielectric interactions of materials with microwaves are further described by two parameters: Power dissipation (P) and depth of penetration (D) of microwaves. The uniformity of heating profile within the material depends upon these factors.

The power dissipation is expressed by Eq.(1.4) [16].

$$P = \frac{1}{2} \omega \cdot \epsilon_0 \cdot \epsilon'' \cdot E^2 \cdot V \cdot e^{-2\alpha z} \quad (1.4)$$

where:

$E$ = electric field through the surface

$V$ = volume

$\omega$ =  $2 \cdot \pi$  \* frequency

$z$ = distance into the specimen

$\alpha$ = attenuation constant

The uniformity of heating within the material depends upon the depth of penetration at which the incident power is reduced to half of its initial value. The depth of penetration is expressed by Eq.1.5[16].

$$D = \frac{3\pi_0}{8.686\pi \tan \delta (\epsilon' / \epsilon_0)^{1/2}} \quad (1.5)$$

The relationship of the penetration depth (D) in terms of frequency is expressed by Eq.1.6 [16].

$$D = \frac{C}{2\pi f \sqrt{2\varepsilon'} (\sqrt{1 + \tan^2 \delta} - 1)^{1/2}} \quad (1.6)$$

The higher values of  $\tan\delta$  and electrical permittivity reduce the depth of penetration at a particular microwave radiation wavelength. Higher frequencies and larger values of dielectric properties allow surfacial heating while lower frequencies and moderate dielectric properties will allow uniform volumetric heating. The penetration depth of microwaves for metallic powders at a given frequency depends upon the electrical and magnetic properties of materials. Bulk metallic materials reflect microwaves of 2.45 GHz at room temperature due to smaller values of skin depth, which results in reflection of radiations. The skin depth of materials in relation to microwave processing is defined as the depth into the materials from the surface at which the value of incident microwave power drops to  $1/e$  (36.8%) times the surface value [17]. It is an important parameter that gives an understanding of the upper thickness limit of materials to be processed through microwave radiation in an efficient manner. However, it is possible to increase the skin depth ( $\delta$ ) of particular material at a particular frequency by changing the temperature dependent parameters i.e. resistivity and magnetic permeability as represented by Eq.1.7 [16].

$$\delta = \sqrt{\frac{\rho}{\pi f \mu_r \mu_0}} \quad (1.7)$$

where,

$\delta$ = skin depth ( $\mu\text{m}$ )

$\rho$ = resistivity ( $\mu\Omega\text{-cm}$ )

$f$ = frequency of microwaves (GHz)

$\mu$ = magnetic permeability (H/m)

$\mu_0$ = absolute permeability (H/m)

$\mu_r$ = relative permeability

The values of dielectric properties play a significant role in power absorption and heating phenomenon of material through microwaves.

To utilize the applications of microwave for processing of non-coupled materials, the research was carried on microwave hybrid heating systems (MHH), which utilizes the concept of both the conventional heating and microwave heating principles. The conventional heating process of materials allows surfacial heating first and then heat transfer takes place within the material with reduced temperature gradients. This can lead to the poor microstructure of the surfaces and it may lead to the surfacial overheating or burning. In contrary to conventional heating, microwave heating mode can lead to the poor microstructure of core, which can cause thermal runaways, cracking and burning of core due to heating from inside the material. To remove the difference in temperature gradients of surface and core, a new approach was used by researchers called two directional heating or MHH, such that heating of materials takes place from the outside as well as from the inside of materials. The different heating phenomenon are shown in Fig.1.6, which shows the approximate flattening of temperature profile by using MHH within the specimen. The MHH produces uniform heating throughout the materials with reduced temperature gradients and rapid heating. These characteristics are absent in conventional or microwave heating processes.

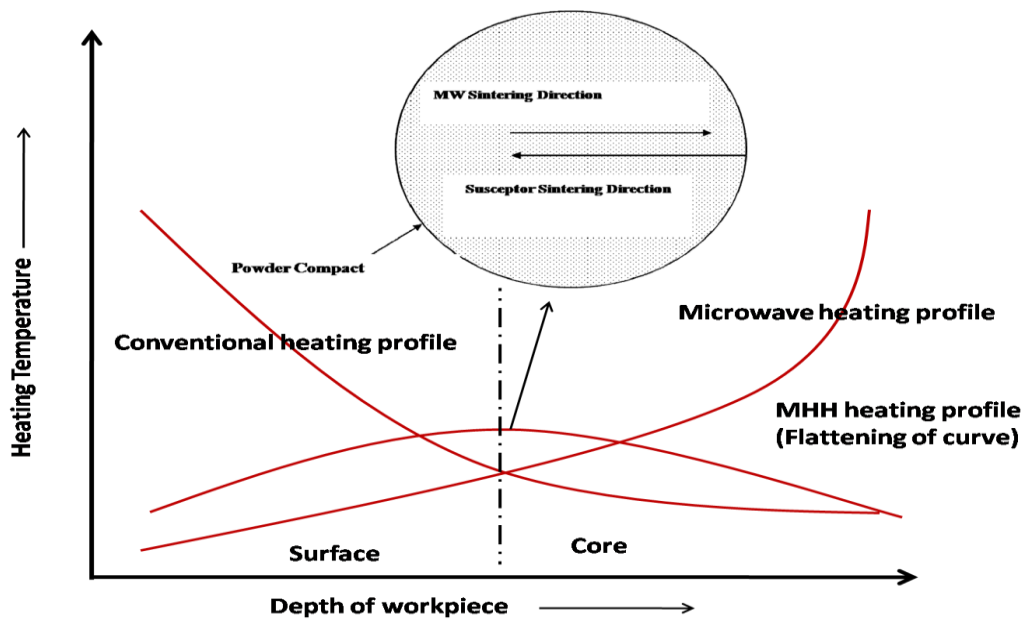


Figure 1.6: Heating profile for conventional, microwave and microwave hybrid heating of materials

### 1.1.3 Historical Developments of Microwave Material Processing

The historical developments in the field of microwaves are shown in Fig. 1.7, which shows that the earlier developments were in the field of low temperature applications.

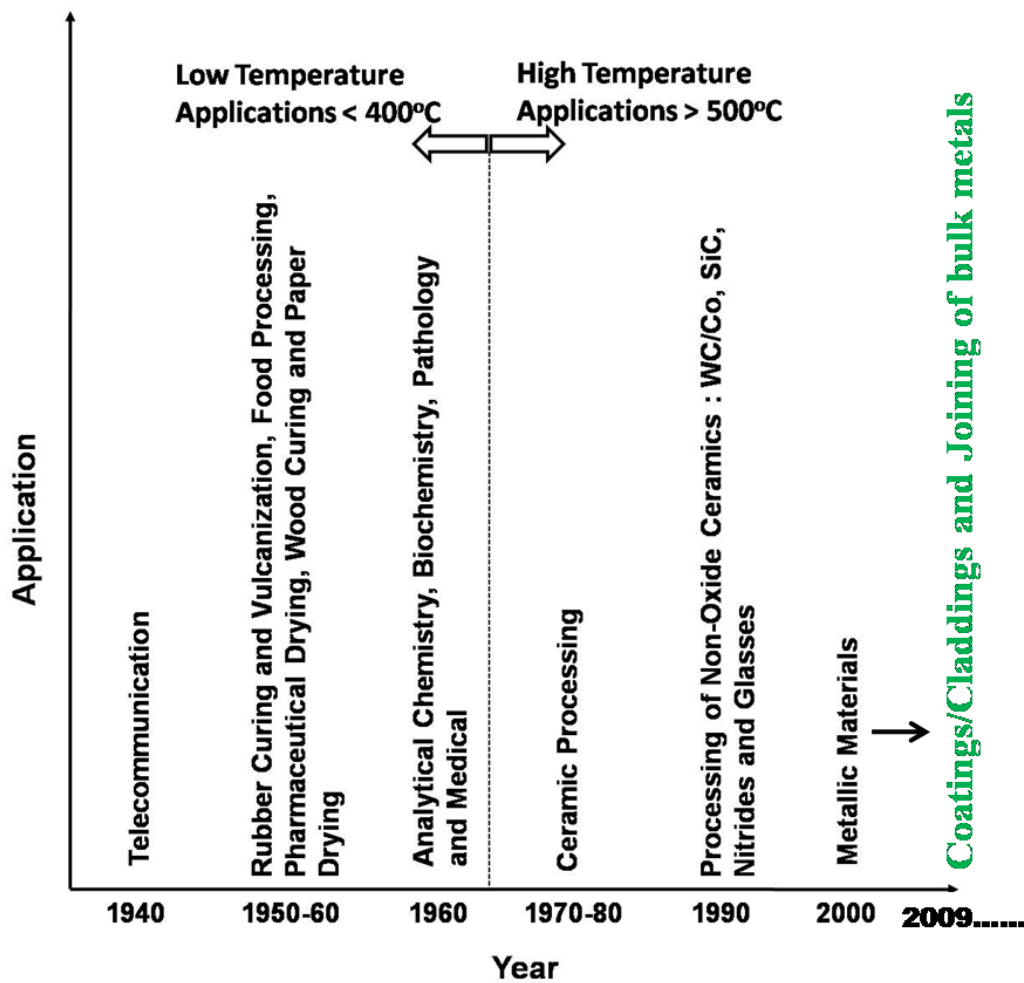


Figure 1.7: Developments in the field of microwave materials processing in chronological order.

These developments were further explored for higher temperature applications, mainly the processing of ceramic materials. In year 1999, the first literature on successful sintering of metallic materials was reported by [Roy et al] [18] and research showed that metallic materials in the form of fine powders can couple with microwaves. This outstanding work led researchers to focus on the processing of metallic powders using microwaves and afterwards successful sintering works on variety of metallic materials have been reported. However, the challenge of processing of bulk metallic materials using microwaves was still left, which was successfully attempted by [Srinath et al., in 2009] [19] in the form of joining of bulk metallic materials by using a domestic microwave oven. This work led the applications of microwaves

for higher temperature applications. Further, [Gupta and Sharma,] [20] in the year 2010 extended the research by using domestic microwave oven for producing claddings of various materials on metallic substrates. These developments further opened broad area for research in microwave processing of materials which requires high temperature up to the order of 1500° Celsius through microwave hybrid heating techniques. The developments in the field of processing of bulk metallic materials using microwave radiations are shown in Fig.1.8. In recent years the applications of attaining of high temperature by microwaves were explored and lot of work was carried out in the field of material joining, claddings and coatings using microwave hybrid heating.

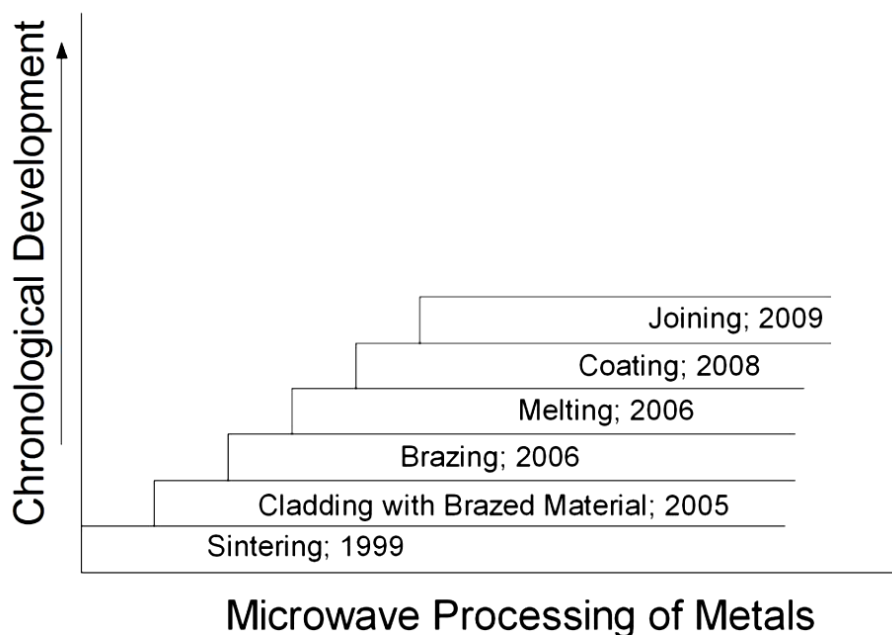


Figure 1.8: Chronological developments in microwave processing of metallic materials.

#### 1.1.4 Advantages of Microwave Processing of Materials

There are certain distinct advantages of using microwaves in heating processes. The characteristics of lower energy consumptions coupled with higher heating rates and lower processing times, led microwave heating as one of the challenging process in material processing. The main advantages of microwave material processing involves better microstructures, lower defect formation, higher efficiency with reduced energy consumptions and lower cost of heating in comparison to conventional heating. The advantages of microwave processing are as follows:

### **Improved Mechanical Properties by Microwave Processing**

The typical requirements for the conventional sintering process include higher heating rates, high temperature and maintenance of this temperature for certain period of time. But to maintain the uniform temperature distributions, the specimens are usually kept for higher soaking periods which lead to the excessive grain growths and defects such as porosity and cracks. These problems can be removed through microwave processing which provides relatively homogeneous microstructures with finer grain growth and enhanced properties in addition to lower processing time. These obtained favorable characteristics are due to the higher heating rates, uniform heating of materials (volumetric heating), and reduced time for achieving higher temperatures. The comparison of microstructures is shown by [Upadhyaya et al.,] [21] in Fig. 1.9.

### **Improved Densification Parameter**

The density of powder compaction decides the properties of the finished product. Higher the density of processed material, lower will be the defects mainly in terms of porosity. Due to uniform heating and volumetric heating of product, the densities obtained are higher in case of microwave sintered or processed parts. The compacts sinterability takes into account the effect of initially pressed density values, which can be related in terms of normalized, dimensionless densification parameter, or factor and is represented by Eq.(1.8) [22].

$$\text{Densification Parameter} = \frac{\text{Sintered density} - \text{Green density}}{\text{Theoretical density} - \text{Green density}} \quad (1.8)$$

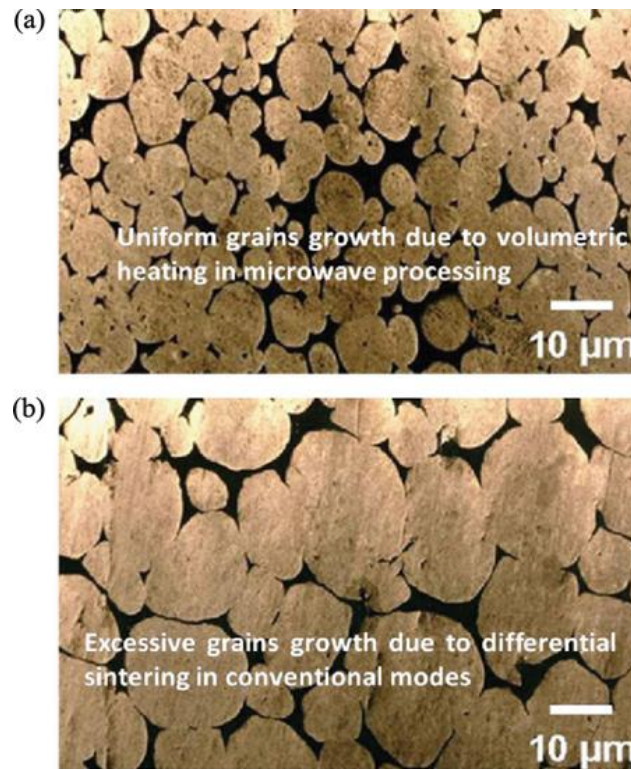


Figure 1.9: Effect of (a) microwave processing and (b) conventional processing, on microstructure of processed material

### Lower Power Consumptions

The microwave heating of materials involves direct absorption of radiations by materials, then converts it into heat energy, and leads to volumetric heating of materials. The literature has reported that microwave heating of materials consumes 10–100 times less energy and 10–200times lesser processing times [23] compared to conventional methods of material processing. The direct transfer of energy eliminates lots of losses such as heating of furnaces, their walls, and heat transfer media. These provide higher rates of heat transfer in comparison to conventional methods and higher temperatures can be attained in shorter times. This reduces the overall consumption of power required for material processing as shown in Fig.1.10 by [Upadhayaya et al.,].

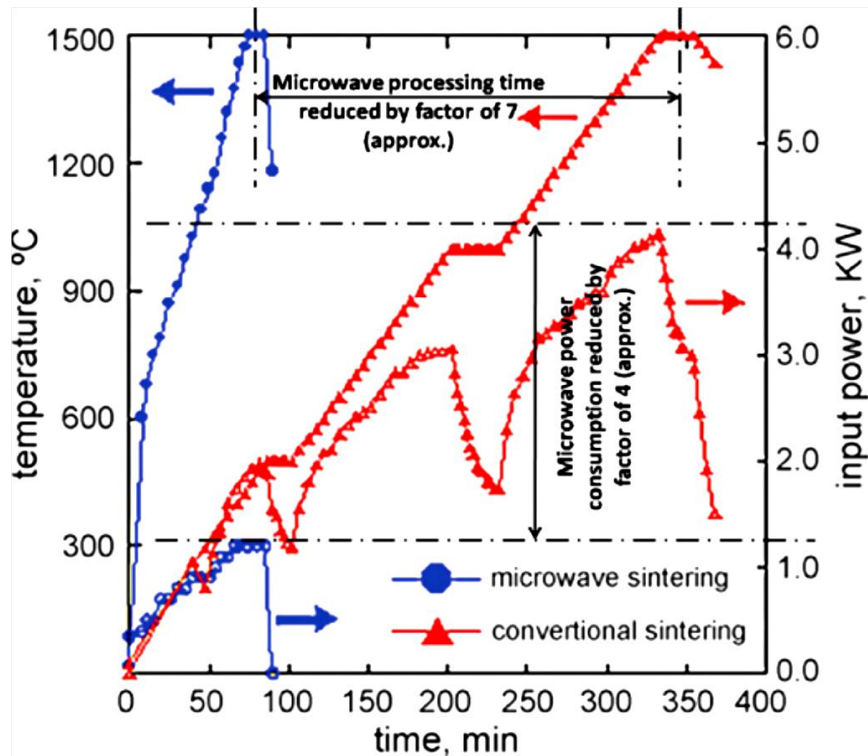


Figure 1.10: Power and processing time comparison during microwave and conventional sintering process

Further a compact system, as small as 20% of the size of conventional systems is required for microwave processing, because of the high applicator energy densities and direct energy absorption by many materials. Microwave energy provides clean transfer of energy to the product being heated.

All these advantages led microwave material processing as a novel technique in microwave processing and enhanced research in this new area.

### 1.1.5 Joining of Materials

The joining of materials allows two or more parts to be assembled together to form big and complex shapes. The joining is mainly considered of two types i.e. permanent joining and temporary joining. The permanent joining of materials can be carried in many ways and is a prime requirement in most of the manufacturing and assembling industries worldwide. Conventionally available techniques of permanent diffusion for joining of materials which includes welding, soldering and brazing; however, they have their own advantages and limitations. The limitations of conventional processes such as arc welding, TIG, MIG and

SAW include higher processing times, higher energy requirements, costly setups, limitations on material to be joined and higher defects produced in the joint region. Further, difficulties in joint processing, environmental hazards and operator safety issues are of primary concern.

Gas tungsten arc welding (GTAW), also known as tungsten inert gas (TIG) welding, is a variant of arc welding process that utilizes a non-consumable tungstenelectrode to carry out the welding. The weld area is protected from atmospheric contamination by an inertshielding gas (argon or helium), and a filler metal is used. It uses a constant-currentwelding power supply to produces electrical energy, which is conducted across the arc through a column of highly ionized gas and metal vapors known as plasma. TIG welding is mostly used to weld thin sections of stainless steel and non-ferrous metals such as magnesium, aluminum and copper alloys. The process allows the operator to have greater control over the weld than competing processes such as shielded metal arc welding and gas metal arc welding. This allows stronger, better and high quality welds. However, TIG welding is comparatively more complex and difficult to master and control process; furthermore, it is significantly slower than most other welding techniques.

Recently microwaves are also employed in joining of materials, mainly steels and better properties are reported in the literature. The detailed review of literature on microwave joining of materials through microwave energy will be provided in the next chapter.

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# Chapter 2

## Literature Survey

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### 2.1 Literature Review

This chapter focuses on the review of the latest developments that have taken place in the field of microwave materials processing, mainly in the domain of joining and processing of steels. A lot of research is going on in the field of microwave processing of metals at high temperature and is reported in this section.

[Gupta and Sharma, 2014] [25] have diversified the domain of microwave materials processing and developed a novel process for surface engineering and it is known as microwave cladding. The authors claimed that the microwave cladding of tungsten based powder on an austenitic stainless steel substrate was developed within a duration of 120 seconds and reported that crack free interfaces were obtained by partial dilution of stainless steel substrate into clad and complete melting and fusion of tungsten carbide based powder. The authors reported that the presence of hard metallic carbides, which were formed during microwave processing, were responsible for higher microhardness of clad obtained. Mean clad hardness of  $945\pm 66$  Hv was reported. It was concluded by the authors that the mechanism of cladding using microwaves has the potential to grow as a novel surface engineering technique.

[Bansal et al., 2014, 2013] [26-27] utilized the microwave radiations for developing bulk stainless steel and mild steel joints using principles of MHH process. The developed joints via microwave processing routes were characterized by relevant techniques such as XRD, SEM, electron probe micro analyzer (EPMA), Vickers microhardness and tensile strength measurements. A nickel based powder of particle size  $40\mu\text{m}$  was used to form interface layer. The BSE images of microwave processed joint confirmed the complete melting and fusion of powders and joining of plates.

[Gupta and Kumar, 2014] [28] focused on joining of stainless steel by using microwave energy. The joining of stainless steel was carried out by using Ni-based powder (EWAC 1002 ET) as a sandwich layer and the concept of MHH have been applied in a domestic microwave oven for joint formation. The authors reported that joint was free from cracking, however, a

small amount of porosity was observed during microstructural investigations. It was reported that with increase in nickel content and exposure time, the joint tensile strength increased significantly.

**[Benedetto and Calvi, 2013]** [29] studied and investigated microwave heating of asphalt. The research was focused on MW heating for production and recycling of road pavement materials. The experiments were performed on a smaller scale in a laboratory for conventional and MW heating of asphalt and to validate the results numerical simulations was carried out. The microwave heating effect was quicker and was able to dry the sample from within, which was not obtained in conventional processes. Full dry samples were obtained with time savings of 200% by using microwaves.

**[Agrawal, 2013]** [30] reviewed and discussed the applications of microwaves in the field of processing of metallic powders in the field of sintering, melting and joining. Author reported the current status of applications of microwaves in the processing of metallic materials, including steel processing.

**[Bansal et al.,2012]** [31] carried out the joining of Inconel 718 plates by using nickel powder of mesh size 0.2 mm in interface layer. The process of joining was carried out at 2.45 GHz using domestic microwave applicator at 900 watt power. Relevant characterization techniques were used and authors claimed that the faying surfaces were well melted and fused with good metallurgical bonding on both sides of the base material. The joint strength analysis showed that average tensile strength was 400 MPa with 6% elongation.

**[Sharma and Gupta, 2012]** [32] developed the carbide reinforced (tungsten carbide based) metal matrix composite cladding on the austenitic stainless steel SS-316 substrate. The formed clad showed the presence of good metallurgical bonding with the substrate by partial dilution of the clad material. The presence of new phases was detected which includes WC, W<sub>2</sub>C, NiSi, NiW and Co<sub>3</sub>W<sub>3</sub>C. The developed clads were characterized by various relevant techniques such as XRD patterns, field emission scanning electron microscope equipped with energy dispersive X-ray detector and Vicker's micro hardness tester. The clad peel-off strength was analyzed by 3-point bend test and showed two distinct load transitions for matrix and reinforced phases. The clad was failed at upper transition load and further load was taken by SS-316 substrate. Due to good stiffness and adhesion characteristics, the developed clad retarded the crack formation due to the presence of soft Ni based matrix which exhibits the ductile behavior.

[Gupta et al., 2012] [33] developed cladding of EWAC + 20% Cr<sub>23</sub>C<sub>6</sub> on the substrate of austenitic stainless steel SS316 by using microwave radiation hybrid technique. The developed claddings were characterized by various relevant techniques like FE-SEM, EDS, XRD and measurement of Vicker's micro-hardness. The results of X-ray diffraction pattern of the clad showed the presence of chromium carbide, nickel silicide and nickel iron phases that contributed to the enhancement of micro-hardness of clads. Clads were formed of 1 mm thickness without any visible interfacial cracks. The microstructure of clads having a cellular like microstructure. The developed clads having a better micro-hardness than the substrate material. It was concluded that these claddings would be effectively used in wear resistance applications.

[Srinath et al., 2011] [34] investigated novel hybrid microwave heating approach for metallurgical joining of highly conductive copper metal. It was stressed that joining of copper using conventional techniques possesses many problems due to high conductivity capabilities. The research on joining of copper was carried out using microwave irradiation in 1 KW multimode applicator at 2.45 GHz and 900 W. The joint was obtained by introducing commercially available copper powder slurry between the pieces of bulk copper. The microstructure study, elemental analysis, phase analysis, micro-hardness testing, porosity measurement and tensile strength testing were used for joint characterization. The results revealed that the hardness of joint area was  $78 \pm 7$  Hv, while the porosity in the joint was observed to be as low as 1.92%. The strength of the copper joint formed by hybrid microwave heating showed approximately 29.21% elongation with an average ultimate tensile strength of 164.4 MPa.

[Srinath et al., 2011] [35] extended the work by joining of dissimilar metals through microwave hybrid processing. It was stated that joining of dissimilar metals is a relatively difficult task due to differences in chemical compositions, mechanical properties and thermal expansion coefficients of materials to be joined. A nickel based powder with a particle size of 40  $\mu$ m was used as a sandwich layer. The microwave joining of stainless steel (SS-316) to mild steel (MS) in bulk form was carried out using a multimode applicator at 2.45 GHz and 900 W, using charcoal powder as susceptor material. The microwave processed dissimilar butt joint showed Vickers micro-hardness of 133 (Hv) with low levels of porosity (approx. 0.58%). The hybrid microwave processed dissimilar joints showed ultimate tensile strength of 346.6 MPa with an elongation of 13.58%. The improved properties were reported due to

the complete melting and fusion of the interface layer and complete bonding with the bulk metals.

**[Gupta and Sharma, 2011]** [36] investigated the sliding wear performance of clad developed through hybrid microwaves novel approach on stainless steel (SS-316). Wear resistance cladding of Tungsten based was developed using microwave irradiations on SS-316 and were characterized through XRD, FESEM and Vickers microhardness. The tribological properties were analyzed using a pin on disc sliding apparatus against an EN-31 (HRC-70) hard faced surface. The developed clad showed significant resistance to the wear due to the presence of uniform distributed hard carbide phase. The microhardness study of microwave developed clad was in the range of  $1064 \pm 99$  Hv and porosity was significantly reduced to 0.89% approximately. Wear tests revealed that microwave clads showed 84 times better resistance to wear as compared to SS-316 steel corresponding to sliding speed of  $0.5 \text{ ms}^{-1}$ .

**[Lin et al., 2011]** [37] have focused on processing of nitride layers on a biomedical stainless steel substrate by the microwave plasma system. The nitrated layers were produced by using microwave plasma in an AST-MW1200W plasma reactor. The microwave plasma was generated by using a constant power of 700W at a frequency of 2.45GHz microwave radiations. The developed layers were characterized by SEM, TEM, XRD and Vicker's microhardness. The developed coating layers were found to have high microhardness and showed increased antibacterial properties.

**[Chandrasekaran et al., 2011]** [38] investigated the melting of tin, lead, copper and aluminium metals with microwave radiations. Melting was carried out using silicon carbide as a susceptor material with various levels of microwave power. It was concluded that microwave processing was twice faster as compared to conventional processing and energy requirements were lesser for microwave melting. It was stated that microwave melting of metals is an efficient and safer process than conventional routes. Further experimental results were modeled by lumped parameter model and predicted results were in agreement with the experimental results at same temperatures and conditions of processing.

## **2.2 Research Gaps**

The extensive literature survey on microwave material processing revealed that microwave irradiations are now used for high temperature applications in the field of manufacturing. The work carried out on joining of bulk metals and claddings on metallic materials enhanced the

research scope of microwave processing. Following gaps are reported which can be further attempted to widen this field of material joining.

1. The literature showed that very less work is reported on the joining of steels such as Hastelloy and SS-316 using microwave energy.
2. The comparison of joint strengths obtained by conventional TIG welding and microwave processed joints has not been reported so far.
3. It has been observed that very less work has been reported on the microstructural analysis of microwave processed and conventionally welded joints.
4. The literature analysis revealed that less work has been carried out on mechanical characterization of Hastelloy joints developed through microwave energy.

### **Objective**

The review of literature allowed the analysis of gaps in research and to fulfill the above stated gaps following research objectives has been defined:

1. To join the commercially available stainless steel SS-316 and Hastelloy steel through microwave energy using domestic microwave applicator at 2.45 GHz frequency using nickel powder slurry at faying surfaces.
2. To weld the steels using a conventional TIG welding process using filler rod of nickel.
3. To carry out the microstructural characterizations of microwave developed joints and TIG welded joints using SEM (scanning electron microscopy), EDS (energy dispersive X-ray spectroscopy), and XRD (X-ray diffraction) techniques.
4. To study the mechanical properties of joints in terms of Vickers microhardness and tensile testing.
5. To compare the results of both the joining process, i.e. microwave joining and TIG welding for maximum tensile strength of joints.
6. To study the fractographic analysis of the joint, fractured area and to understand the failure mode of joints.

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# Chapter 3

## Methodology

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### 3.1 Methodology

The research gaps were formed and objectives were developed for the present work on the basis of literature survey. To complete the objectives following methodology was used:

In the first phase of work pilot experiments were carried to join the Hastelloy and SS-316 using MHH principles in domestic microwave applicator. After studying the optimal processing time for joint formation, actual joints were processed. The joining process was carried out at 900 watts of microwave power for 8 minutes in domestic applicator. A Slurry of nickel powder was introduced between the faying surfaces and joint was protected from susceptor (Charcoal) by using thin sheet of the separator in the form of graphite sheet.

In the second phase of work conventional TIG welding was carried out on the Hastelloy and SS-316 steels using a filler rod of nickel.

The third phase consists of sample preparation for microstructural analysis of developed joints by microwave and TIG process and for tensile testing. For characterizations samples were polished using emery papers of grade 800, 1000, 1500, 2000 and 4000. Further polishing was carried out using 1 $\mu$ m alumina paste on cloth wheel machine at RPM of 1000. For tensile testing specimens were prepared as per ASTM Designation: E8/E8 M-09 standard having gauge length of 18 mm and width 3 mm.

In the fourth phase microstructural characterizations and mechanical characterizations were carried out using relevant techniques of SEM, EDS, XRD, Vickers microhardness, tensile testing and fractographic analysis.

In the final phase of work comparative evaluation of both the processes of joining was carried out to evaluate the quality of joints obtained.

The brief methodology is shown in Fig. 3.1, which shows the flow chart depicting the main phases of work.

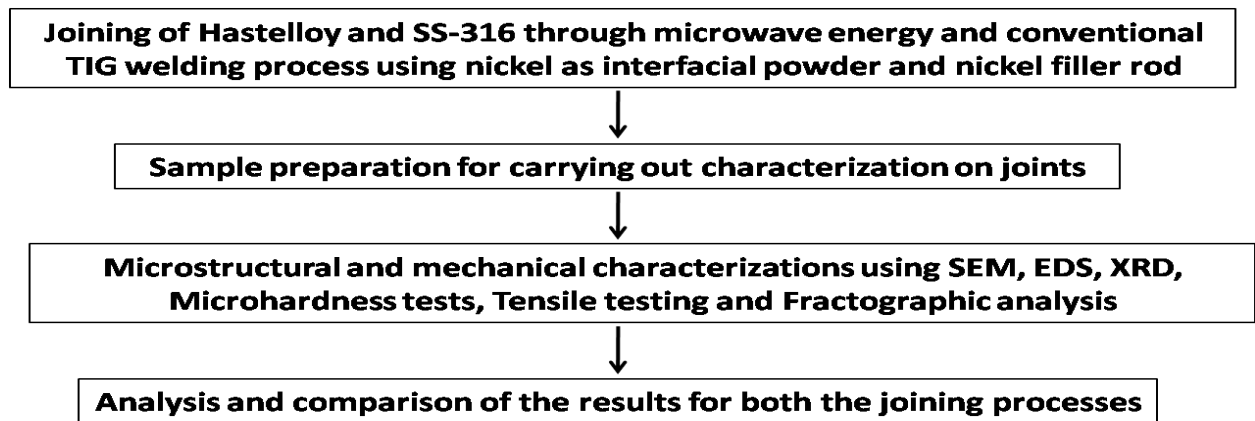


Figure 3.1: Flow chart showing the brief methodology

### 3.2 Selection Of Work Material

The material selected for joining process was determined by literature survey, which revealed that stainless steels such as SS-316 and corrosion resistance steels (Hastelloys) are used in many industrial applications including automobile parts, aerospace parts, marine applications, machines and structural components. These steels offer numerous advantages including improved life cycle, cost performance, improved reliability and lower maintenance. The effects of the addition of various alloying elements in these steels improve many properties. Additions of nickel, chromium, molybdenum and other elements are needed for many applications where a high level of corrosion resistance is required. Chromium provides resistance to oxidizing environments while molybdenum improves resistance to reducing environments. A combination of both chromium and molybdenum increases resistance to localized corrosion (pitting and crevice corrosion). Further, additions of tungsten increases resistance to localized corrosion of steels. The chemical compositions of selected steels for present work are given in Table 3.1.

Table 3.1: Chemical composition of selected steel materials

Sr. No.	Material	Elements Weight Percentage					
		Fe	Ni	Cr	Mo	C	Other
1	Stainless Steel SS-316	60 - 67	9 -12	18 – 21	2 -3	0.08	0.73 Si, 1.5 Mn
2	Hastelloy-C276	4 - 7	55-63	14.5-16.5	15 - 17	0.02	3-4.5W

### 3.3 Development Of Joints Through Microwave Energy

The joints of Hastelloy steel and SS-316 steel were developed in domestic microwave applicator by using techniques of microwave hybrid heating. The process parameters for carrying out microwave joining of steels are shown in Table 3.2.

Table 3.2: Process parameters for microwave joining of steels

Process Parameters	Descriptions
Microwave applicator	Domestic multimode microwave (Made: LG, Model: Charcoal)
Working frequency and maximum power rating	2.45 GHz and 900 watts
Exposure time	480 seconds
Workpiece material	Stainless steel (SS-316) and Hastelloy steel
Interfacial powder	Nickel powder (40 $\mu$ m average particle size)
Susceptor material	Fine grained charcoal powder
Separator material	99.9% pure thin graphite sheet

The schematic process of joining of steels through microwave hybrid heating is shown in Fig. 3.2 (a-b), which also shows the actual process of joint in microwave applicator.

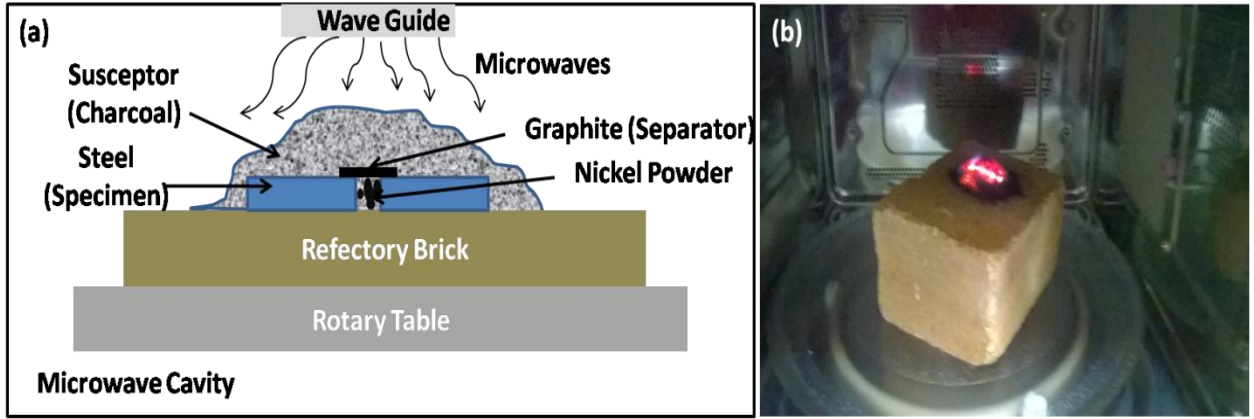


Figure 3.2: (a) Schematic microwave hybrid heating joining process and (b) actual processing of steel joints

For carrying out microwave joining, slurry of nickel powder and epoxy resin is applied at the faying surfaces such that on initial heating, the resin got evaporated from the joint region and leaving nickel powder. The nickel powder initially does not couple with microwaves and reflect the microwaves due to lower skin depth. The skin depth related to material for microwave processing is defined as the distance into the material at which the incident power drops to  $1/e$  (36.8%) of the surface value. On increasing the skin depth in a material, penetration depth of microwave radiation can be increased within the material. The skin depth ( $\delta$ ) is given by Eq.(3.9).

$$\delta = \sqrt{\frac{\rho}{\pi f \mu_r \mu_o}} \quad (3.9)$$

Where,

$\delta$  = skin depth ( $\mu\text{m}$ ),  $\rho$  = resistivity ( $\mu\Omega\text{-cm}$ ),  $f$  = frequency of microwaves (GHz),  $\mu_r$  = relative permeability,  $\mu_o$  = absolute permeability (H/m)

The skin depth of nickel powder was calculated using Equation (8). The calculated value of skin depth for nickel (considering  $\rho=8.707 \mu\Omega\text{-cm}$ ,  $f =2.45 \text{ GHz}$ ,  $\mu_o=4\text{p}\times 10^{-7} \text{ H/m}$  and  $\mu_r=600$ ) is approximately  $0.12 \mu\text{m}$ , which is relatively less than the particle size of the nickel powder particles used in the experiments. Hence, nickel particles cannot be processed directly with microwave radiations at room temperature and will tend to reflect the microwaves. To remove this restriction of skin depth, microwave hybrid heating was employed such that nickel powder was heated initially through conventional modes by

susceptor material, which raises the temperature of nickel powder quickly and powder starts coupling with the microwaves. After complete melting and fusion of powder, bulk solid interlayer is obtained which further do not allow microwaves interactions.

### 3.4 TIG Welding of Steels

Tungsten Inert Gas (TIG) welding or Gas Tungsten Arc Welding (GTAW) is an arc welding process that utilizes a non consumable tungsten electrode for welding. A tungsten electrode is used because it can withstand very high temperatures without getting damaged and minimal melting or erosion. Tungstenelectrodes are made by powder metallurgy route and are formed to size after sintering process.

In TIG welding electrodes small quantities of other metallic oxides are added which can offer the following benefits:-

- Facilitate easy arc starting .
- Increase arc stability .
- Improve current-carrying capacity of the rod .
- Reduce the risk of weld contamination .
- Increase electrode life by increasing erosion resistance and improving melting point.

The main oxides added are those of zirconium, thorium, lanthanum, yttrium or cerium. The torch of TIG contains provision for the flow of shielding gases, which prevents the contamination of weld. The schematic representation of the TIG welding process is shown in Fig. 3.3

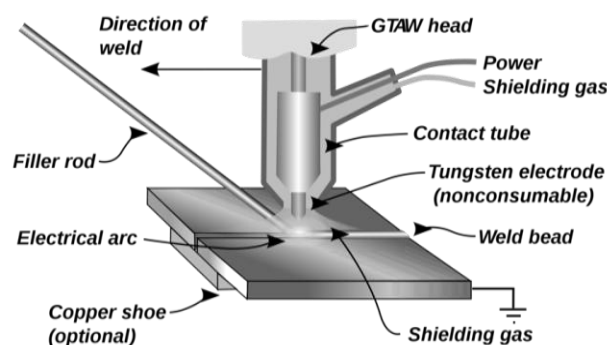


Figure 3.3: Schematic representation of TIG welding process

For welding of materials filler metal is required, which is melted by using electrode and fills the weld area. However, autogeneous welds do not require filler metal. The process of TIG is mainly used for welding of thin sections of stainless steel and non-ferrous metals such as aluminum or copper, and allows better control of operator on process in comparison to other conventional welding process. On the other hand it is one of the most difficult processes to master and require skilled labor. In present work nickel filler rod is used for welding of thin sections of stainless steels (SS-316) and hastelloy steel.

### **3.5 Characterization Techniques**

The term characterization in material science refers to the processes by which the materials microstructure, properties and correlation between structure and properties are investigated and measured. It is one of the fundamental processes in the field of materials science, without which scientific understanding of engineering materials is not possible. The scope of these processes can be different; some refer to the techniques which study the microscopic structure and properties of materials, while others refer to materials property analysis process including mechanical testing, thermal analysis and density calculations. There are various characterization techniques available which are being practiced for centuries, while some of them emerged recently. The techniques used in the present work for characterizations of microstructure and mechanical properties are briefly described in this section.

#### **3.5.1 X-Ray Diffraction (XRD)**

The X-ray diffraction (XRD) is an analytical technique which is primarily used for phase identifications of a crystalline material and it provide information on unit cell dimensions. The analysis of this technique can provides the average bulk composition of materials. X-ray diffraction is based on constructive interference of monochromatic X-rays and a crystalline sample. These X-rays are generated by a cathode ray tube which is filtered to produce monochromatic radiation, which are collimated to concentrate and directed on the sample. The interaction of the incident rays with the sample produces constructive interference and a diffracted ray when conditions satisfy Bragg's Law ( $n\lambda=2d \sin \theta$ ). This law relates the wavelength of electromagnetic radiation to the diffraction angle and the lattice spacing in a crystalline sample. These diffracted X-rays are then detected, processed and counted. By scanning the sample through the various ranges of  $2\theta$  angles, all possible diffraction directions

of the lattice can be attained. The conversion of the diffraction peaks to d-spacing allows identification of the mineral because each mineral has a set of unique d-spacing. Typically, this is achieved by comparison of d-spacing with standard reference patterns. All diffraction methods are based on generation of X-rays in an X-ray tube. These X-rays are directed at the sample, and the diffracted rays are collected. A key component of all X-Ray diffraction is the angle between the incident and diffracted rays. The X-ray diffractometers consist of three basic elements: an X-ray tube, a sample holder, and an X-ray detector as shown in Fig 3.4.

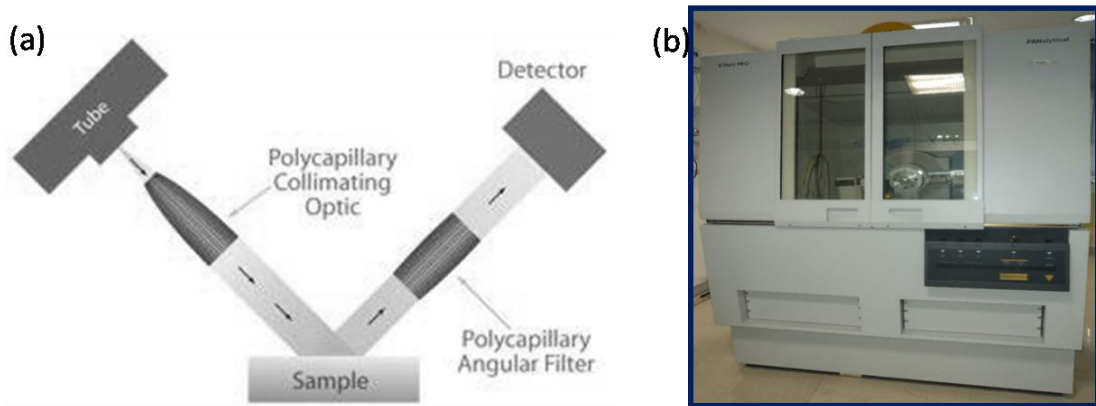


Figure 3.4(a) The main elements of X-Ray Diffraction system and (b) Actual X-Ray Diffractometer (Photo courtesy: SAI Labs, TU, Patiala)

X-rays are generated in a cathode ray tube by heating a filament to produce electrons which are accelerated toward the target by applying a voltage and bombarded on the target material. When electrons have sufficient energy to dislodge inner shell electrons of the target material, characteristic X-ray spectra are produced which produces the information regarding the phases present in the material. In the present work XRD is used to determine the phases of nickel powder, nickel rod, joints developed by microwave and TIG welded joints.

### 3.5.2 Scanning Electron Microscope (SEM) Equipped With Energy Dispersive X-ray Spectroscopy (EDS)

The scanning electron microscopy (SEM) is used to scan the surface area and is used to determine the microstructures of samples at higher magnifications which uses an electron beam to scanned sample's surface. When the beams of electrons strike the sample surface, then a variety of signals are generated and it is the detection of specific signals which produces an image or a sample's elemental composition. The three signals which provides the

greatest amount of information in SEM are the secondary electrons, backscattered electrons, and X-rays.

The secondary electrons are emitted from the atom which occupies the top surface of the sample and produces a readily interpretable image of the surface. The contrast in the image is determined by the sample morphology. A high resolution image can be obtained because of the small diameter of the primary electron beam. Backscattered electrons are primary beam electrons which are reflected from atoms of solid surface. The contrast in the image produced is determined by the atomic number of the elements in the sample. The image will therefore show the distribution of different chemical phases in the sample. Because these electrons are emitted from a depth in the sample, the resolution in the image is not as good as for secondary electrons.

The interaction of the primary beam with atoms in the sample causes shell transitions which result in the emission of an X-ray. The emitted X-ray has an energy characteristic of the parent elements. The detection and measurements of the energy permit elemental analysis (Energy Dispersive X-ray Spectroscopy or EDS). An EDS can provide rapid, qualitative, or with adequate standards, quantitative analysis of elemental composition with a sampling depth of 1-2 microns. X-rays may also be used to form maps or line profiles, showing the elemental distribution in a sample surface. Fig. 3.5 shows the principle of SEM analysis and SEM equipment equipped with EDS.

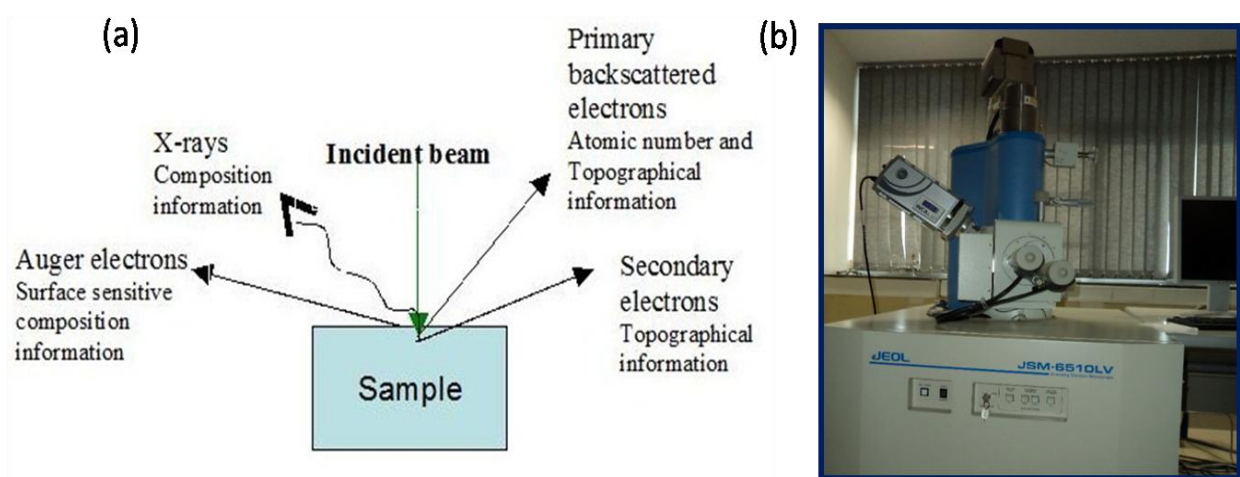


Figure 3.5: (a) SEM principle for analysis of surface and (b) SEM equipment equipped with EDS (Photo courtesy: SAI Labs, TU, Patiala)

### 3.5.3 Vickers Microhardness Testing

The hardness test and tester was developed in 1921 by Robert L. Smith and George E. Sandland at Vickers Ltd. as an alternative to Brinell hardness testing. The basic principle, as with all common measures of hardness, is to observe the questioned material's ability to resist plastic deformation from a standard source. The Vickers test can be used for all metals and has the widest scales among hardness tests. The unit of hardness given by the Vickers test is known as the Vickers Pyramid Number (HV) or Diamond Pyramid Hardness (DPH). Micro hardness testing is a method for measuring the hardness of materials at a microscopic scale. The Vickers microhardness tester is shown in Fig 3.6.

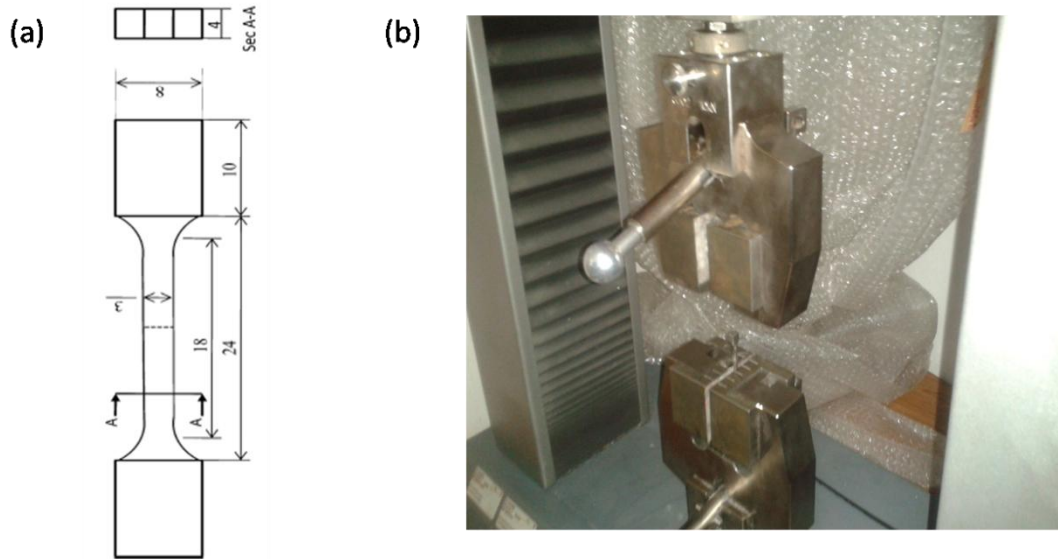


Figure 3.6: Microhardness tester (Photo courtesy: Mechanical Department, TU, Patiala)

### 3.5.4 Tensile Testing

Tensile testing is one of the fundamental material tests in which sample is subjected to a desired and control tensile load until the failure occurs. The main data which is acquired from the tensile test includes ultimate tensile strength, maximum elongation and reduction in area. These results can be further utilized to obtain main properties of the material including Young's modulus, Poisson's ratio, yield strength and strain-hardening characteristics. For

isotropic materials, uniaxial tensile testing is carried out and for an anisotropic material biaxial tensile tests are carried out. Tensile tests are carried out as per standards decided by material testing agencies worldwide such as ASTM standards. For the present work ASTM E8/E8 M-09 standard specimens were formed and dimension of standard specimen is shown in Fig. 3.7 (a) and tests were carried on Zwick-Roell Z010 tensile testing machine at a strain rate of 0.083 mm/s. The tensile testing equipment is shown in Fig. 3.7(b).



All dimensions are in mm

Figure 3.7: (a) Standard tensile test specimen and (b) Zwick-Roell Z010 tensile testing machine (Photo courtesy: Chemical department, TU, Patiala)

All the testing and characterizations were carried out in Thapar University, Patiala.

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# Chapter 4

## Results And Discussions

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This chapter presents the experimental and characterization results of the SS-316 and Hastelloy steel joints obtained by microwave hybrid heating and conventional tungsten inert gas welding process. An attempt has been made to study the microstructures and joint strength obtained by two different processes such that the microwave processing is compared and its usefulness is evaluated with conventional welding process. Further, fractographic analysis was carried out to understand the failure mechanism of joined samples under tensile testing.

### 4.1 Characterization of Nickel Powder And Nickel Filler Rod

The joints through microwave energy were obtained by applying a slurry of nickel powder at the faying surface. The typical SEM image of nickel powder is shown in Fig 4.1, which shows spherical particles of powder having an average size of 40  $\mu\text{m}$ . The XRD pattern of powder is shown in Fig 4.1 which shows the dominant presence of nickel.

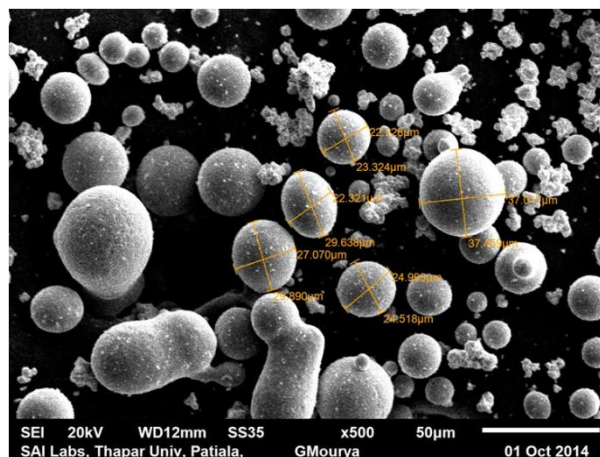


Figure 4.1: Typical SEM image of nickel powder showing spherical morphology

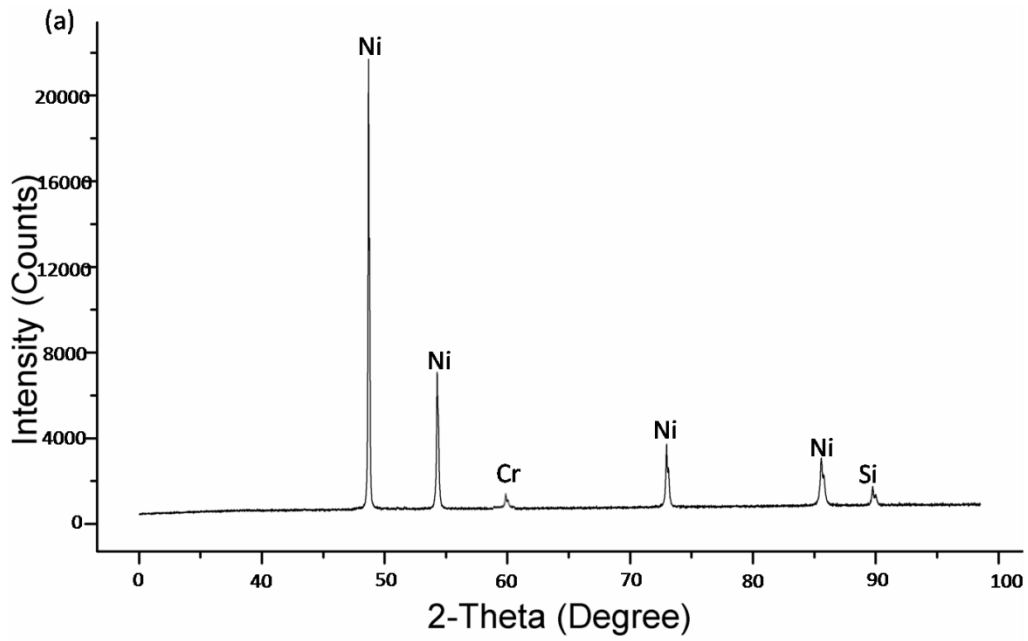


Figure 4.2: Typical XRD pattern for nickel powder

The TIG welding of steels was carried out by using a nickel filler rod and XRD pattern of rod is shown in Fig.4.3, which shows the presence of nickel as main constituent with chromium and iron.

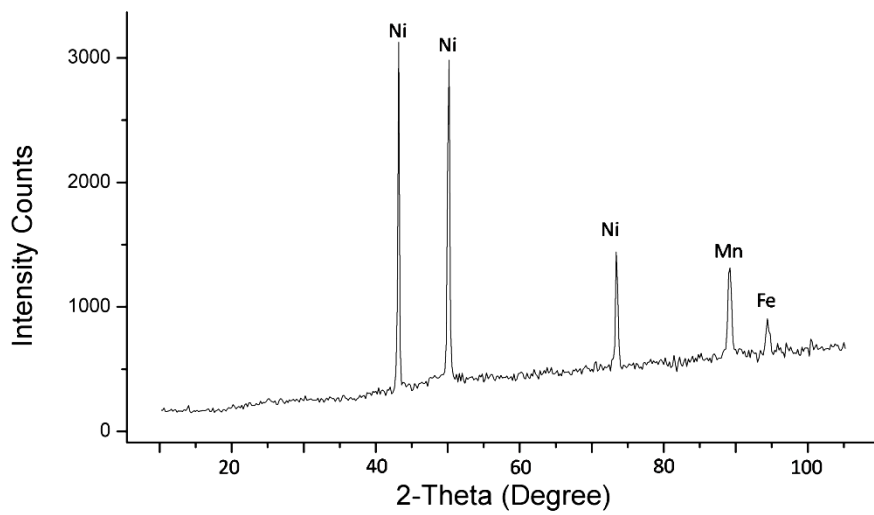


Figure 4.3: Typical XRD spectrum of nickel filler rod

The XRD diffraction analysis of nickel powder and filler rod was carried on X'PERT PRO of PAN ANALYTICAL using  $\text{CuK}\alpha$  radiations at a scanning rate of  $1^\circ \text{ min}^{-1}$  in the  $2\theta$  range of  $10\text{--}110^\circ$ .

## 4.2 Characterization of Joints

The microwave processed joint and TIG welded joint of SS-316 is shown in Fig 4.4 (a-b). These joints were subjected to metallurgical characterizations by using XRD, SEM and EDS analysis at joint cross sections.

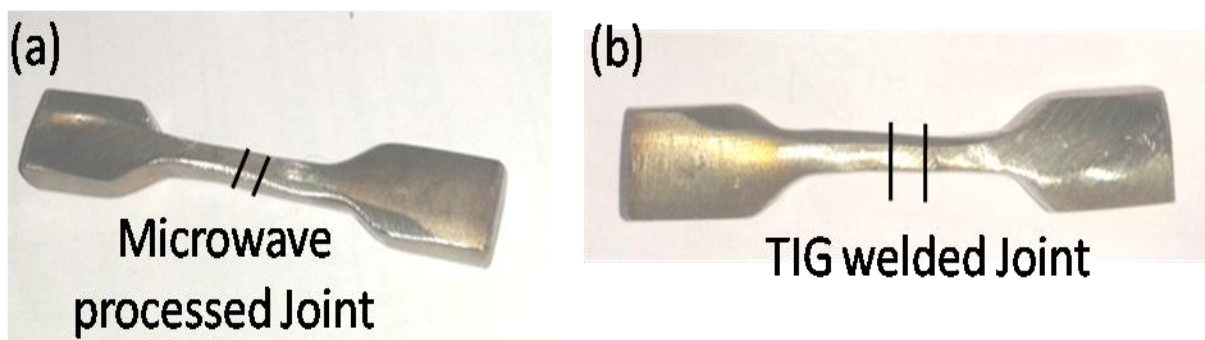


Figure 4.4: Joints of SS-316 steel by (a) Microwave joining and (b) TIG welding

Prior to the characterizations, samples were prepared by cutting the joint cross sectional using low speed diamond cutter. The samples were polished properly as stated in methodology section and were kept in plastic bags to prevent any unwanted contamination of joints.

### 4.2.1 XRD Analysis of Joints

The typical XRD spectrums of microwave processed joint of SS-316 and Hastelloy steel are shown in Fig. 4.5 and Fig.4.6, which were obtained by placing nickel powder at the interface.

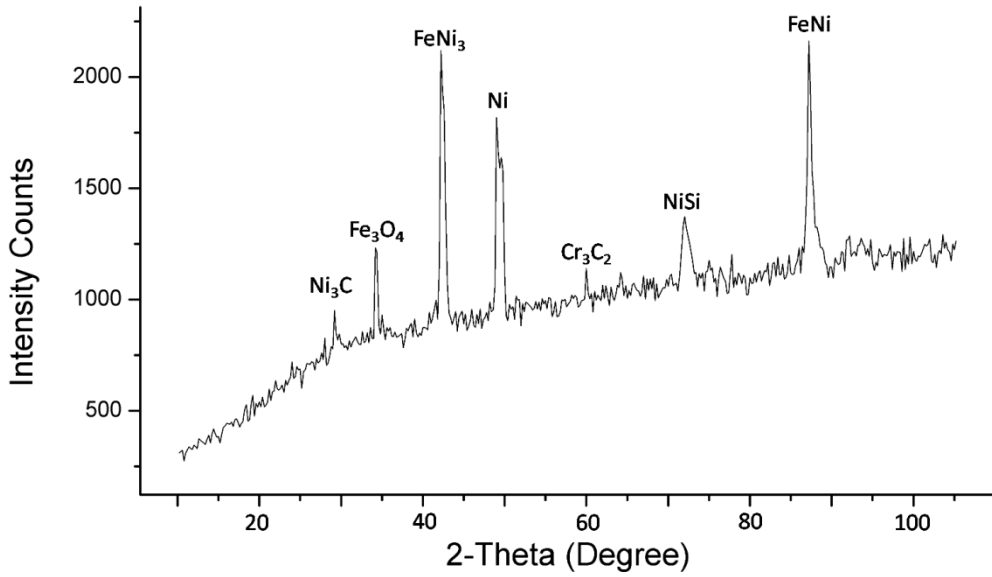


Figure 4.5: XRD spectrum of SS-316 microwave processed joint

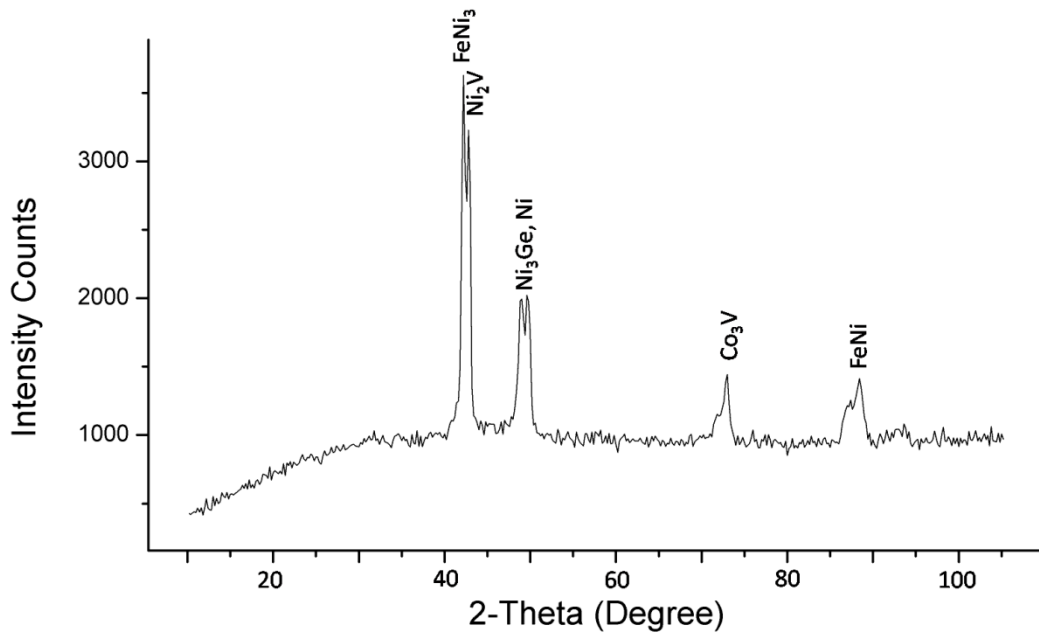


Figure 4.6: XRD spectrum of Hastelloy steel microwave processed joint

The results of XRD tests showed the presence of phases of free nickel in both the microwave processed joints of SS-316 and Hastelloy, which is the major constituent of interfacial powder. In SS-316 joint presence of phases of Ni<sub>3</sub>C, Fe<sub>3</sub>O<sub>4</sub>, FeNi<sub>3</sub>, Cr<sub>3</sub>C<sub>2</sub>, NiSi and FeNi were reported. The phases of nickel are due to the slurry of powder, however, the presence of

iron phases revealed that elements from the substrate were mixed with the interfacial powder. This shows complete fusion and metallurgical bonding of powder with the bulk steel. Microwave processing allowed the formation of some hard carbides and intermixing of elements from substrate to powder and vice versa. In the Hastelloy joint more phases of substrate elements were present mainly phases of Vanadium. The presence of  $\text{FeNi}_3$ ,  $\text{Ni}_2\text{V}$ ,  $\text{Co}_3\text{V}$ ,  $\text{Ni}_3\text{Ge}$  and free Ni were revealed.

The typical XRD spectrum of conventional TIG welded joint of SS-316 steel and Hastelloy is shown in Fig.4.7-4.8, which were obtained by using a filler rod of nickel. The dominant presence of nickel with traces of manganese and iron were reported in a XRD spectrum of rod.

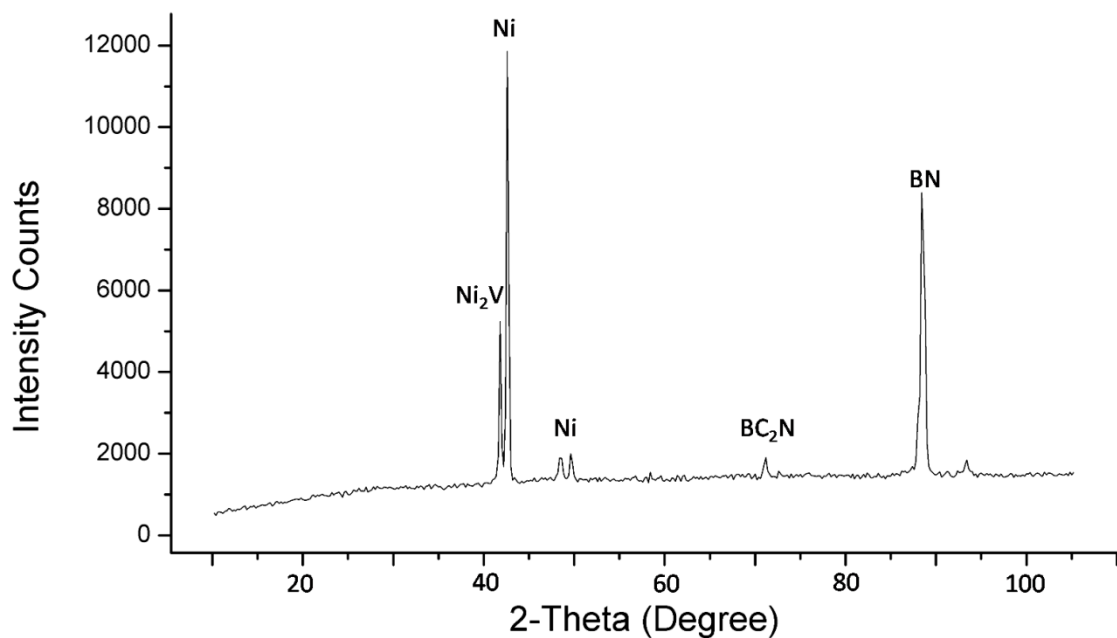


Figure 4.7: XRD spectrum of SS-316 steel joint processed by TIG welding process

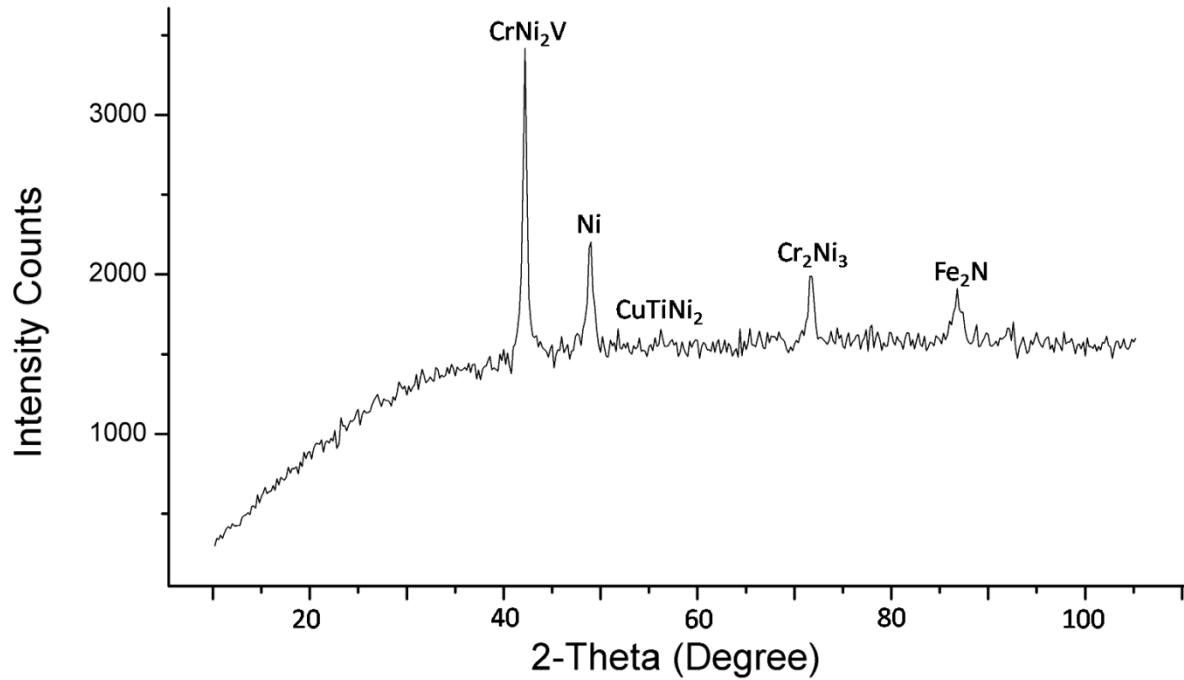


Figure 4.8: XRD spectrum of Hastelloy steel joint processed through TIG welding process

The presence of nickel is shown in both the materials with the formation of some complex phases such as  $\text{Ni}_2\text{V}$ ,  $\text{BC}_2\text{N}$  in SS-316 and  $\text{CrNi}_2\text{V}$ ,  $\text{Cr}_2\text{Ni}_3$  and  $\text{CuTiNi}_2$  in the joint of hastelloy steel. Presence of copper may be due to the contamination of nickel rod. The formations of carbides are lower in case of conventional TIG welding whereas, presence of carbides in microwave processed joints were may be due to the absorption of carbon from the graphite sheet during heating.

#### 4.2.2 Microstructural Analysis of Joints

The joints formed by microwave processing of SS-316 and Hastelloy steel are shown in Figure 4.9 (a-b), which clearly shows the joint zone in SEM images.

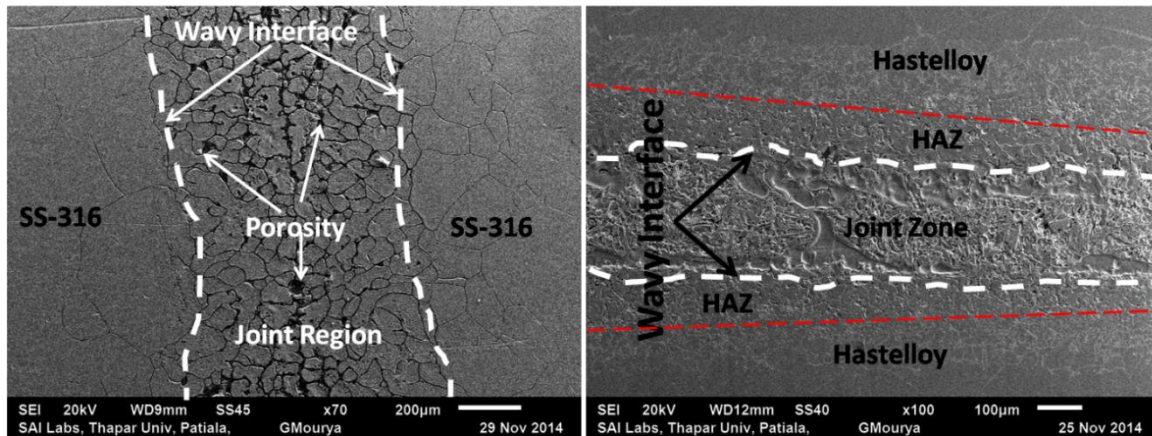


Figure 4.9: Microwave processed joints of (a) SS-316 steel and (b) Hastelloy steel

The joint region obtained by a microwave shows wavy interface which confirms the mutual diffusion of base metal and interfacial powder. Joint of SS-316 shows the presence of porosity; however, clean joint is obtained in case of Hastelloy joint. Heat affected zones are also visible in the SEM micrographs.

At higher magnifications, SEM micrograph shows the cellular structure of the joints obtained through microwave processing as shown in Fig. 4.10 (a-b). The EDS analysis of microstructure revealed that joint cells are mainly nickel and iron rich; however interfaces consists of carbide rich (chromium, nickel and iron) matter as shown in Fig.4.11.

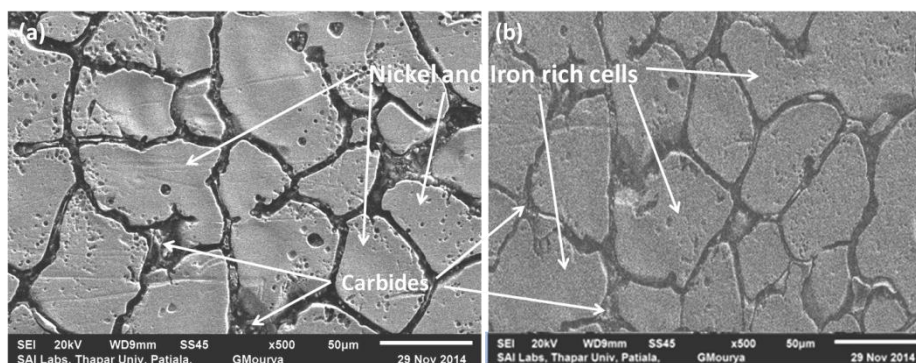


Figure 4.10: Microstructure of microwave processed (a) SS-316 and (b) Hastelloy joint

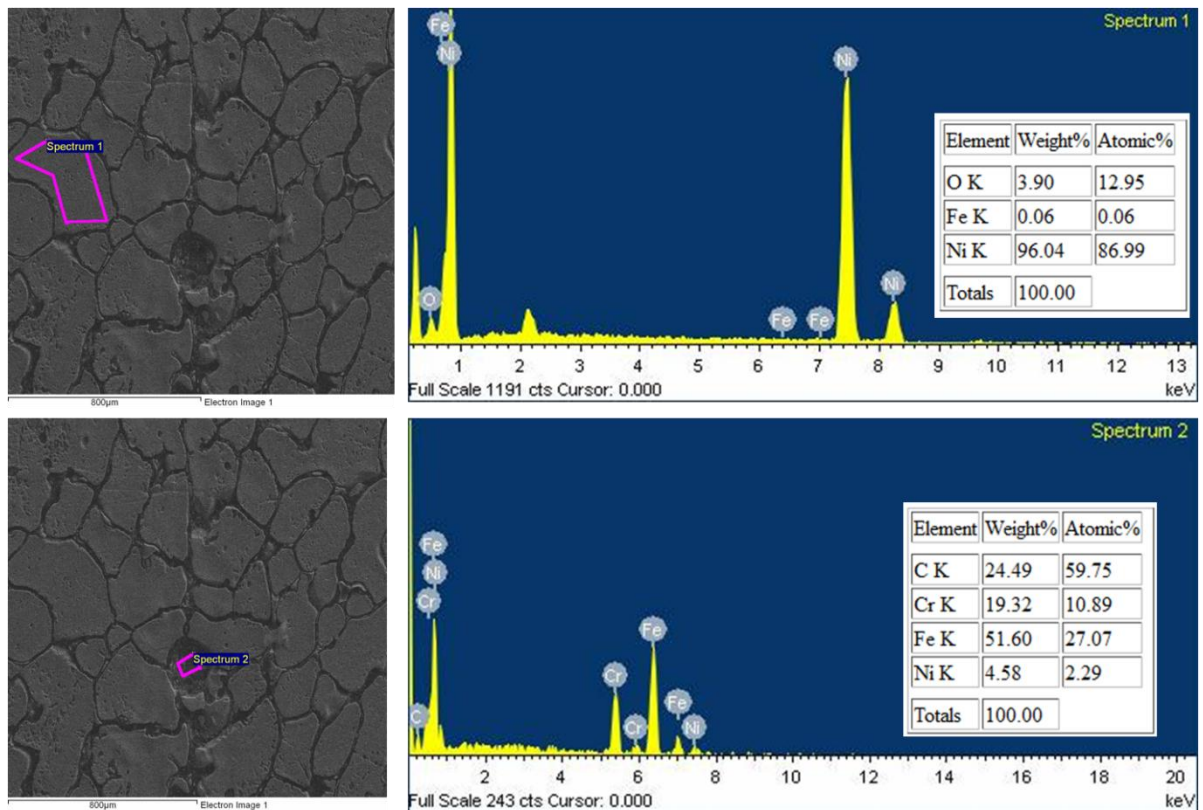


Figure 4.11: Elemental distribution on cell surface and cell boundary

The microstructure of TIG welded steel joints are shown in Fig. 4.12, which shows welding bead and base metal.

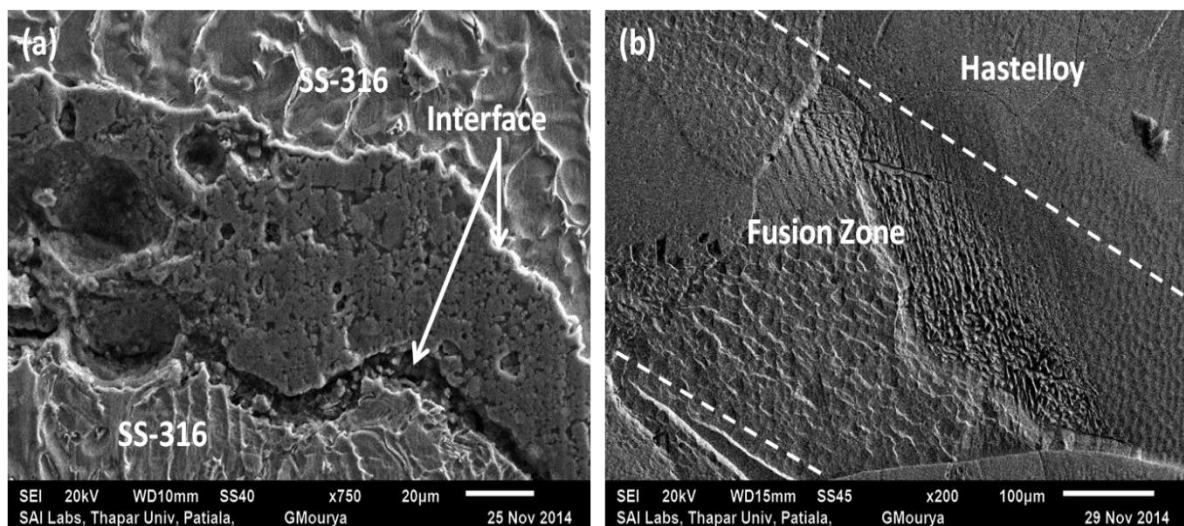


Figure 4.12: SEM micrographs showing joint of TIG welded (a) SS-316 and (b) Hastelloy steel

steel

The weld zone is clearly visible in the SS-316 joint; however, it is very difficult to observe joint region in Hastelloy steel due to highly diffusion and homogeneous structure of weld zone. Higher magnification SEM micrographs show skeleton like structure with dendrites in weld joint as shown in Fig. 4.13 (a-b).

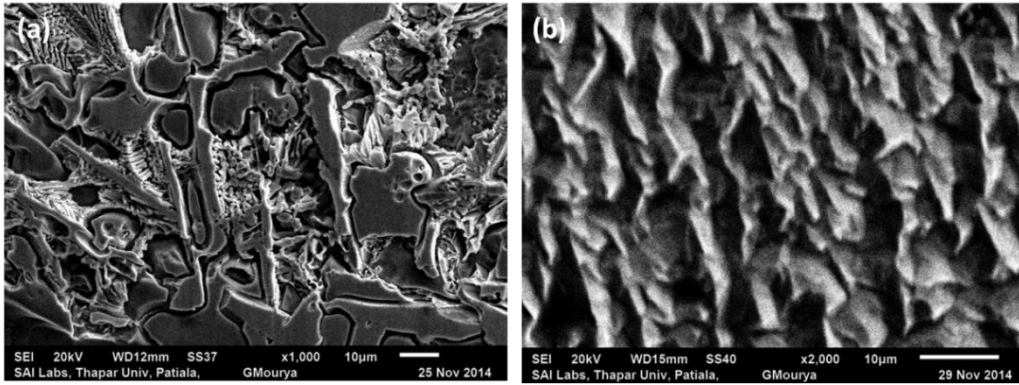


Figure 4.13: Dendrite type structure of TIG welded (a) SS-316 and (b) Hastelloy steel

The results of EDS showing the elemental distributions of TIG welded joints are shown in Fig.4.14 (a-b).

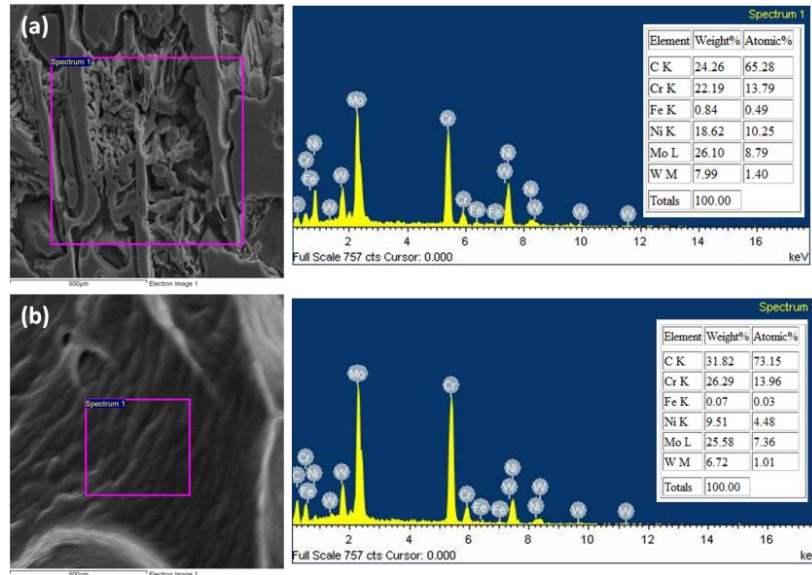


Figure 4.14: EDS analysis of TIG welded (a) SS-316 and (b) Hastelloy steel

The analysis shows the presence of tungsten in the weld region, which may be due to the diffusion of tungsten electrode during welding. Further, presence of chromium, nickel, molybdenum, iron and carbon can be seen.

### 4.2.3 Mechanical Characterization of Joints

The joints were characterized by mechanical testing of joints. Vickers microhardness and tensile testing was carried out on the joints to evaluate the strength of joints obtained by microwave and TIG process. The values of Vickers microhardness of specimens are shown in Table 4.1 and typical indentations are shown in Fig.4.15.

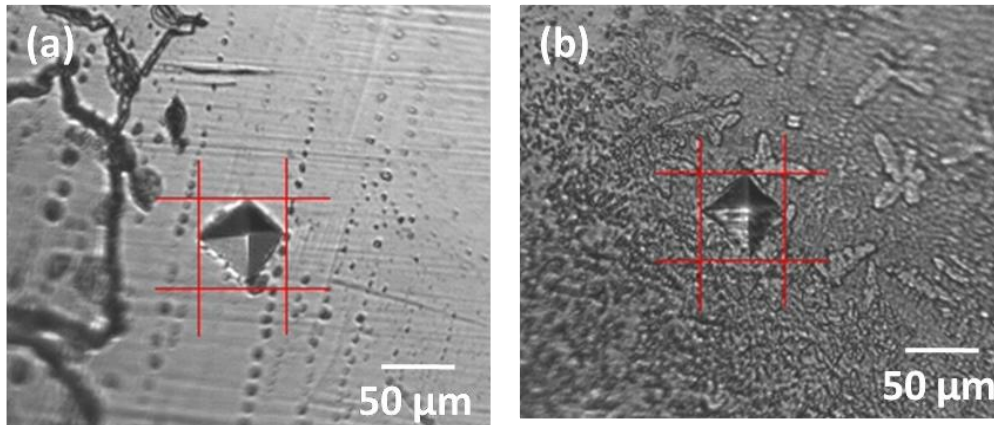


Figure 4.15: Typical SEM images showing indentations of diamond indenter on SS-316 steel  
(a) substrate and (b) joint

Table 4.1: Readings of Vickers microhardness tests

Material	Vickers Microhardness (HV)				
	Base Metal	Microwave Processed		TIG Welded	
		Interface	Joint	Interface	Joint Region
SS-316	230±5	280±5	348±10	292±5	323±10
Hastelloy-C276	325±5	387±5	478±10	388±5	450±10

Microhardness results revealed that in the joint region the value increased significantly due to the presence of carbides in the region for both the processes. Hardness at interfaces is better in TIG welded joint which may be due to the presence of the tungsten element in the microstructure. However, the hardness values in the joint region for microwave processed steels, weld was more than TIG steels due to the higher amount of carbides in the joint zone.

Indentation resistance in the joint region is higher due to the harder phase, but deeper and elastic indentation is formed on the base metal surface.

The results of the tensile testing of joints are shown in Table 4.2, which presents the ultimate tensile strength (UTM) and % elongation.

Table 4.2: Results of tensile strength of joints

Material	Microwave Processed Joint		TIG Welded Joint	
	$\sigma_M$ (MPa)	% elongation	$\sigma_M$ (MPa)	% Elongation
SS-316	344	1.6	285	6.4
Hastelloy	318	1.8	258	1.8

The fractured samples of tensile test are shown in Fig.4.16.

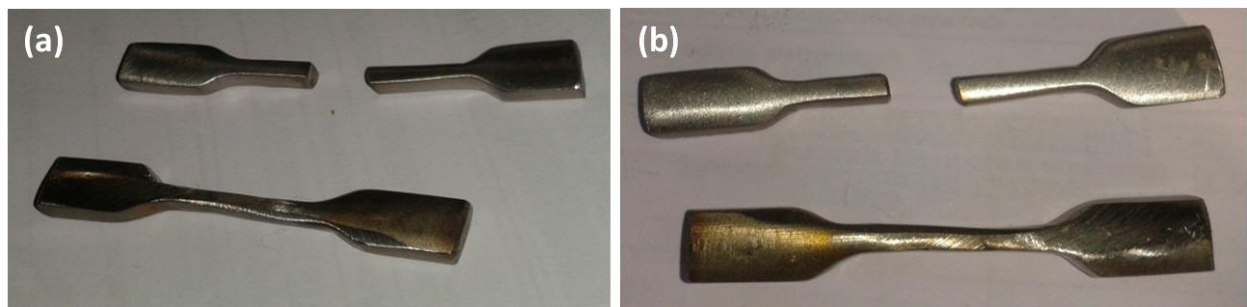


Figure 4.16: Tensile tested SS-316 specimens (a) Microwave processed and (b) TIG welded

Results revealed that the maximum tensile strength of microwave processed joints are better than the TIG welded joints; however the percentage elongation of TIG welded joints are better. The possible reason for a higher strength of microwave joints may be lower defects like porosity and cracks, better diffusion of nickel powder with base metal. To understand the lower percentage elongation, fractographic analysis was carried out and is discussed in detail in the next section.

### 4.3 Fractographic Analysis

Study of fractured surfaces allows the understanding of the behavior of the mode of failure during tensile testing. Testing of specimens has shown that TIG welded joint showed cup-cone type ductile behavior, but no information was obtained for microwave processed joints. The SEM images of fractured surfaces are shown in Fig.4.17.

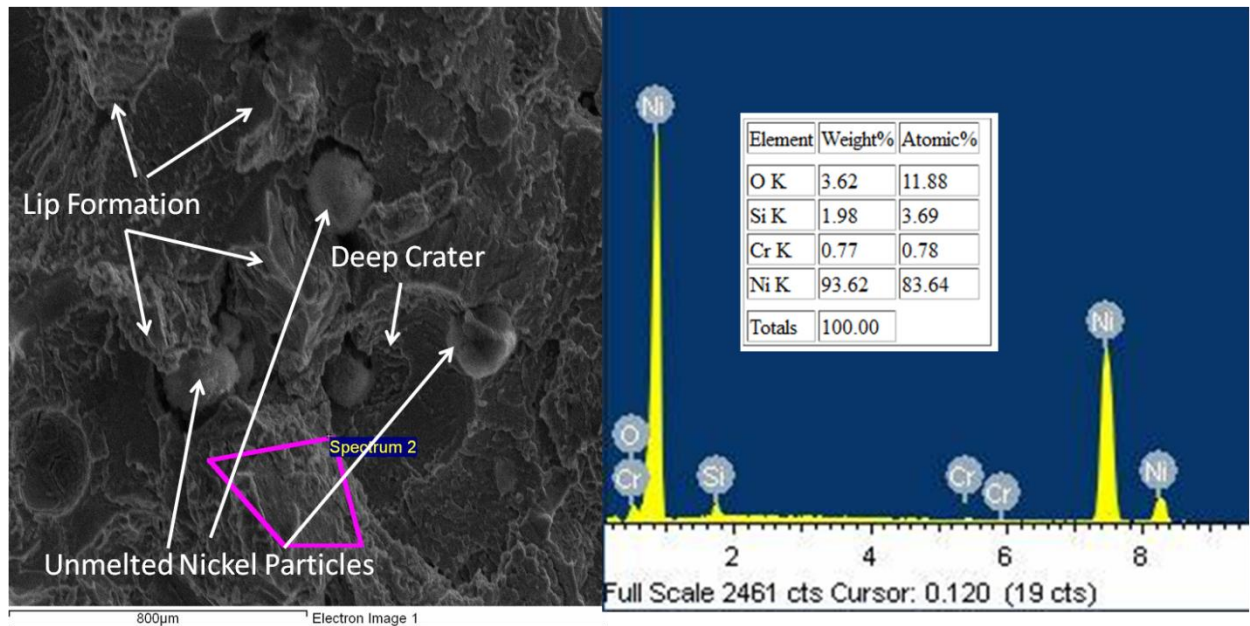


Figure 4.17: SEM image of microwave processed Hastelloy fractured surface with EDS analysis

Analysis of microwave processed joints fractured area shows a mixed mode of failure with ductile as well as brittle fracture. The formation of deep craters shows brittle fracture whereas lip formations with dimples on nickel surfaces show ductile behavior. The EDS analysis has shown that the main element at the fractured surface is nickel. The reason for the low percentage elongation of microwave processed joints may be due to unmelted and unfused particles of nickel present in the joint. This may be due to less exposure of joints to the microwaves, which can be reduced by increasing the exposure time.

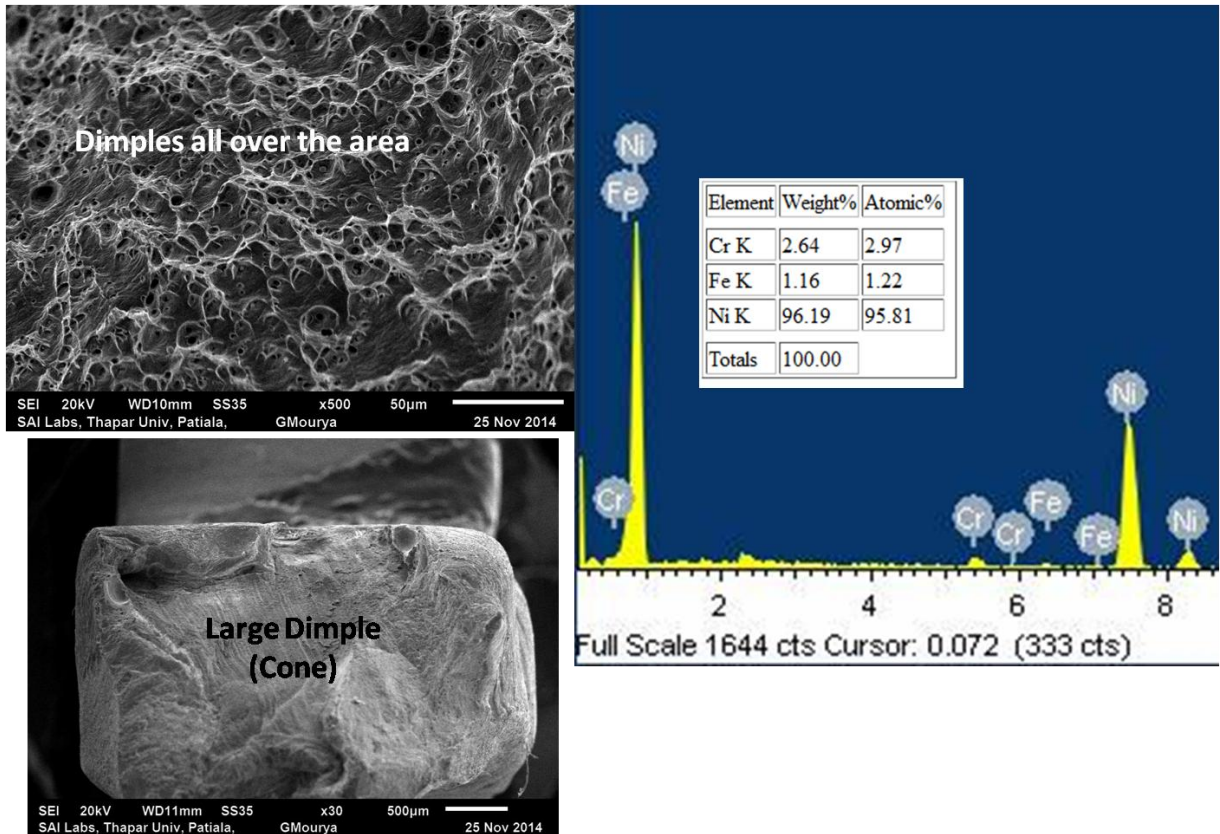


Figure 4.18: Fractured area of TIG welded hastelloy steel with EDS analysis

The fractured surface of TIG welded joint of hastelloy shows cup-cone type ductile behavior with formation of large dimples on fractured area. Typical morphology of ductile behavior is shown in Fig.4.18, which shows lip formation everywhere. The EDS analysis again shows higher percentage of nickel matrix at fractured surface.

#### 4.4 Comparative Analysis of Joint Properties

The microstructural characterizations of microwave processed joints showed lower defect formations including porosity and cracks whereas TIG welded joints showed cracks and porosity. These defects, weaker phases and higher heat affected zone leads to lower ultimate tensile strength in comparison to microwave joined steel joints. The compared results are shown graphically in Fig.4.19, which shows that percentage increase in  $\sigma_M$  of microwave processed joints is 20.7% for SS-316 and 23.25% for Hastelloy.

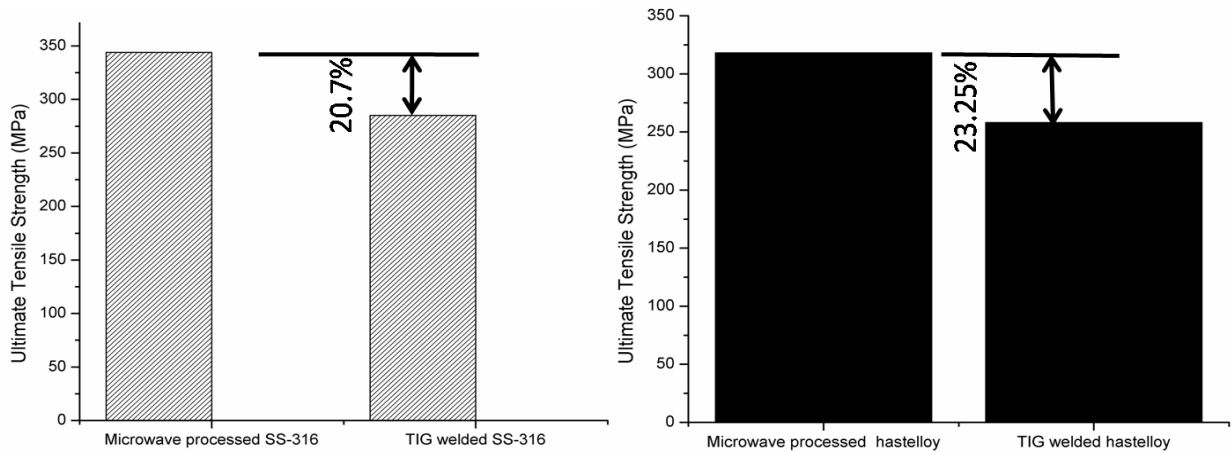


Figure 4.19: Comparison of tensile strength of SS-316 and Hastelloy steels joints produced by microwave and TIG

The percentage increase in Vickers microhardness of microwave processed jointly, with respect to TIG joint of SS-316 is 6.22% and for Hastelloy steel is 7.73%. The comparison of results is shown in Fig.4.20.

The overall results of analysis of joints showed that microwave processed joints produced superior mechanical properties, however the percentage elongation was lower than TIG welded joints. This provides good scope for future investigations in the process of material joining through microwaves.

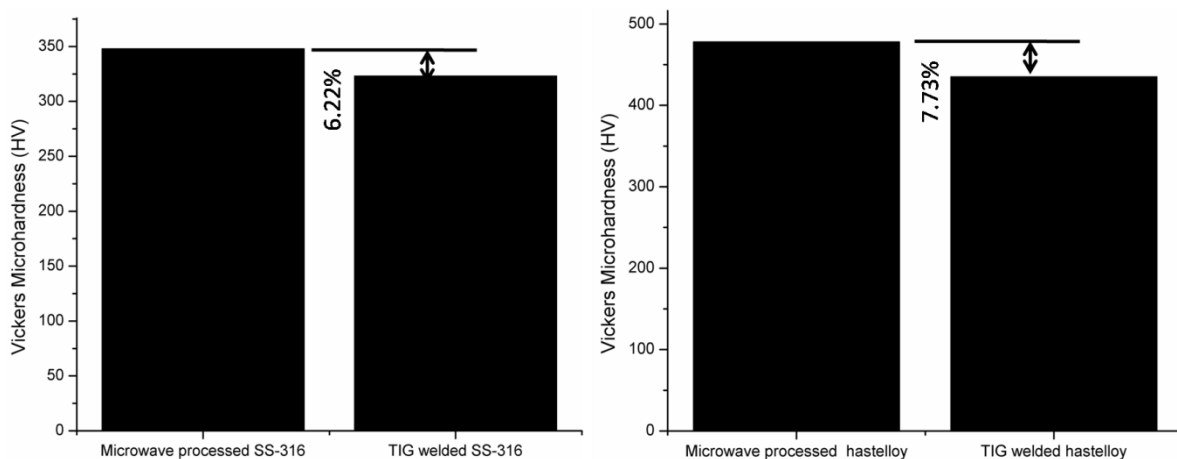


Figure 4.20: Comparison of Vickers microhardness at joint section of microwave and TIG welded steels

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# Chapter 5

## Conclusion And Future Scope

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### 5.1 Conclusion

The processing of materials through microwave energy has emerged as a novel material processing technique, which has extended its domain from sintering of ceramics to metallic materials. Further, high temperature applications using microwaves were carried out such as joining of bulk metals and claddings on the bulk metallic substrate. The present work was based on joining of steels through microwave energy and conventional TIG welding of SS-316 and Hastelloy. Following major conclusions are drawn from the work:

1. The joining of steels was successfully attempted by using nickel based powder at the faying surfaces, through microwaves in domestic multimode microwave oven.
2. The characterizations were carried out on the joints and it was shown that microwave processed joint showed cellular structure with lower defects.
3. The mechanical characterizations of developed joints showed that microwave processing, enhanced the mechanical strength due to better metallurgical bonding and diffusion, with lower processed defects. Ultimate tensile strength was 20.7% more than TIG welded SS-316 joint and 23.25% more than TIG welded Hastelloy steel.
4. Higher hardness was obtained in microwave processed joints due to the absorption of carbon from graphite sheet, which led to the formation of carbides in the microstructures.
5. Fractographic analysis showed a mixed mode fracture for microwave processed joint and ductile behavior for TIG joint. The brittle behavior was due to the presence of unmelted nickel particles in the joint region, which allowed brittle failure by particle pull out to create craters.

## 5.2 Future Scope

Following points can be considered for future work in the field of microwave material joining:

1. To study the effect of microwave exposure time on ductility of joint in terms of percentage elongation.
2. Evaluation of microstructure elemental composition by using different separator like alumina or silicon carbide sheets, which will prevent the addition of carbon in the joint.
3. To study the effect of microwave exposure time on fractographic analysis of joints.
4. The joining of hard to weld materials can be carried out by using microwaves.
5. Optimization of welding parameters can be carried out.
6. Computational work on microwave processing of metallic materials can be attempted through multi physics software's such as ANSYS and COMSOL.

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