JOINING AND CHARACTERIZATION OF SS-430 USING MICROWAVE PROCESSING METHOD

A dissertation submitted in partial fulfilment

of the requirements for the degree of

Masters of Engineering

in

Production Engineering

by

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July, 2016

CERTIFICATE

Certified that the thesis entitled "Joining and Characterization of SS-430 using Microwave Processing Method" which is being submitted by me, Navjot Singh (Roll No.801482017) to the Department of Mechanical Engineering, Thapar University, Patiala, Punjab in partial fulfilment of the requirements for the award of the degree of **Master of Engineering**, in **Production Engineering** a record of bonafide research work carried out by me under the guidance of Dr. Vinod Kumar Singla. The matter presented in this thesis has not been submitted in any other university or institute for the award of any other degree.

Date: 14-07-2016

(NAVJOT SINGH)

This is to certify that the above statement made by the candidate is correct to the best of our knowledge.

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ABSTRACT

Heating of the materials by using the radiations of the microwave is not a new application; some newer applications are emerging in the field of processing of materials by using the microwave radiations due to which it becomes a novel processing technique. Many authors have worked on ceramic materials and their sintering by using the microwave radiations. Now a days the applications of the microwave is being extended up to the sintering of the metallic powders. In the past years; many researches has processed the metallic powders and their sintering by using the microwave radiations; and used them in different applications like cladding as well as joining of the materials. The present work is based upon the joining of SS-430 materials through microwave radiations. XRD, EDS and SEM were used to characterize the prepared samples. Mechanical characterization like tensile testing was performed to saw the strength of the joint and Vicker's micro hardness tester was used to check the hardness of the joint. Observed results showed that the SS-430 material was successfully joined by the microwave radiations by using nickel as a base powder (EWAC). A cellular structure having less defects was observed by using the metallurgical characterizations. The mechanical characterizations of joints shows that due to better metallurgical bonding and diffusion, mechanical strength was increased and also the defects were less. The ultimate tensile strength which was observed during the experiment was 471 MPa. Hardness was higher of the joint due to the presence of carbon which was absorbed from graphite sheet resulted in the formation of carbides in the microstructures of microwave processed joints.

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials.
EDS	Energy Dispersive X-Ray Spectroscopy.
EM	Electromagnetic Spectrum.
HAZ	Heat Affected Zone.
MHH	Microwave Hybrid Heating.
SEM	Scanning Electron Microscopy.
UTS	Ultimate tensile strength.

CHAPTER 1 INTRODUCTION

1.1 INTRODUCTION

In the present time where the demand is changing day by day in every field; we need such kind of techniques which will not only be effective but also will be efficient. The processing of different materials like metal, non-metals, ceramics, composites etc. with good quality, less processing time and having less impact on the environment is quiet challenging. So we need such kind of techniques which will not only be less time consuming but also should be eco-friendly. The unique characteristics of material processing by microwaves is attracting researchers and lot of research is being carried out in joining, cladding and casting of material with the help of microwaves due to its inherent properties like less processing time, volumetric heating, very less heat transfer to the environment etc.

1.2 METAL JOINING METHOD

Two work piece having same or different material are joined together by suitable heat and pressure or heat alone or pressure alone by using some filler material or without that to form a metallurgical bond is known as joining. Adhesive bonding, welding, soldering, brazing etc. comes under the conventional joining method whereas LASER welding, microwave joining, ultrasonic welding, electron beam welding etc. are some of the advanced joining method. From all of above techniques; microwave joining process is newly developed and has so many unique characteristics which are absent in other processes.

Metal joining process depends on number of factor some of them are as follow:

- Types of material to be joined.
- Physical, chemical and metallurgical properties of metal.
- Economy and cost effectiveness of the joining process.
- Properties to be obtained by the joint.

1.2.1 Different Types of Metal Joining

Metal joining mainly divided into two broad categories Fusion and other welding techniques as shown in the Figure 1.1.

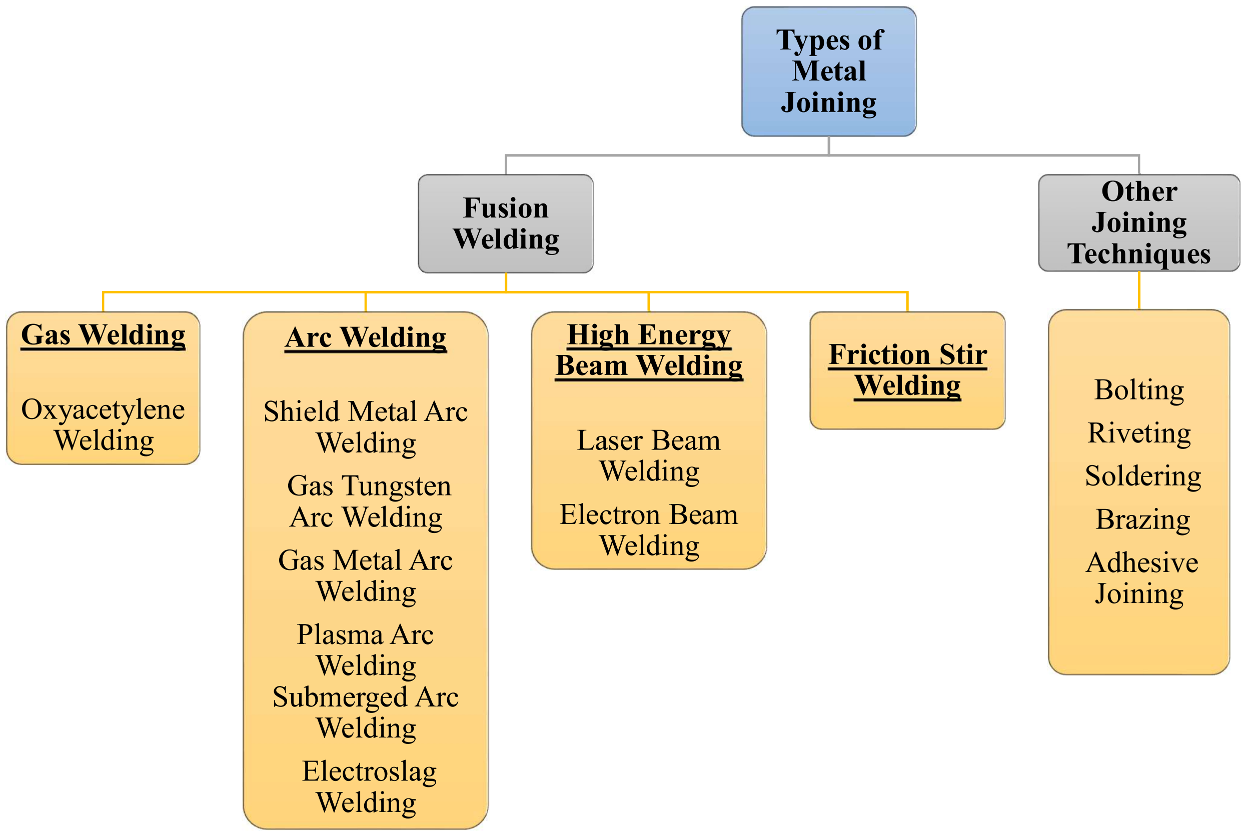


Figure 1.1 Different types of joining process methods

1.3 INTRODUCTION OF MICROWAVES

The frequency of microwaves having range of 0.3Hz to 300GHz [1] is being the part of electromagnetic spectrum. Microwaves are produced by the propagation of electrical and magnetic field perpendicular to each other. The vast range of frequency spreading of microwaves allows it to be used for number of purposes like in material processing medical purposes, communication system, industrial heating and food processing etc. [2]. The basic frequency on which local oven in India works in 2.45GHz, which is basically used for heating food stuffs. The heaters have been produced for material handling purposes which are utilized as a part of numerous mechanical applications and they chip away at higher frequencies running from 915MHz to 18GHz [3-4] as shown in Figure 1.2.

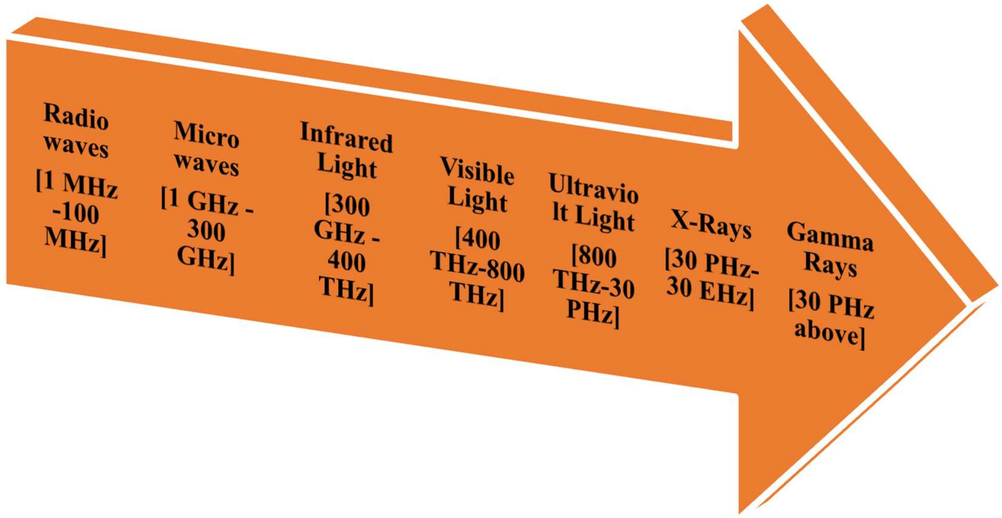


Figure 1.2 Different frequency ranges of electromagnetic spectrum

The heating power of microwaves was discovered [5] accidently during the Second World War in 1945. One day an American engineer called Percy Spenser, was working on a unit to produce high intensity microwaves which can be used in RADAR. During his experiment he observed that the chocolate bar in his pocket was started to melt. Then he placed corn near microwaves producing unit magnetron tube was rewarded with flurry of pops after this experiment Spenser got the idea that what microwave can do in the kitchen. After some years of hard work, the first domestic microwave oven came into the market and became very popular for domestic use because of its feature like higher heating rate, less energy consumption and less processing time and after few years it became the king of kitchen. More research is being carried out in processing of other materials using microwaves.

The field is still new. More and more research is being carried day by day to increase the applications of the microwave in different fields [7, 8]. The applications like processing of ceramics, metallic materials, vulcanization of rubber and chemical reactions etc. [9] are boon to the manufacturing sector. The primary characteristics of microwaves are shown in Figure 1.3. The volumetric heating coupling of microwaves at the atomic level gives advantage of heating all atoms at one time, which leads to faster heating. By using these unique characteristics; many researchers reported processing of ceramics with improved properties [10-12]. Selective heating can be done using microwaves which results in lower heat affected zone and lower defects. There is huge saving in processing time and power requirements [13-15] reported by various authors.

Figure 1.3 Characteristics of microwave material processing

1.3.1 Production of Microwaves

The real engineering in the microwave oven lies in creating the magnetron which generates high powered microwaves which is truly amazing revolutionary device. The magnetron is made of copper tube (Anode) with a Thorium plus tungsten (Cathode) rod running down the centre and the vacuum is maintained between cathode and anode, then the high voltage is applied across the two conductors which causes electron to move through the vacuum from the rod to the tube but they don't move in straight line. The tube is held in a magnetic field which applies force on the electron which makes them spiral outwards as the electron spin out land, then they passes through a small opening inside the tube. The electrons in the cavity behave like air in the bottle when we make node by blowing across the top. In the bottle air vibrate in and out of the bottle opening; creating sound which helps the electron spiralling pass cavities of the magnetron are bit like you blowing across the top of bottle they make the electron move in and out in a cavity in response produces microwaves. The general description of microwave producing device named as magnetron is given in Figure 1.4.

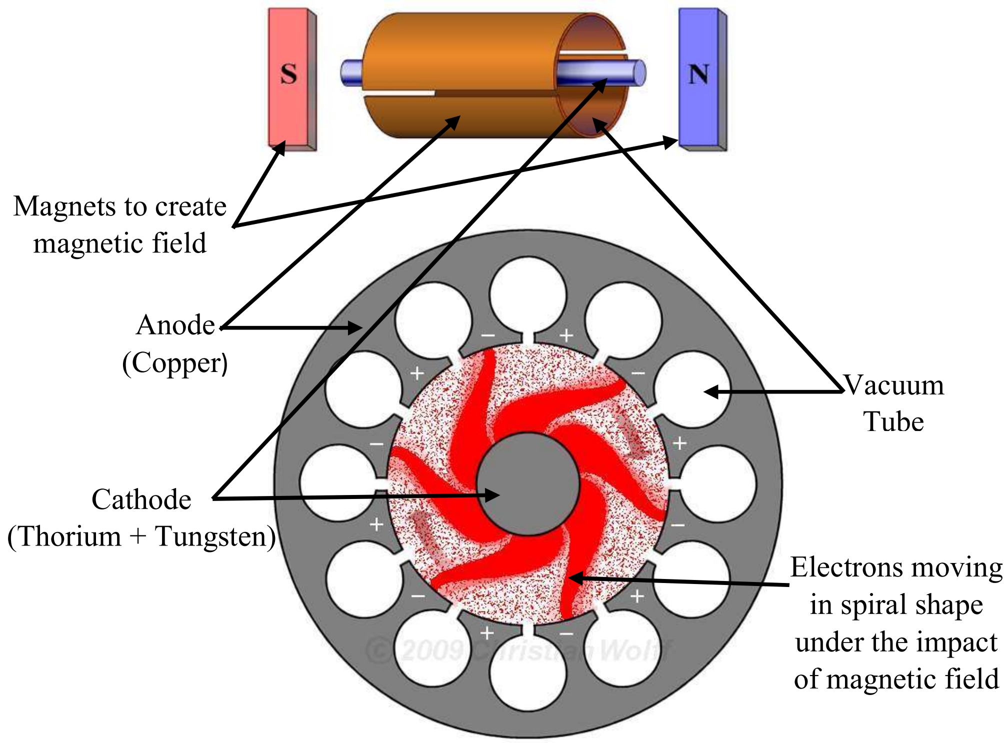


Figure 1.4 Description of Magnetron

1.3.2 Basic Principal of Microwave Heating

In microwave heat is transferred at molecular level. The transfer of energy resulting increase in motion of the molecule in a substance which increases temperature. This can be well understood by taking an example; as our food is filled with water, the molecules of water is positively charged and at one end negatively charged. Then they are exposed to electromagnetic waves that emanates from the tube by waves having electrical and magnetic field which changes direction rapidly for domestic oven direction of field changes 2.45 billion times a second water molecules try to alien with radiation of electrical and magnetic field the changing field rocks the water molecule back and forth rapidly and molecular friction from this creates heat as the motion disrupts hydrogen bond between neighbouring water molecules as a result produces heat. The general description of how a water molecule and electromagnetic field looks like is shown in Figure 1.5.

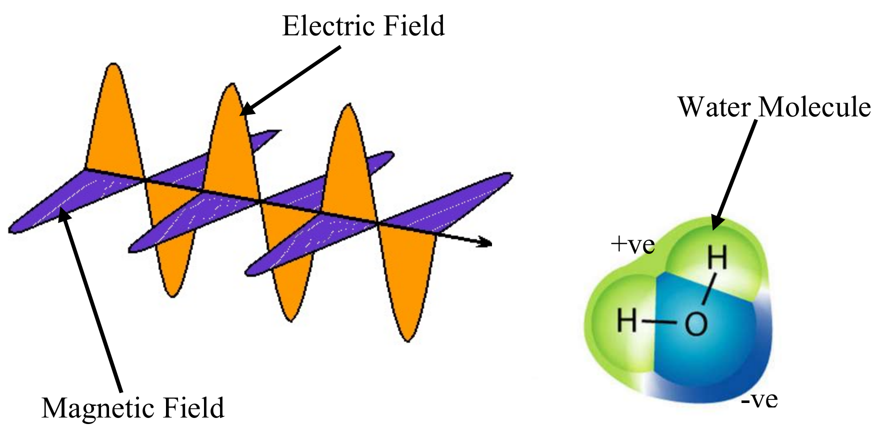


Figure 1.5 View of electromagnetic Field with water molecule

1.3.3 Microwaves Interaction with Different Materials

The heating of material is effectively and efficiently and also the physical properties of material has great importance. Different material behaves differently when they come in contact with microwave which is depicted in Figure 1.6.

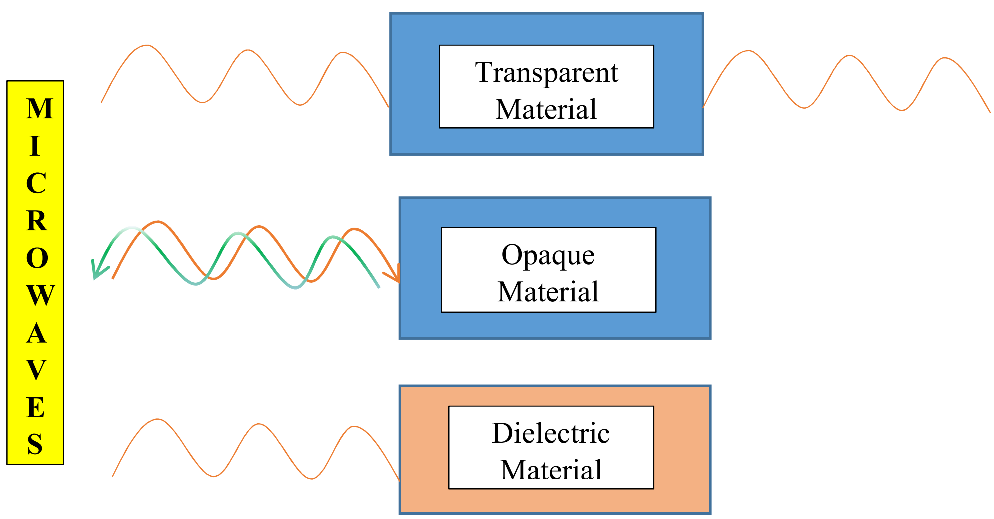


Figure 1.6 Interaction of Microwaves with different materials

Mainly the materials are classified into three different types:

a) Transparent material
b) Opaque material
c) Dielectric material

The transparent materials for example glass does not have the capacity to retain microwaves and it specifically permits them to go through without having any sort of loss and thus heating does not occur when microwaves come in contact with it. When the opaque material comes in contact with microwaves it don't permit microwaves to pass and absorb them; but it reflects them. The dielectric material has the tendency to absorb microwaves when it comes in contact with them as a result heating of the material takes place by continuous motion of dipole with changing electric and magnetic field.

1.3.4 Theory of Microwave Hybrid Heating

The processing of material which don't couple with microwave at room temperature was very challenging task, to process such types of material with microwaves phenomenon; hybrid heating was used. In this type of heating phenomenon; concepts of conventional heating as well as microwave heating is used as shown in Figure 1.7.

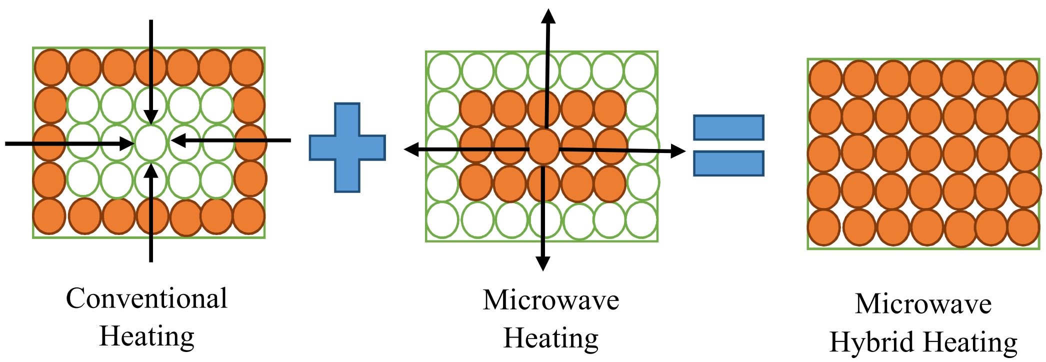


Figure 1.7 Phenomenon of Hybrid Heating

1.3.4.1 Conventional Heating: In conventional heating surface heating takes place first then slowly heat moves inwards from the outer most layer to the inner most layer or molecule of the material.

1.3.4.2 Microwave Heating: In microwave heating core molecule of the material starts heating first then the heat moves to the outer layer, but in actual all the molecules of the material come in contact with microwaves at same time. There is just a little difference which is negligible.

1.3.4.3 Microwave Hybrid Heating: Hybrid heating is used for the materials which cannot be processed directly with the help of microwaves so in this type of heating; suceptor material is used for heating as the skin depth is not appropriate and the material reflects the microwaves at room temperature. Due to this problem suceptor material is used which absorbs the microwaves initially and then heats the material conventionally at elevated temperature. During this process; skin depth of the particles increases as shown in Figure 1.8.

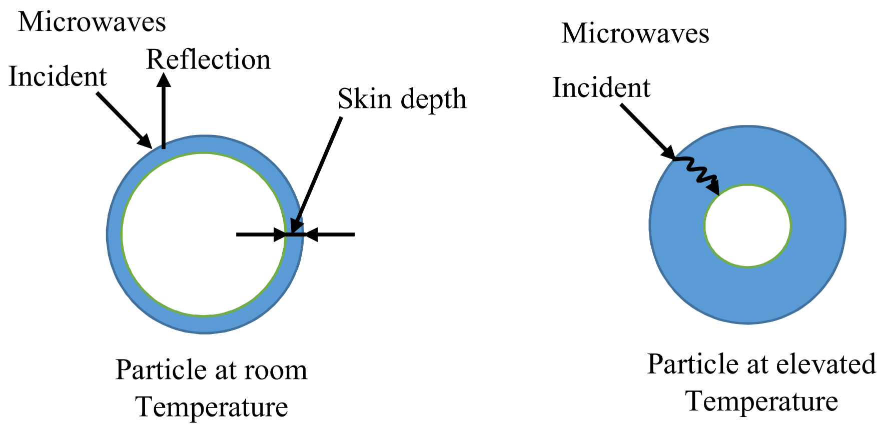


Figure 1.8 Concept of Skin Depth

As a result material starts absorbing microwaves which leads to the volumetric heating of the material. The skin depth of material can be found out by the Eq. (1.1) [16] given below.

$$\delta = \sqrt{\frac{\rho}{\pi f \mu_r \mu_\circ}} \tag{1.1}$$

δ =Skin depth (μm)

ρ =Resistivity (μΩ-cm)

f =Frequency of microwaves (GHz)

μ = Magnetic Permeability (H/m)

$\mu_\circ$ = Absolute Permeability (H/m)

μ_r =Relative Permeability

1.4 HISTORICAL DEVELOPMENTS IN MICROWAVE PROCESSING

Microwaves were initially developed for the telecommunication purpose, but with time it was discovered that they can also be used for the purpose of food processing. Also later in the period of 1950-65 wood curing paper drying and vulcanization was came into picture. This was just a time when the temperature was below 450°C, after the progression of time the scope of dealing with material went up to 1000°C which leads to the easy processing of ceramics, nitrides and glasses. These all changes with time are represented in the Figure 1.9.

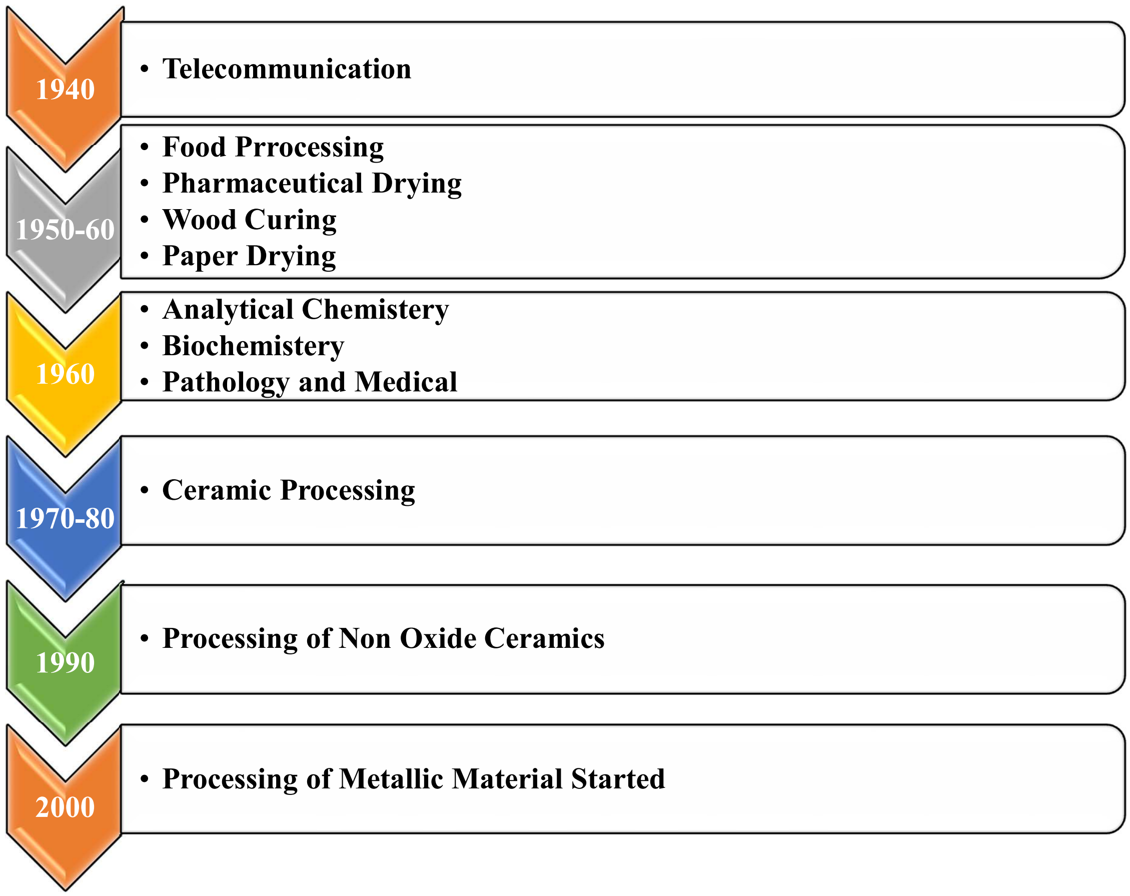


Figure 1.9 Historical Development in microwave material processing

After nineties; research was further carried out for higher temperature. In 1999 first research was published on sintering of metallic material [17]. After 2000 the research in the field of metallic material opened the broad area for processing of material having temperature up to 1500°C and more, it become easy to achieve such high temperature in simple domestic oven of 900W with frequency of 2.45GHz, which leads the research to the new heights for many useful applications like cladding, brazing, Melting, coating and joining; which results a fine contribution in the field of advance manufacturing. The order of different metallic application development is shown in Figure 1.10.

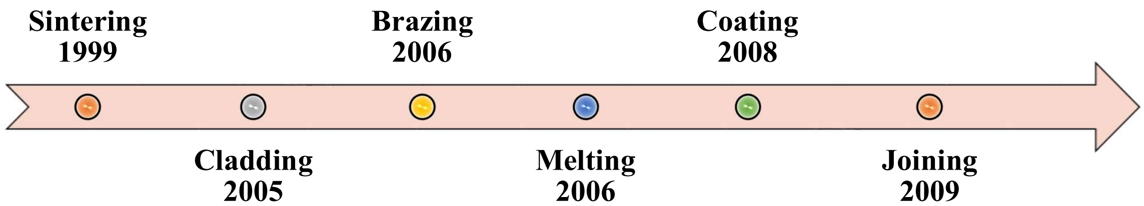


Figure 1.10 History of development of metallic material

1.5 MICROWAVES HEATING VS CONVENTIONAL HEATING

Table 1.1. Comparison of conventional heating and microwave heating

Sr. No.	Microwave assisted Heating	Conventional Heating
1.	In microwave heating molecules of the material comes in direct contact with electromagnetic field and due to changing field results in molecular vibration which produces heat.	In conventional heating surface is heated by radiant and convection heating then the heat transfer take place within the material by conduction or convection method.
2.	Vessel is kept in microwave cavities.	The vessel should be in direct contact with the heat source.
3.	Heating takes place by electromagnetic waves.	Heating is achieved by using both the thermal as well as electric source.
4.	The temperature of mixture can be raise above its boiling point.	The highest temperature (for an open Vessel) is depend upon the boiling point of the mixture.
5.	The heating procedure is highly Controlled as heating of material starts and stops immediately when power is switched on or off.	The conventional heating is difficult to control.

6.	Reduction in unwanted side reactions. (Reaction Quenching), so purity of the final product is high.	There may be unwanted side reactions, so final product is less pure.
7.	Less heat transfer to environment.	More heat transfer to environment.
8.	Processing rate of material by microwaves is very high, as processing of material which takes several hours or days can be processed with microwaves in minutes.	Processing rate is less.
9.	Average reaction time using microwave heating is 15 minutes.	Average reaction time using conventional heating is 6 hours.
10.	High efficiency of heating since selective heating can be done.	Less efficiency of heating since selective heating cannot be done.
11.	Microwave heating is also known as Green manufacturing process.	Conventional heating is not a green manufacturing process.
12.	High investment costs.	Low investment costs.

1.6 PROSPECTS OF MICROWAVE MATERIAL PROCESSING

Every technology when gets invented, it's not perfect from every aspect for which it's made or the purpose for it is used to be. There are lots of parameters and specifications, lots of challenges have to be controlled while dealing with newer technologies. These have lots of advantages and little bit limitations, because of which, this technology has been emerged in material processing.

1.6.1 Advantages of Microwave Material Processing

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 which leads 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 follow.

Rapid Heating: Microwaves achieve the article as same as the pace of light. Firstly, these voyages towards an article as waves and gets ingested after that protest creates heat. Rapid heating is conceivable in microwave heating on the grounds that in this sort of heating, the heat is produced by the article by its own particular with infiltration of microwaves by the item [18].

Volumetric Heating: Volumetric heating is seen amid microwave heating, so there is no temperature gradient as saw in traditional heating as the temperature differs from core to surface. In microwave material heating, the temperature stays same all through the material which prompts a superior quality item.

Variety of Production: There is an assortment of creation in microwave material handling, as a result of the scope of the temperature control and distinctive heating parameters. This prompts the microwave innovation in the field of metal preparing through microwaves and having assortment of production in various territories of generation like, cladding/Coating, sintering, joining of metallic materials.

Environment-Friendly: Microwave vitality is said to be totally environment well disposed of when contrasted with conventional vitality techniques for material preparing. Customary techniques for heating the metals as in heater, heating creates exhaust, risky gasses, smoke and so forth which prompts environment corruptions and this thing can be maintained a strategic distance from in microwave handling so its surroundings well disposed of starting here of perspective[18].

Energy And Cost Effective: Microwave heating prompts vitality sparing and in addition cost sparing. The vitality prerequisite to rise the temperature in heater for liquefying a material is high yet this necessity of vitality can be satisfied effortlessly and inconceivably with microwave vitality. Vitality sparing specifically or in a roundabout way prompts cost adequacy, which likewise tends to spare the time in view of its high productivity.

Improved Mechanical Properties: The run of the mill necessities for the traditional sintering process incorporates higher heating rates, high temperature and support of this temperature for a certain timeframe. Be that as it may, to keep up the uniform temperature conveyances, the examples are typically kept for higher dousing periods which prompt the over the top grain developments and abandons, for example, porosity and breaks. These issues can be removed through microwave preparing which gives relatively homogeneous microstructures with better grain development and improved properties in expansion to lower handling time. These acquired great attributes are because of the higher heating rates, uniform heating of materials (volumetric heating), and lessened the time for accomplishing higher temperatures. The examination of microstructures is represented by Figure 1.11[19].

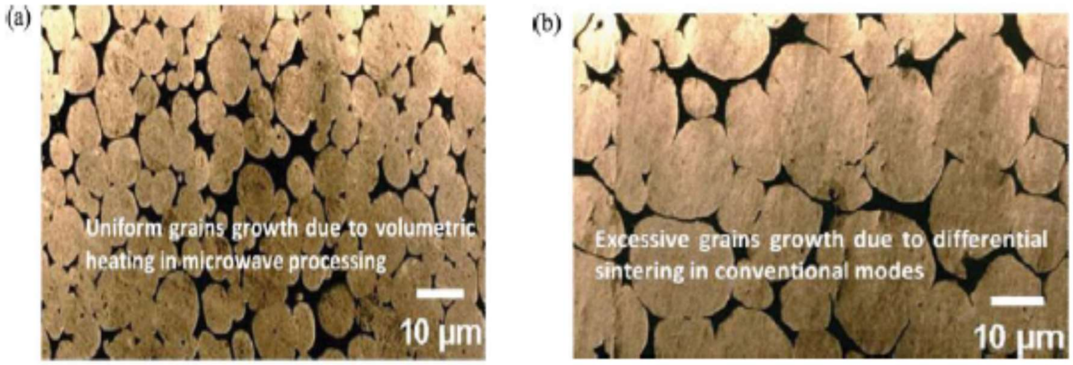


Figure 1.11. Effect of (a) microwave processing and (b) conventional processing, on microstructure of processed material [19].

Improved Densification Parameter: The thickness of powder compaction chooses the properties of the completed product. Higher the thickness of prepared material, lower will be the deformities mostly as far as porosity. Because of uniform heating and volumetric heating of an item, the densities acquired are higher on the off chance that of microwave sintered or prepared parts. The compacts sinter ability considers the effect of at first squeezed density values, which can be connected as far as standardized, dimensionless densification parameter, or consider and is represented by Eq.(1.2) [20].

$$\text{Densification factor} = \frac{\text{Sintered Density} - \text{Gre} \quad \text{Density}}{\text{Theoritical Density} - \text{Green Density}} \quad (1.2)$$

The other advantages may be explored as:

- Up to 70% less energy usage than conventional energy.
- Greatly improved throughputs resulting from deep penetration of microwave energy into particular materials, giving rapid heating throughout the bulk of the product being heated.
- Reduced environmental pollution due to increased energy efficiency and clean transfer of energy to product being heated.
- Removal of gases from porous materials without cracking.
- Improvement in product quality and yield.
- Synthesis of new materials and composites.
- Economic advantage by time and energy saving.

1.6.2 Limitations of Microwave Material Processing

As the coin has two flanks, having head and tails, likewise every procedure has a few upsides and downsides so following are the limitations:

a) The unknown temperature inside the microwave cavity.

b) Proper sculptor material must be used otherwise chances of fire hazard.

c) Due to high heat generation it requires high careful handling.

CHAPTER 2 LITERATURE SURVEY

2.1 LITERATURE REVIEW

This chapter mainly deals with the up to date happening in the field of microwave joining also the other important works performed in the field of microwave dealing with metals and also gives the information of the work which has been not attempted yet. This chapter also gives the overview of research objectives to full fill the purpose of problem formulation.

Srinath et al. [21] investigated joint of stainless steel (SS-316) and mild steel (MS) can be formed by using microwave hybrid heating method. He applied the nickel powder paste mixed with epoxy resin in between the two substrate with exposer time 450s in homemade microwave oven at 2.45GHz and 900W. The formed joint was characterised and it shows Micro hardness to be 133Hv, porosity was very less just .58%, measured ultimate tensile strength to be 346.6 MPa with elongation percentage of 13.5% also FESEM and XRD was done to study the microstructure of joint with formation of various complex carbides.

Srinath et al. [22] worked on a highly conductive material like copper. Earlier it was difficult to join copper but by using microwave hybrid heating method it became simple to join copper. Domestic microwave oven 2.45GHz and 900W was used paste of copper powder mixed with epoxy resin was introduced between the joints of .5mm thickness and charcoal is used as a susceptor material which interacts with microwaves to give heat to the work piece for a time 900s to form a joint. The formed joint was characterised and it shows Micro hardness to be 78Hv, porosity was very less just 1.92%, measured ultimate tensile strength to be 164.4MPawith elongation percentage of 29.2% also FESEM and XRD was done to study the microstructure of joint with formation of various carbides.

Bansal et al. [23] investigated the joining of stainless steel-316 microwave hybrid heating method. Domestic microwave oven 2.45GHz and 900W was used paste of stainless steel powder mixed with epoxy resin of 1mm was introduced between the joints and SiC is used as a susceptor material which interacts with microwaves to give heat to the work piece for a time 720s to form a joint. The characterisation of joint shows Micro hardness to be 275Hv, porosity was very less just 0.94%, measured ultimate tensile strength to be 425 MPa with elongation percentage of 9.44% also the concept of skin depth was properly explained by the researcher also FESEM and XRD was done to study the microstructure of joint with formation of various carbides helps in making the joint stronger.

Badiger et al. [24] investigated the joint formation by introducing the paste of 1mm nickel based powder between the two work pieces of Inconel-625 alloy by using domestic microwave oven 2.45GHz and 900W was used experiment time 1260s. The characterisation of joint shows tensile strength of 326 MPa with elongation 9.04% which is 35% more than the base metal and micro hardness was absorbed to be 360Hv also FESEM and XRD was done to study the microstructure of joint which shows formation of various rich cells of nickel and carbides in the form of chains surrounding it.

Zafar et al. [25] investigated formation of clad on AISI 304 stainless steel of WC-12Co cermet using 1.4KW industrial multimode microwave developed clad was characterised by FESEM, XRD, micro hardness and porosity. Result shows that there is excellent metallurgical bonding of clad material with base metal the microstructure shows skeleton like structure which indicates formation of various complex and stable carbides which indicates high hardness of the developed clad 1135HV porosity was found to be .98%. Research was also carried out on the inside temperature measurement of microwave oven using the pyrometer at small interval of 30s.

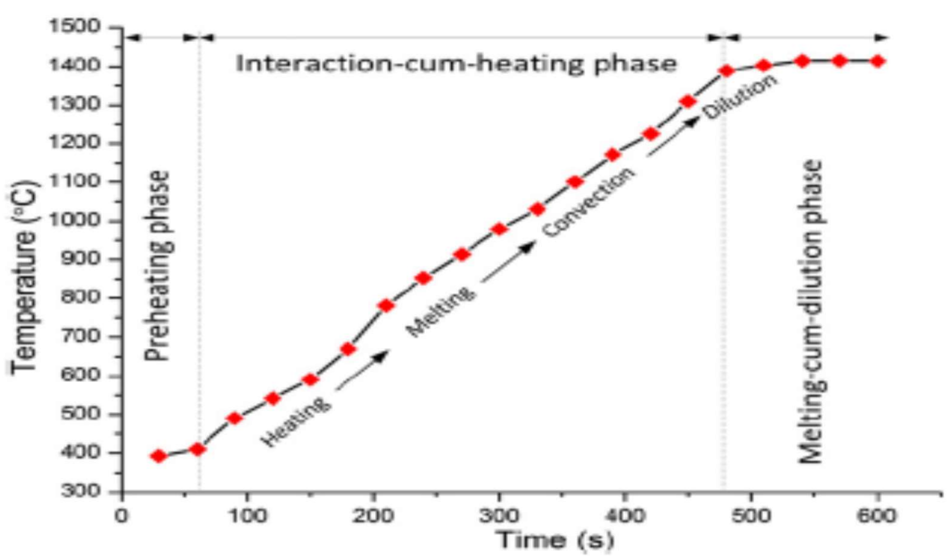


Figure 2.1 Time temperature plot during WC-12Co microwave cladding

Gupta and Sharma [26] investigated the clad formation using the stainless steel (SS-316) as a substrate material and nickel based powder as a clad material. The clad was formed by the new developed technique of hybrid heating using the domestic microwave oven of 2.45GHz and 900W with experiment time 360s and clad thickness 1mm then the formed clad was characterised which shows very less porosity (1.09%) and hardness to be 304Hv. FESEM and XRD was also done which shows the formation of various carbides and cell rich of nickel give strength to the clad and the concept of skin depth was also explained thoroughly.

Gupta and Sharma [27] investigated the clad formation using the stainless steel (SS-316) as a substrate material and WC10Co2Ni powder as a clad material. The clad was formed by the newly developed technique of hybrid heating using the domestic microwave oven of 2.45GHz and 900W with experiment time 360s and clad thickness 2mm then the formed clad was characterised which shows very less porosity (.89%) and hardness to be 1064Hv also FESEM and XRD was done to study the microstructure of joint which shows formation various carbides in the form of skeleton like structure gives higher hardness to clad. The tribological study of the joint was also carried out to study the wear characteristics of the formed clad using the pin on disc sliding method.

Gupta and Sharma [28] worked on the formation of clad WC10Co2Ni on the substrate material austenitic stainless steel. The cladding was done by the newly developed technique of hybrid heating using the domestic microwave oven of 2.45GHz and 900W with experiment time 120s. The formed clad was characterised by FESEM and XRD which gives the knowledge of formation various carbides in form of skeleton like structure that gives the evidence of higher hardness and proper metallurgical bonding with the substrate.

Sharma and Gupta [29] worked on surface building and its impacts on the microstructure and flexural strength of cladding created through microwave handling. In this examination, metal-artistic composite cladding was produced on the substrate. As a substrate material, austenitic steel was utilized and Ni-based EWAC having 20% WC10Co2Ni as a reinforcing operator was utilized for composite cladding. Nickel was picked on account of its strength property and consumption resistance at raised temperatures and WC builds the hardness and also adds to higher wear resistance. Subsequently, a cladding delivered with this structure should have ductile and compressive anxiety bearing ability. Through microwave preparing, a deformity free cladding with 0.89 porosity was made. This strengthening creation on a substrate expanded the hardness of SS from 200HV to 416±20HV and flexural quality of clads 629±8N was accomplished.

Prasad and Gupta [30] worked on a microwave processed cladding of nickel-based lanthanum oxide composite powder molecule having molecule size of 40 µm on mellow steel. The substrates were cut into normal measurements of 10mm × 10mm × 5mm. At that point, they were cleaned with emery paper to get the simulated surface. In this study, mellow steel was utilized as substrate and composite powder was put on it. The specimen was set in microwave oven with 2.45 GHz frequency, 900 W power and uncovered for 240 sec. The roughly 500 µm clad thickness has been produced on gentle steel. The vicker's smaller scale hardness of created clad specimen was acquired around 319 Hv and clads demonstrated a superior wear resistance and mechanical properties.

Gupta and Wong [31] utilized microwave hybrid heating to sinter the metallic materials and enhanced the general mechanical properties of the fabricated item. Al and Mg and lead-free solder were selected to perform sintering procedure. The sintering procedure was done by utilizing domestic microwave oven with 2.45 GHz frequency and 900 W power level. SiC was utilized as a susceptor for empowering coupling amongst microwave and the material. The sintering was brought out through microwave hybrid heating (MHH) in light of the fact that microwave heating has an element to heat the material from inside to outside and then again susceptor (SiC) bolstered the sintering from outwards to inwards. The sintering of every one of the three chose materials was effectively completed utilizing microwave heating. A superior mix of tensile properties was acquired in microwave sintering as an aftereffect of the decreased level of high-temperature thermal exposure.

Clark and Sutton [32] worked on the capacity of microwaves to process metallic materials. The material being handled through microwaves, associates for a brief span of time with the microwaves instead of radiant heat contact in customary procedures. In this innovation, the heating is volumetric and particular or quick heating can be accomplished in light of the fact that the heat is produced by the material itself. The rate of heating is corresponding to the power. These elements prompt consistency and assortment of production. The equipment requires less floor zone and quicker generation all through the procedure which compares to the legitimate usage of the facilities. Authors compressed the benefits of microwave handling over the conventional ones and diverse regions in which this innovation ought to be investigated. The microwaves at the nuclear level can be transmitted, absorbed or reflected by materials and these properties may shift from material to material. Each of the three states (liquid, gasses and solids) can communicate with microwaves and can be heated. In contrast with traditional heating procedures, microwave handled materials ordinarily shows the higher temperature at the middle than on the outside surface since heat ventures out from inside to outside. Different parts of the microwave handling have been additionally highlighted i.e. heating and drying rates, electric field dispersions, temperature profiles, power ingestion and hybrid heating.

Bansal et al. [33] completed the mechanical and metallurgical portrayal of MS-MS joint created through microwave heating utilizing nickel powder as interfacing material. Joining of the bulk metallic materials is the most difficult errand with the assistance of microwave vitality, due to the poor coupling of microwaves with the metallic materials and microwaves are by and large reflected by the metals. In this study, microwave hybrid heating (MHH) procedure was utilized for joining. A nickel-based powder of particle size 40 μm was utilized as the interface layer for the creation of the joint. The bulk plates of MS were put in such a way, to the point that a butt arrangement and a gap of around 0.5 mm was kept up between both the specimens. The gap was loaded with the slurry, having the blend of EWAC (Ni-base powder) and epoxy. As a result of the microwaves reflection marvel by metallic materials, it was extremely hard to manage the microwaves at room temperature. To make it conceivable that microwaves get consumed into the material, an encasing veil of fine charcoal was conformed to the joint and thusly, metal was not specifically presented to the microwaves.

Bansal et al. [34] investigated joint of austenitic stainless steel (SS-316L) and Inconel 718 can be formed by using microwave hybrid heating method. He applied the Inconel powder paste mixed with epoxy resin in between the two substrate with exposer time 600s in homemade microwave oven at 2.45GHz and 900W. The formed joint was characterised and it shows Micro hardness to be 230Hv, porosity was very less just .94%, measured ultimate tensile strength to be 517.5 MPa with elongation percentage of 18.8% also FESEM and XRD was done to study the microstructure of joint with formation of various complex carbides.

Chandrasekaran et al. [35] did an experimentation to melt the metals through microwave processing. Microwave heating is a bit more mindful on account of its significant points of interest, for example, high heating rates because of hybrid heating idea, which specifically compares to improve the handling time, less utilization of power and less environmental hazards. The microwaves are reflected by the metals, in light of the fact that their skin depth is of a couple of microns Thus Microwave melting of low-temperature materials, for example, lead, tin, aluminium and copper was done with the guide of susceptors (SiC). Aluminium and copper samples were melted in the climate of argon/nitrogen (idle gas) to minimize the danger of oxidation. On contrasting it and the melting through the traditional heater, microwave melting was observed to be twice quicker.

2.2 GAPS IN LITERATURE REVIEW

The thorough literature study on microwave material processing projected that microwaves are being used for high temperature applications in manufacturing sector the microwave dealing with metals and applications like cladding and joining enhanced the research scope of microwave processing of material. Following are the gaps we got from the literature survey to contribute in the field of microwave joining.

1) Literature shows no work has been reported on the joining of SS-430 using microwaves.
2) As the microwave joining field is still new so very less work has been reported on the microstructural analysis of the formed joint.
3) Literature shows no work has been reported on mechanical characterization of SS-430 developed joints through microwave energy.

2.3 OBJECTIVES OF PRESENT WORK

The complete literature survey has depicted certain gaps so to fill these gaps objectives has been decided as follow.

1) To join SS-430 by introducing nickel powder paste between the joints using the domestic microwave oven of 900W with frequency 2.45GHz.
2) The joint will be characterized using tests as given below.
    - SEM (Scanning Electron Microscopy)
    - EDS (Energy Dispersive X-ray spectroscopy)
    - XRD (X-ray Diffraction)
    - Vickers Micro hardness
    - Tensile Testing

CHAPTER 3 METHODOLOGY

3.1 METHODOLOGY

On the basis of literature survey; foundation of research was setup in the form of gaps then further gaps were explored to get the definite objectives of the research which will ultimately lead to formation of complete methodology to meet the desired objectives.
The methodology to complete the research objectives is divided into three stages as follows:

Initial Stage: To carry out trial experiments, two work pieces of stainless steel 430 were taken then slurry of nickel powder (EWAC) was introduced between the two work pieces having composition of 25% epoxy resin and 75% EWAC powder. Then a graphite sheet (separator) was placed on prepared joint to avoid the mixing of charcoal with slurry; and the whole work piece was covered with charcoal powder. The charcoal is used to avoid the direct contact of microwaves with metal as metal reflect microwaves at room temperature. The prepared setup was placed inside microwave oven on a refractory brick. After applying so many hit and trials, the suitable optimum time of 12min was found at which the metallurgical bond will be formed between the two work pieces.

Intermediate Stage: In this stage; the samples were prepared using emery papers of grade 80,150,400,800,1000,1500,2000 used for the purpose of polishing of the samples so that proper characterization of the joint can be done by different techniques also etching was done using ($HCl+C_2H_6O$). The techniques used for the characterization are as follow.

- SEM (Scanning Electron Microscopy)
- EDS (Energy Dispersive X-ray spectroscopy)
- XRD (X-ray Diffraction)
- Vickers Micro hardness
- Tensile Testing

For tensile testing samples were prepared by using the ASTM standard E8/E8 M-09 having gauge length of 18mm and width 3mm.

Final Stage: In this stage; the results obtained from the intermediate stage were analysed to make the final conclusions about the quality of joint formed by microwave heating. The whole methodology is shown in Figure 3.1.

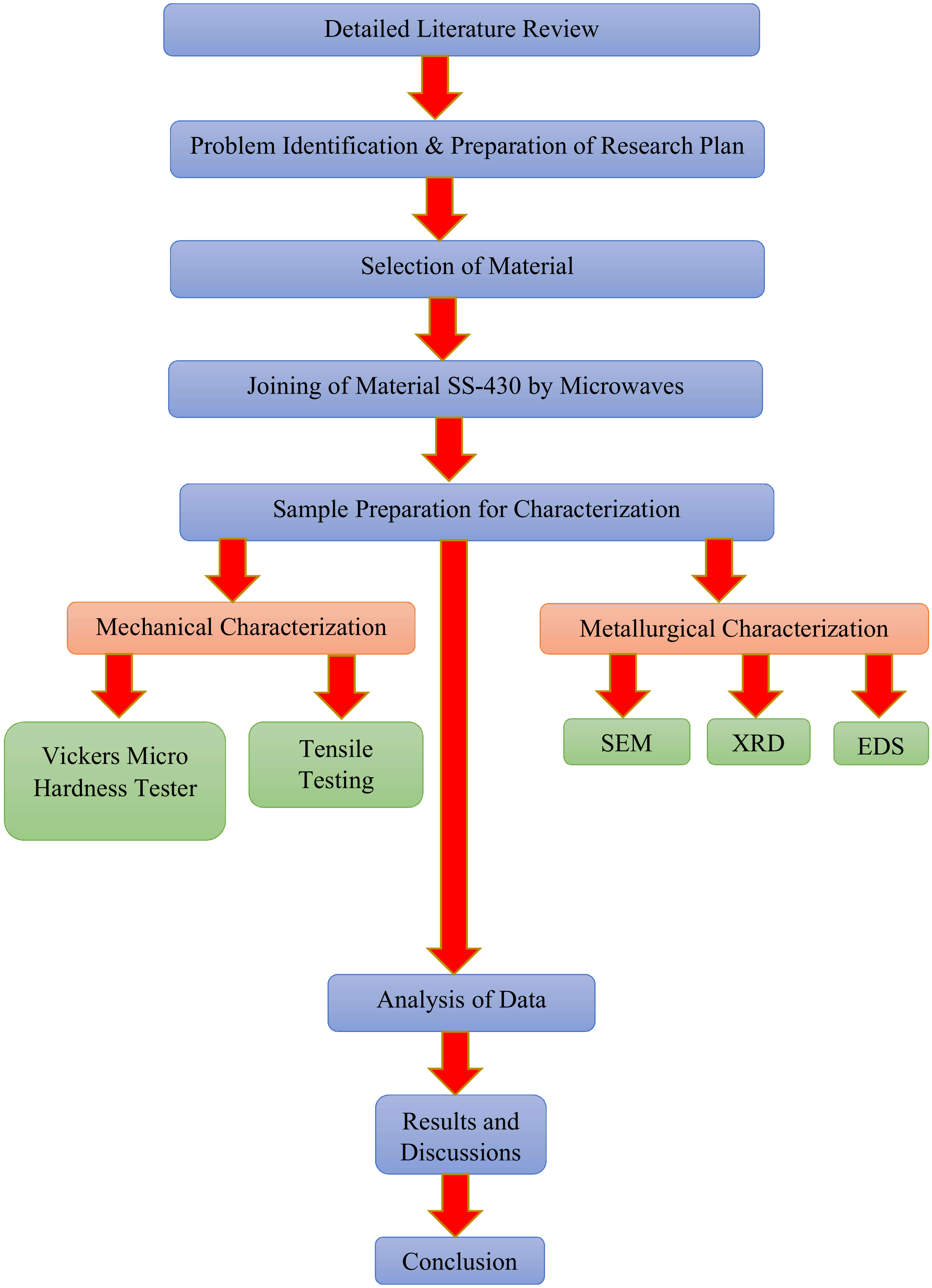


Figure 3.1 Flow Chart Showing brief Methodology

3.2 MATERIAL SELECTION

Based on the literature review SS-430 was selected as a work material for the purpose of experimentation. It has certain inherent properties like good oxidation resistance capacity at elevated temperature, good formability and corrosion resistance; which makes it suitable for many applications like in kitchen utensils, in washing machining parts, in parts of furnace, in heat exchangers and also in the petroleum refining equipment etc. Table 3.1 shows the chemical composition of the work material. It has chromium which improves the wear resistance capacity, Nickel increases the densification, carbon imparts brittleness, manganese gives strength etc.

Table 3.1 Chemical composition of SS-430

Material	Elements Weight Percentage							
	C	**Mn**	**P**	**S**	**Si**	**Cr**	**Ni**	**Fe**
SS-430	0.12	1.00	.040	.030	1.00	16-18	.75	79.06

3.3 DEVELOPMENT OF JOINTS USING MICROWAVE ENERGY

Microwave hybrid heating technique was used to join the materials of SS-430. Table 3.2 shows the process parameters which were used during the experiment.

Table 3.2 Process parameters for joining of SS-430

Process Parameter	Descriptions
Microwave applicator	Domestic multimode microwave (Made: LG, Model: Charcoal)
Working frequency and maximum power rating	2.45 GHz and 900 watts
Exposure time	12min
Work-piece material	SS-430
Susceptor material	Fine graded charcoal powder
Separator material	99.9 % pure thin graphite sheet
Interfacial Powder	Nickel powder (EWAC)

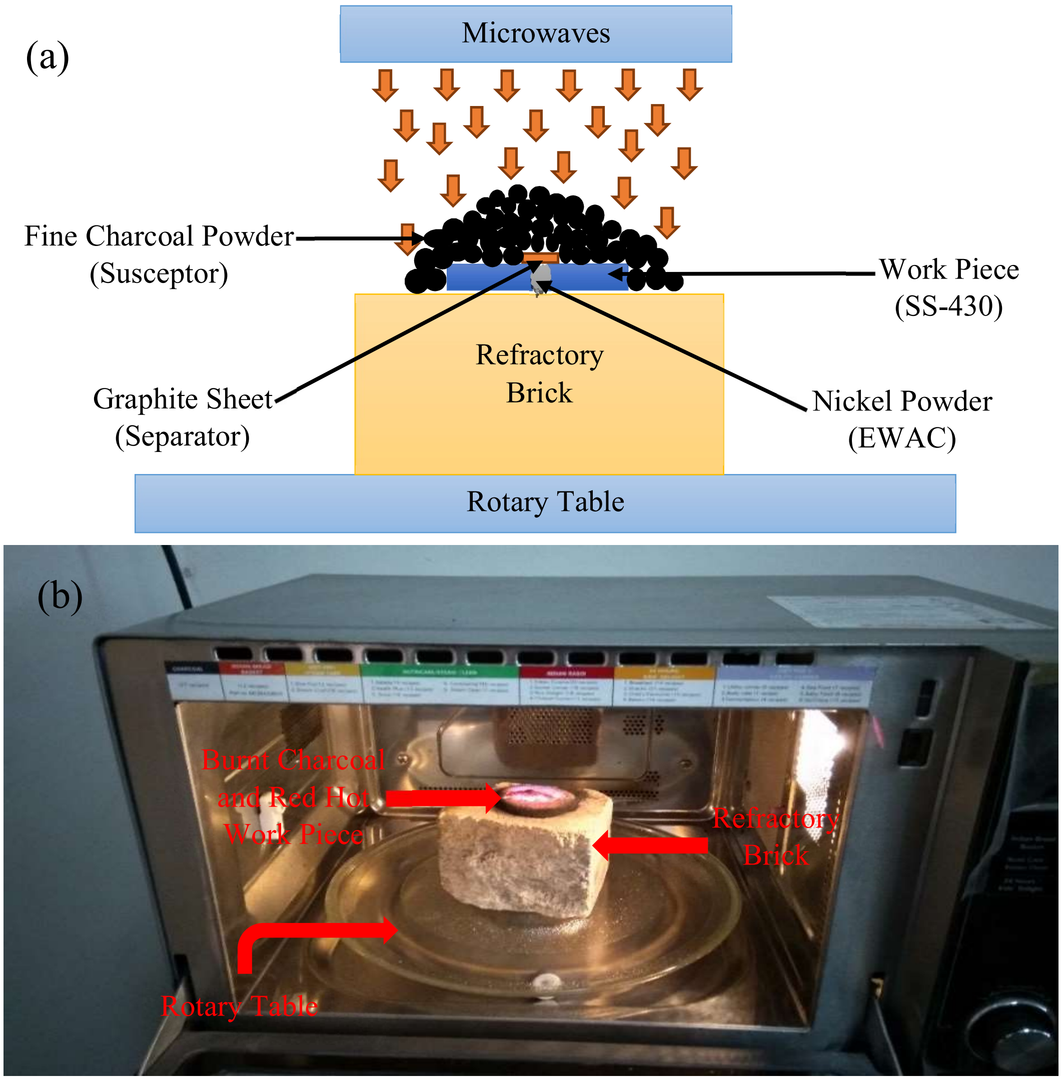


Figure 3.2 Experimental setup (a) Diagrammatic setup (b) Actual setup

To carry out the experiment, nickel power was used in form of slurry made by mixing with epoxy resin. The combination of both was applied between the work materials. At start at room temperature where the skin depth is lower, the nickel is not able to couple with the microwaves; as a result it reflects all the microwaves.

The skin depth of material can be found out by the Eq. (3.1) given below.

$$\boldsymbol{\delta} = \sqrt{\frac{\boldsymbol{\rho}}{\boldsymbol{\pi f \mu_r \mu_\circ}}} \tag{3.1}$$

δ =Skin depth (μm)

ρ =Resistivity ($\mu\Omega$-cm)

f =Frequency of microwaves (GHz)

μ = Magnetic Permeability (H/m)

$\mu_{\circ}$ = Absolute Permeability (H/m)

μ_r =Relative Permeability

Based on the equation 3.1; skin depth was calculated. The calculated value for nickel (considering ρ =8.707 $\mu\Omega$-cm, f =2.45 GHz, $\mu_{\circ}$=4p×10-7 H/m and μ_r=600) was approximated 0.12 μm. From the calculated value it is clear that, it is less than the particle size of the nickel powder which was used during the experiment. It proves that nickel particles directly are not able to process with the microwave radiation which results in reflection of the microwaves. To remove such obstacles related to the skin depth; microwave hybrid heating was used in which firstly the nickel powder was heated by traditional method by using the suspector material, which will accelerate the temperature of the powder and powder will start to form the bonds with the microwave. Once the process is completed of melting followed by fusing; a solid layer at the joint will be formed which will restrict the reflection of the microwaves.

3.4 CHARACTERIZATION TECHNIQUES

Characterization is mainly used to saw the microstructure, properties and also the correlation between the properties as well as the structure of the work material. Metallographic analysis is a powerful tool (Metallurgical microscope, EDX, SEM, XRD) to investigate the microstructural examination and primary characteristics of the work piece. Some techniques are also used to analyse the thermal as well as mechanical testing of the work piece.

3.4.1 Disc Polisher (Polishing machine)

After cross-sectional cutting the samples, they were subjected to disc polisher to polish them up to the desired finish. Disc polisher is a polishing machine having two rotating wheels on which emery papers are mounted having a jet for water supply and speed of rotation can be control by a knob. Samples were firstly polished by low grades (coarse) emery papers such as 80, 150, 400, and 800 under a jet of flowing water to remove the scratches which may come on the surface because of abrasive particles coming out after degradation. After polishing on coarse grades, they were polished on fine grades emery papers such as 1000, 1500 and 2000 to obtain a super finished surface. The soft velvet cloth was mounted on the second wheel and then the samples were polished on it by applying diamond paste of 2µm particle size. Disc polisher is shown below in Figure 3.3.

Figure 3.3 Disc polisher (Courtesy: CNC Lab, Thapar University)

3.4.2 Scanning Electron Microscopy (SEM)

The examination of microstructures and chemical compositions are by and large completed by utilizing Scanning electron microscope and energy dispersive X-ray spectroscopy. The scanning electron microscopy (SEM) is utilized to check the surface region and is utilized to decide the microstructures of specimens at higher amplifications which utilizes an electron beam to examine the specimen surface. The electrons collaborate with the atoms of sample and produce flags that contain data about the microstructural investigation of produced pictures. It can create high-resolution pictures of the sample surface, uncovering points of interest short of what one nanometer in size. The scanning electron microscope used for this work is a product of Oxford Instruments (Model No.: JEOL JSM-6510LV) which is available at **SAI Labs, Thapar University, Patiala** is shown in Figure 3.4. It is a high-performance and low vacuum SEM for quick characterization and imaging of fine structures having amplification from 5X to 300,000X. For the most part, EDS is likewise appended with SEM, in light of the fact that it tells about the elemental composition peaks for those microstructural pictures that are captured by SEM. EDS gives the quantitative examination of its basic compositional % age having a depth up to 1-2 microns and information is spoken to as crests and dispersion of reinforcement at different focuses.

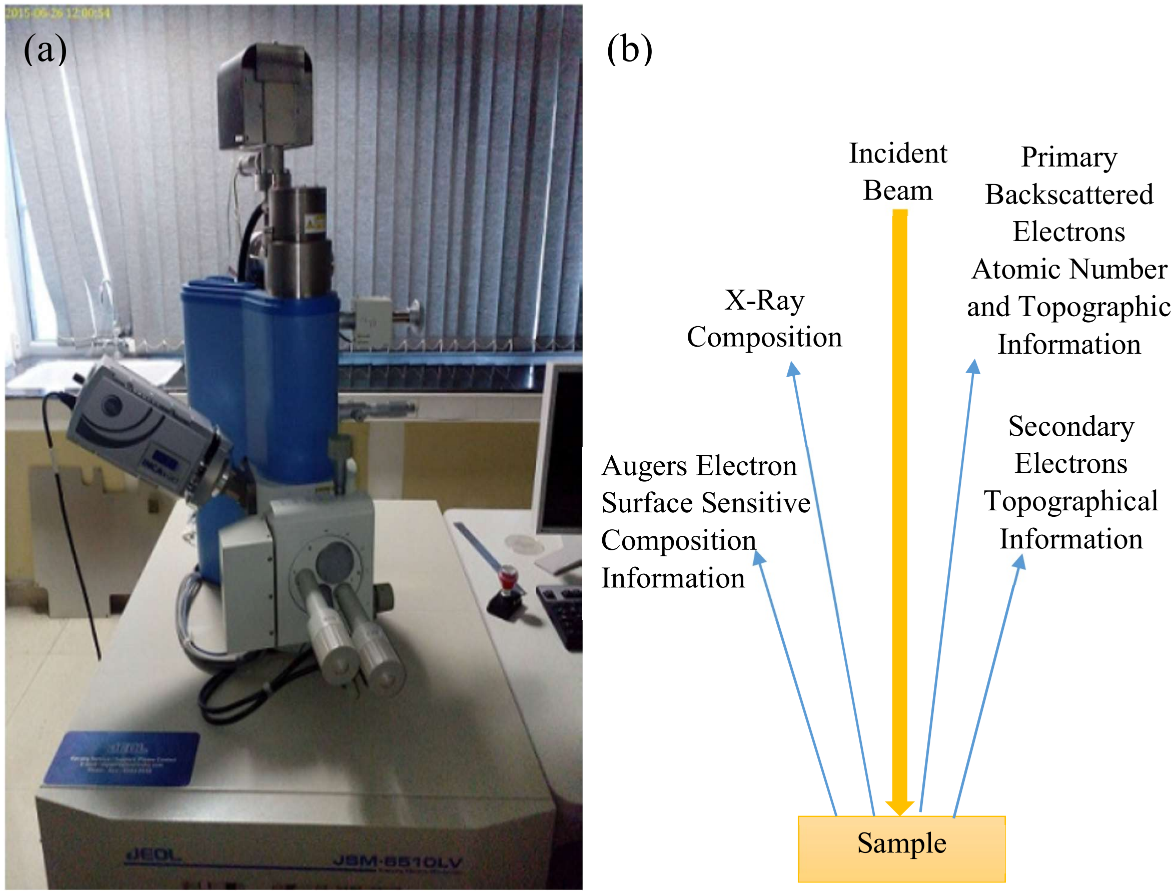


Figure 3.4 Scanning Electron Microscope (SEM) (Courtesy: SAI Labs, Thapar University)

3.4.3 X-Ray Diffraction (XRD)

X-ray diffraction procedure is a standout amongst the most capable apparatuses for subjective and quantitative investigation of materials. The investigation of this system gives the average bulk composition of materials. The fundamental guideline behind the XRD is that the monochromatic radiation is delivered by the X-rays, which are produced through a cathode tube and focused on the specimens. In the present work, XRD is utilized to decide the elemental composition of the fabricated joint, delivered through microwave processing. X-ray Diffract meter used for this work is shown in below Figure 3.5. 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 2θ 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. 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.

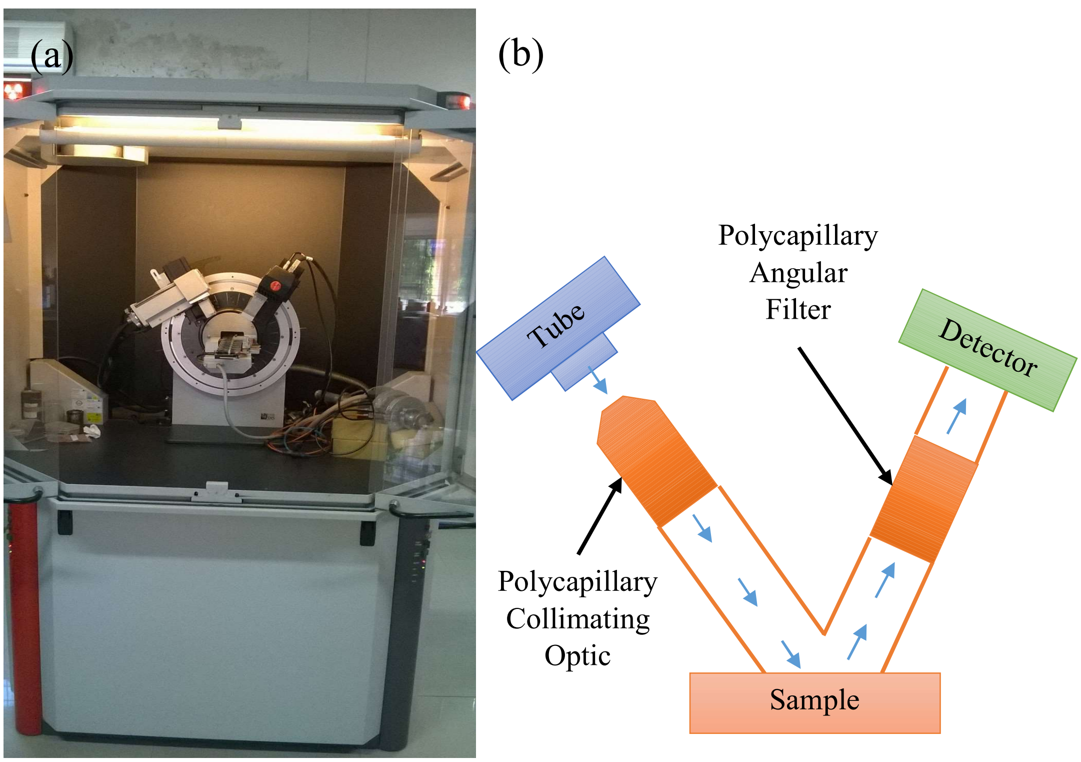


Figure 3.5 X-ray Diffract meter (XRD) (Courtesy: IIT, Roorkee)

3.4.4 Vickers Micro Hardness Tester

The micro-hardness of the formed joint was assessed utilizing a Vicker's micro-hardness tester. The specimens after cleaning were set up for assessing the micro-hardness. In Vicker's micro-hardness test strategy, firstly with the help of the camera attached to it, the sample was fixed at the desired position. After positioning, the required load was applied through the lever. Then the machine was set for the required dwell time and loading of indenter starts. After the completion of the dwell time, the indenter unloads automatically and with the help of the camera, indentation marks were visible on the surface. By focusing the camera on the indentation mark, indentation was measured by measuring the two diagonals that were marked on the sharped ends. It must be keep in mind that whatever load was applied by the lever, the same load should also be selected while measuring the diagonal. The estimated value was based on the selected load, which may differ for different loads. After measuring the indentation, the hardness value was ascertained. The same process was repeated for 3 or 4 times to check the deviation and then an average value of all was considered as the hardness value for that particular sample. The indenter utilized for indentation can be utilized for all materials regardless of the hardness of materials. The load applied for calculating hardness of the samples in present work was 500g and for 20 seconds dwell period. The Complete setup of Vicker's Micro-hardness tester used for this work is shown in below Figure 3.6.

Figure 3.6 Vickers Micro-hardness Tester

3.4.5 Tensile Testing

Tensile testing is mainly done to test the strength of the material in which a suitable load is applied on the work material which gives result in form of ultimate strength after which the failure of the material occurs. During the testing; elongation of the work material occurs due to which certain reduction in the area of the work material occurs. These results may be utilized to obtain the material properties like modulus of elasticity, ultimate or yield strength of the work material, Poisson ratio and also the characteristics of the strain hardening. Uniaxial and biaxial tensile testing are carried out for isotropic and anisotropic materials respectively. ASTM standards are used to carry out the tensile testing. E8/E8 M-09 ASTM standard was used for the present study. Dimension of the specimen which was used during the experiment is shown in Figure 3.7(a) and Figure 3.7(b) also shows the equipment used during tensile testing.

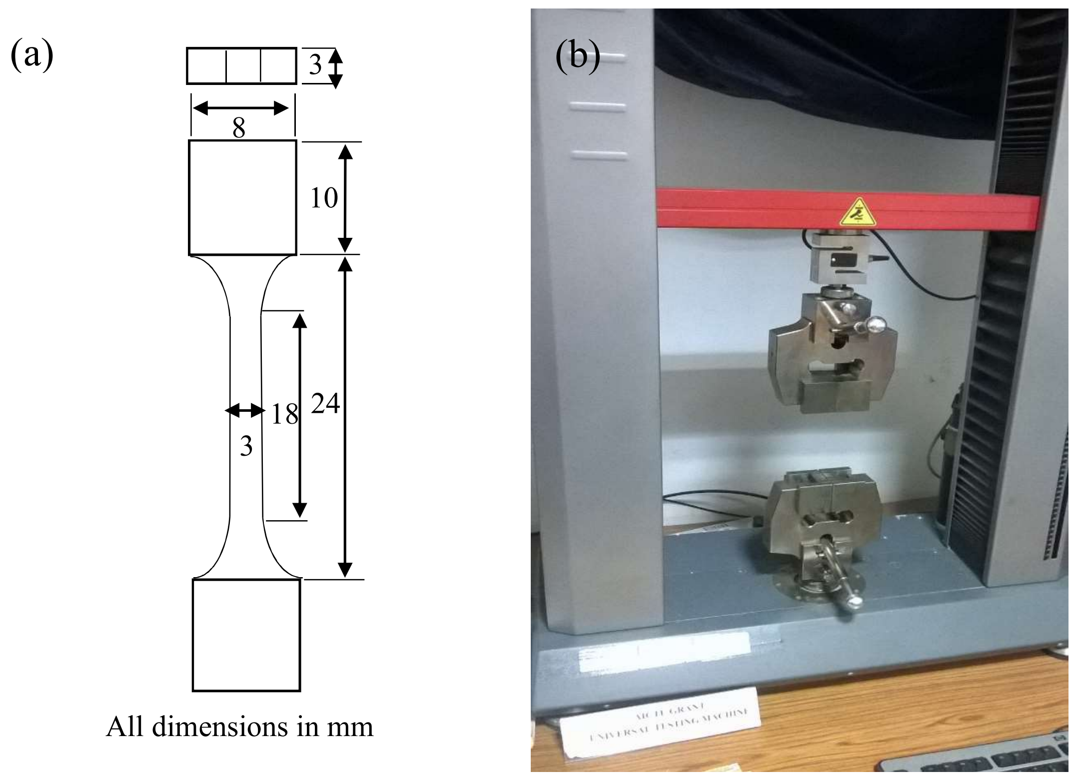


Figure 3.7 (a) Standard tensile test specimen and (b) Zwick-Roell Z010 tensile testing machine (Photo courtesy: Chemical department, TU, Patiala)

CHAPTER 4 RESULTS AND DISCUSSIONS

This chapter mainly deals with the results and discussions of mechanical, physical and metallurgical characterization of the work material SS-430 which was joined by using the microwave. Hardness of the joint was also performed to check the strength of the joint. SEM micrographs of the microwave joint and the nickel powder are presented. Tensile testing of the joint is also presented which was done to calculate the ultimate tensile strength of the work material SS-430. XRD of the joint is also presented which was done to saw the internal mechanism of the joined work material.

4.1 CHARACTERIZATION OF THE NICKEL POWDER

Fig 4.1 shows the SEM micrograph of the nickel powder. From the figure it is clear that the particles of the nickel powder are spherical in nature having average particle size of 40µm.

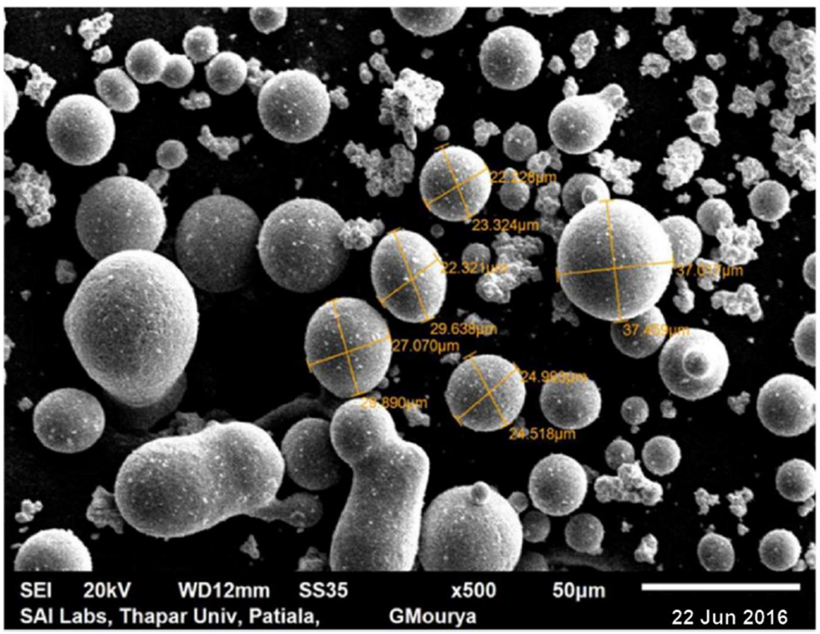


Figure 4.1 Typical SEM image of nickel powder particles having spherical shape

Figure 4.2 shows XRD spectrum of nickel powder The XRD diffraction analysis of nickel powder was carried using CuKα radiations at a scanning rate of 1° per minute in the 2θ range of 10–120°. The spectrum shows most of high peaks are occupied by element nickel which proves dominating percentage of nickel in powder but along with that spectrum also shows small percentage of chromium and silicon.

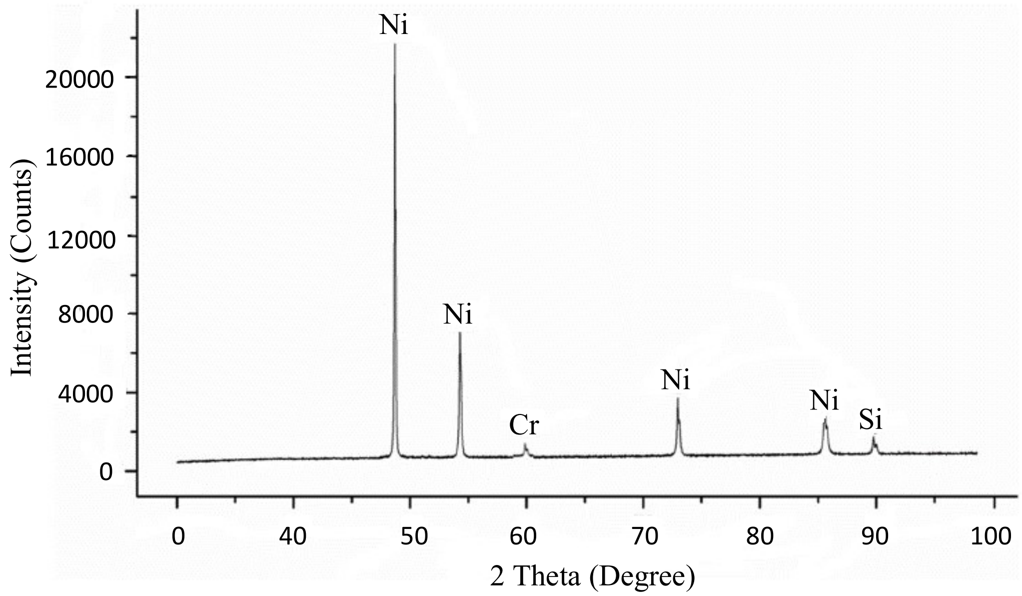


Figure 4.2 XRD Pattern of Nickel Powder

4.2 CHARACTERIZATION OF JOINTS

Figure 4.3 shows the joint produced by the microwave joining. Characterization like SEM, EDS and the XRD were performed to analyze the joint made by the microwave technique. XRD helped in knowing the chemical composition of joint and chemical composition helps in getting information about mechanical property of joint.

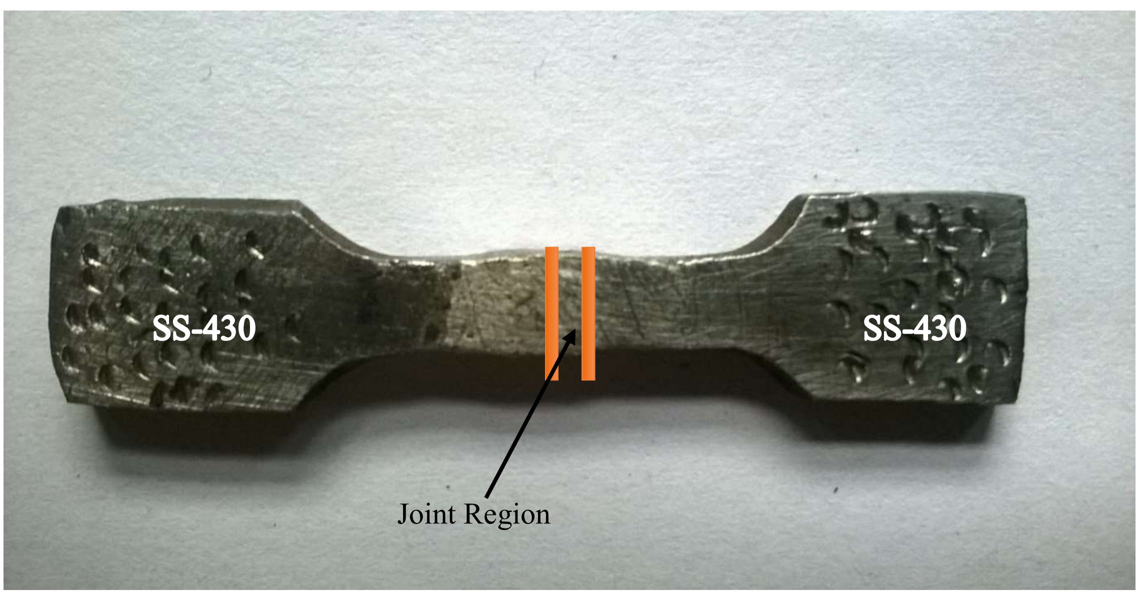


Figure 4.3 Microwave Processed Joint

4.2.1 XRD Analysis of Joint

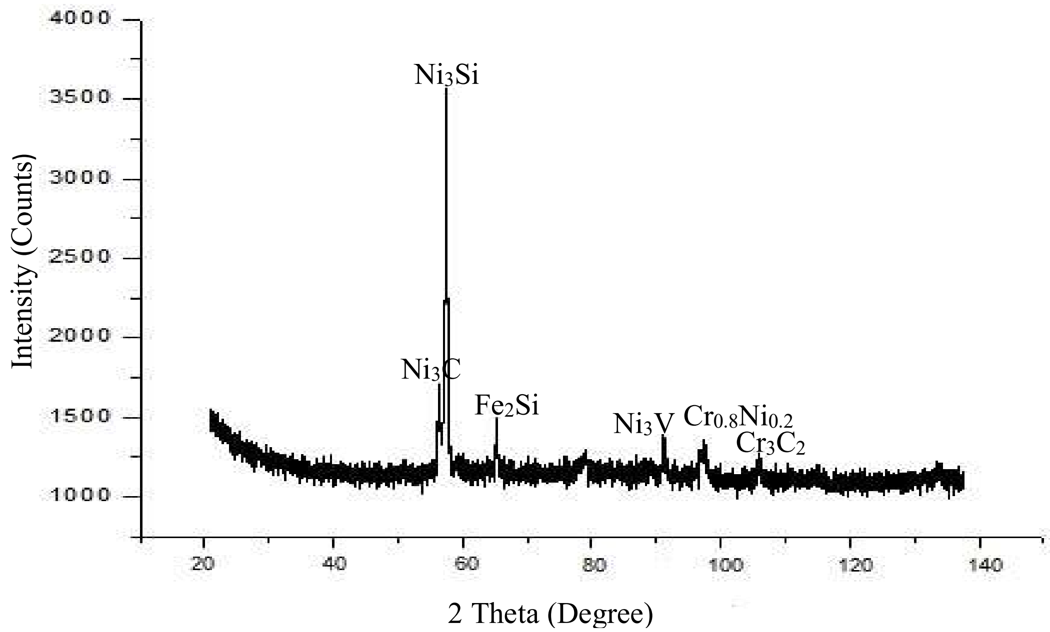


Figure 4.4 XRD Spectrum of Microwave Processed Joint

The results of XRD tests showed the presence of phases of nickel based compounds in the microwave processed joints of SS-430, which is the main constituent of interfacial powder. It is observed that during MHH, the powder particles form various carbides and intermetallics in the joint zone, although, in the starting all the elements were present in the form of a solid solution. This is attributed to a strong affinity of the element like Cr towards carbon at high temperature. In SS-430 joint, the major phases of Ni_3Si, Fe_2Si, Ni_3C, Cr_3C_2, Ni_3V are present. 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.

4.2.2 Microstructural analysis of the joint

Figure 4.5 shows the joint of SS-430 material formed by the microwave technique. From the micrographs it is clear that the joint is having the wavy interfaces which proves that the diffusion of the base metal and the powder is occurred. From the SEM micrographs, heat affected zones can be easily seen.

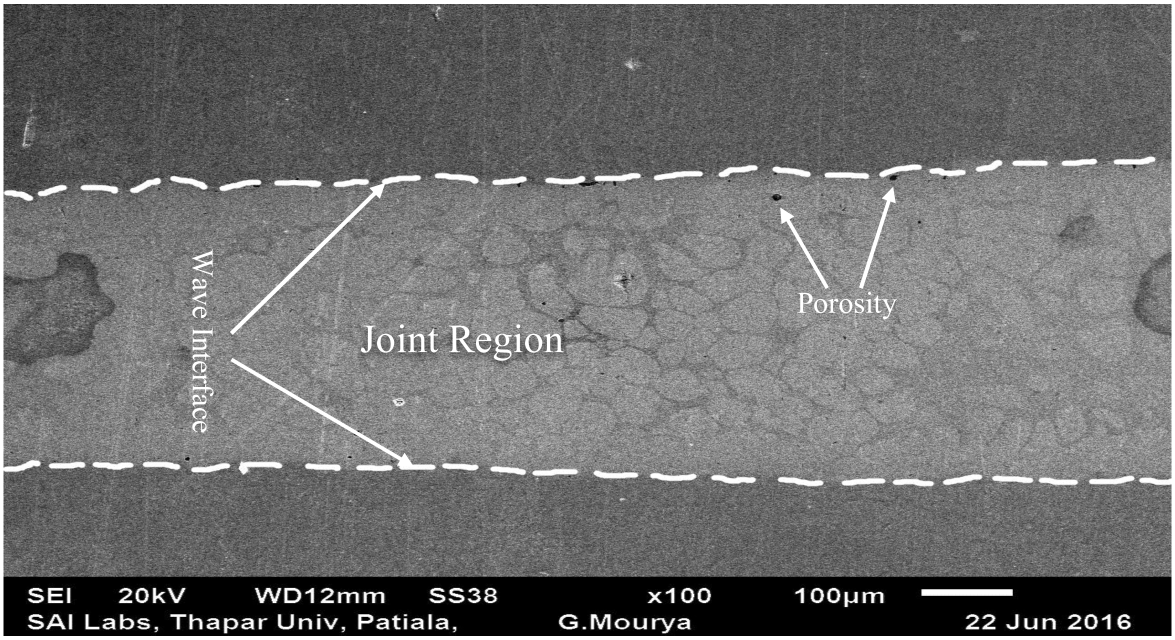


Figure 4.5 SEM image of Joint Region

The EDS was also done as shown in Figure 4.6 to check the chemical composition of the joint. The result shows that the joint has mainly nickel and iron as all the high peaks in the spectrum are occupied by these elements.

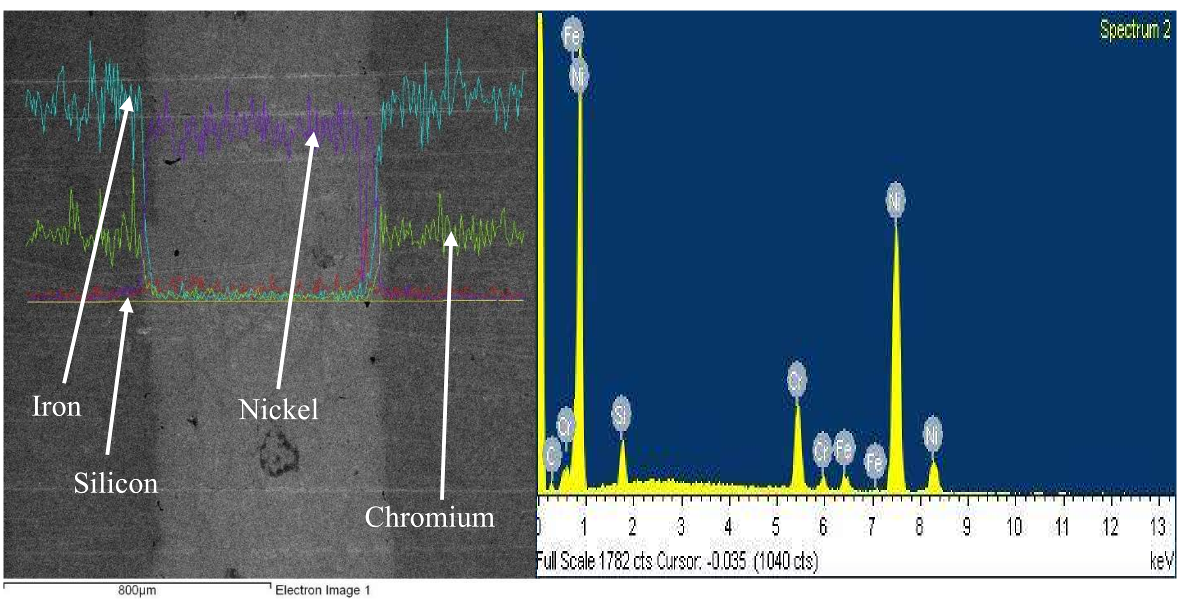


Figure 4.6 Elemental analysis of Joint

The SEM image shown in Figure 4.7 shows complete fusion of base metal with the interfacial powder. The formation of nickel and iron rich cells was observed enclosed by carbides and intermetallic chains.

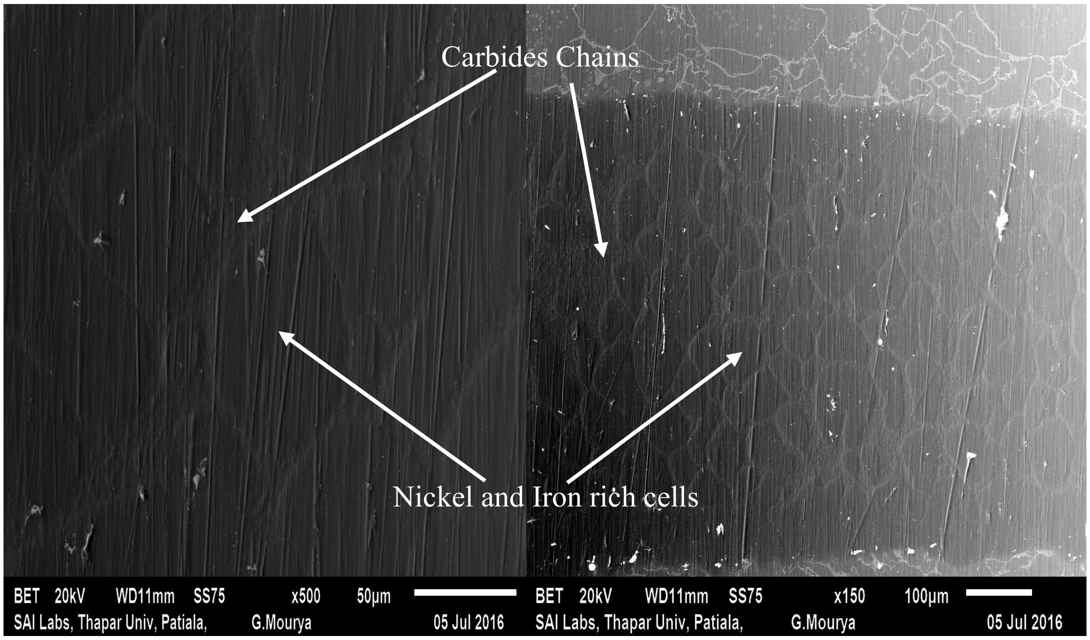
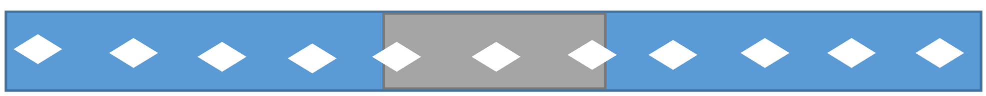


Figure 4.7 SEM image showing Carbide Chains and Nickel rich cell

4.2.3 Mechanical characterization of joints



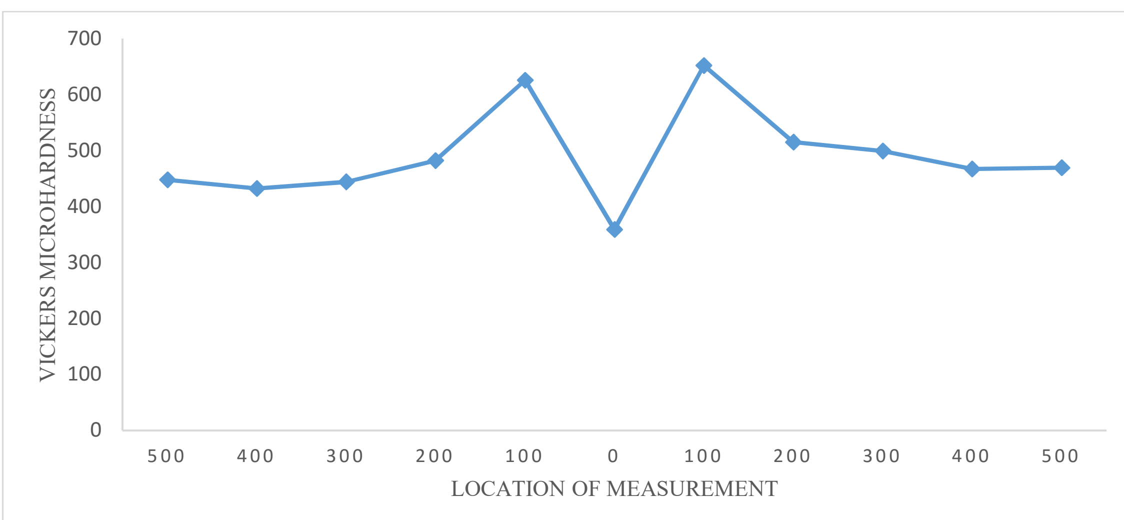


Figure 4.8 Micro hardness of Joint at various points

Micro hardness tester and UTM under mechanical testing machines were used to characterize the joint strength of the work material. The values obtained by the micro hardness tester has shown in Figure 4.8 Hardness at the boundary of filler material and base material is higher than hardness at center and base material while hardness at center is lowest. The reason behind it is, formation of martensite [36] at boundary of filler material and base material and formation of Ni_3Si and Ni_3C. Hardness of martensite is too much high that is why peaks at boundary is highest. While hardness of Ni_3Si and Ni_3C is not too much high it varies from 350 to 400 at room temperature and value obtained from test are in this range. It has been verified that reason behind such low hardness at center is formation of Ni_3Si and Ni_3C.

The tensile strength of the joint calculated by using the UTM was observed to be very high as shown in table 4.1 but there was no elongation as the fracture was brittle. The reason behind the brittle facture of joint may be due to Ni_3Si is a part of binary eutectic matrix, Binary eutectic is composition of Nickel solid solution and Nickel Silicide (Ni_3Si). Carbides are also present in the eutectic matrix but they are in very percentage by volume. Ni_3Si is brittle in nature [37]. It has one amazing property that's corrosion resistance because of formation of high silicon layer. 29.1% of Ni_3Si was found in the welded region As James studied in his paper that formation of Ni_3C is brittle in nature [38]. And formation of Ni_3C in welded joint was around 11.2% which is quite dominating in percentage by volume. Both Ni_3Si and Ni_3C are present in the welded region and both are dominating in volume percentage wise Since it was seen that Ni_3Si and Ni_3C both are brittle in nature so that's how fracture that occurred during testing was brittle.

Table 4.1 Shows the Results of the tensile testing

Material	**Microwave Processed Joint**	
	Strength (MPa)	% elongation
SS-430	471	No elongation

CHAPTER 5 CONCLUSION AND FUTURE SCOPE

5.1 CONCLUSIONS

Microwave processing technique is used to process different materials ranging from the ceramic sintering to the metallic materials sintering. Further, microwave processing technique is being used for joining and cladding of the work materials. The present research work was based on utilization of microwave energy to join the work material SS-430. Major conclusions drawn from the present study is shown below.

- SS-430 work material was successfully join by the microwave technique using nickel as a base powder.
- Characterization was done to check the joint and from the results SEM it was clear that structure of the joint was cellular, having less porosity and almost no cracks.
- Due to good metallurgical bonding between the work materials; mechanical strength of the joint was observed to 471 MPa.
- Due to formation of the carbides and martensite at the joint; higher hardness of the joint was obtained.
- Tensile testing proves that the behaviour of the work material was brittle in nature due to presence of Ni_3Si, Ni_3C, and addition of carbon from the graphite sheet and also the formation of martensite mainly at the boundaries.

5.2 FUTURE SCOPE

Based on the study following points can be considered for the future work by using the microwave technique:

- To analyse the effect of the microwave energy exposure time on the ductility of the joint which can be considered in terms of percentage of elongation.
- Alumina or SiC sheet can be used as a separator which will prevent the addition of the carbon content into the joint which further reduce the brittleness of joint.
- To analyse the effect of the microwave energy exposure time for the fractographic analysis of the joints.
- Microwaves can be used to weld the materials ranging from the soft to hard material.
- Microwave processing technique can also be used to optimize the parameters of the welding.

- Software’s like ANSYS and the COMSOL can be used to perform computational work for processing of work materials by using microwave technique.
- Brittle Characteristic shown by formed joint of SS-430 can be rectified using the post heat treatments.

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