

Friction stir welding of nuclear grade material Cu-Cr-Zr and SS 304

A

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CERTIFICATE

I hereby declare that the report entitled "Friction Stir Welding of nuclear grade material Cu-Cr-Zr and SS 304" is an authentic record of my work carried out as requirements for the award of the degree of **Master of Engineering** at **Thapar Institute of Engineering and Technology, Patiala** under the supervision of **Dr. Ajay Batish (Professor, DD & DOCA), Dr.Dinesh W Rathod, Mechanical Engineering Department**. No part of the matter embodied in this report has been submitted to any other university or institute for the award of any degree.

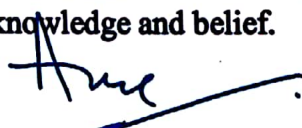


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It is certified that the above statement made by the student is correct to the best of my knowledge and belief.



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.....**Anoop singh**

ABSTRACT

Dissimilar metal joints are used in several industries sectors like petrochemical, thermal, nuclear, hydrolytic pairs and refrigeration and air conditioning. Dissimilar weld joints between copper and SS are demanded in different units of air refrigeration and air conditioning system. Particularly in nuclear industries, the cold leg condenser units of reactor pressure vessel system demands the joint between Cu-Cr-Zr alloy and SS 304 L materials. Owing to varying and distinctive metallurgical and material properties joining of these alloys remain a big challenge. In thick section arc welding processes are the conventional option to weld them together lower joints could exhibit with metallurgical deterioration caused by arc welding process. Solid-state welding process are low heat input processes and can be used for joining these type of material to address the issue of metallurgical deterioration of weldment region. In present study, low heat input solid state welding process, friction stir welding process have been used to investigate the feasibility of joint on comparatively thin sections of 3 mm thickness. Significant amount of trial experiments were performed as preliminary investigation to decide the root map for investigation the failure of tool and its causes and effects were recorded and analysed in the study. The virtually inspected and qualified as welded joint between SS 304 and Cu-Cr-Zr alloy has been used in this study. The specimen fabrication and experimentation procedure is described along with the welding and testing procedure. Several mechanical and metallurgical test were performed and the results were described in the study. The results of mechanical, metallurgical and fractography test were analysed and correlated with each other in weld qualification test (cross-weld tensile test), the specimen fractured from the HAZ /TMAZ regions of Cu-Cr-Zr alloy and the present investigation analyse the failure case of HAZ/TMAZ zone. The analysis of test result validated by other test also elaborated in this study. Heat produced in the FSW influences the Cu-Cr-Zr alloy side in through thickness in metallurgical aspect and resulted in different mixed mode fracture failure. However, study suggest the feasibility of joint using friction stir solid state welding process.

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1.1 Friction stir welding

In today's aerospace and automotive industries, the requirement of strong parts having light weight and high strength is very challenging in this advanced scenario. Hence advanced welding process need to be used over conventional methods. In high technology applications such as micro welding where high precision is required, unconventional welding i.e. Friction stir welding is used. Joining of dissimilar metals provides combined benefits of mechanical properties, overall cost reduction, and metallurgical properties at two different ends. Various combination of material are explored and continuously being used for critical components in nuclear industries in product like heat exchanger, fusion reactor, particle accelerator etc. [1-3] Friction welding (FW) and Friction stir welding (FSW) are two types of unconventional welding widely used in the advanced industries. Both the methods are capable of producing equally strong joints. Both the processes have few differences. Both similar and dissimilar metals can be joined by this process [4, 5]. Due to energy efficiency and environment friendliness FSW is considered as the green manufacturing technique. The welding institute (TWI) of United Kingdom invented the process of friction stir welding in 1991. Solid state metal joining process was initially applied to aluminium alloys[6].

In nuclear industries, stainless steel and copper plays an important role where copper has high thermal conductivity whereas stainless steel has high strength and nature of corrosion resistance. welding of cu and stainless steel is considered to be challenging because of its unique and different properties .They have different melting point, thermal conductivity and having different coefficient of thermal expansion.[7].Due to aforementioned differences, to obtain joints between these two materials fusion welding process are proved weak.

By different techniques such as explosive welding ,electron beam welding ,shielded metal arc welding, hot isostatic pressing, tungsten/metal gas suspended arc welding , brazing ,Friction stir welding Cu and SS can be joined but various problems arises during solidification like porosity , cracking and residual stress that triggered permanent failure[8].

EW which is solid state welding process involves explosive materials whereas electron beam welding and low beam welding have higher degree of complexity. Hot isostatic pressing enforces repetitive heating and cooling cycles thus it makes heterogeneous joints because it restricts grains growth. Apart from these processes, friction stir welding (FSW) shows excellent feasibility relative to these parameters.

1.2 Process

FSW is a hot shear solid state process. Tool has a specific diameter of shoulder and pin having a specific angle of 63.7 degree taper and .threads. The main reason behind the taper and threads on the pin is to influence flow of the material towards inwards direction. Taper pin with the effect of centripetal forces when plunges into the work piece the workflow of material becomes faster and in inwards direction .Thus probability of welding increases and number of defects decreases. The quality of weld and finishing depends on the materials, rotational speed and the feed provided to the tool. This is a hit and trial method and we have to find the optimum welding parameters by changing rotational speed and feed. Throughout this process, material experiences intense plastic elongation and plastic deformation.

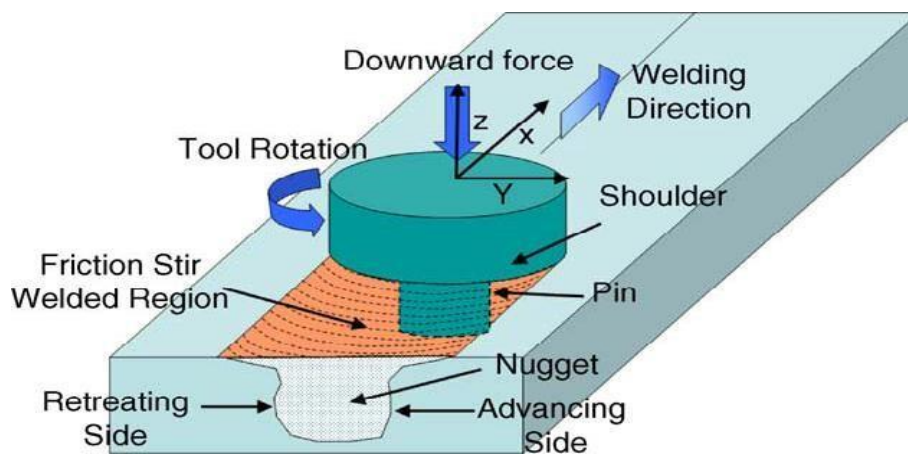


Fig 1.1 Schematic diagram of friction stir welding (Mishra and Ma 2005)

A non-consumable profiled pin rotating tool with some rotational speed plunged into the interface between two plates and generate sufficient frictional heat and deformation to federate the two materials together [4].frictional heat is sufficient to plasticize the material beneath the tool shoulder. In order to utilize the unique properties of Cu-Cr-Zr/SS304 i.e. high electrical and thermal conductivity, pleasing combination of strength and ductility, supreme resistance to corrosion made this alloy favourable to nuclear industries [9].

1.3 Weld zones

After friction stir welding, weld zone on the basis of microstructures regions are divided into 3 parts

- The weld Nugget
- Heat affected zones(HAZ)
- The thermomechanical heat affected zone

- The heat effected zone

Parent metal or unaffected region:

The region which is non-effected and non-deformed during welding region and far away from the weldment region although during welding it may experience some thermal cycle.in this region , there is no effects on the micro structure and mechanical properties due to this heat generation.

Heat affected zone (HAZ):

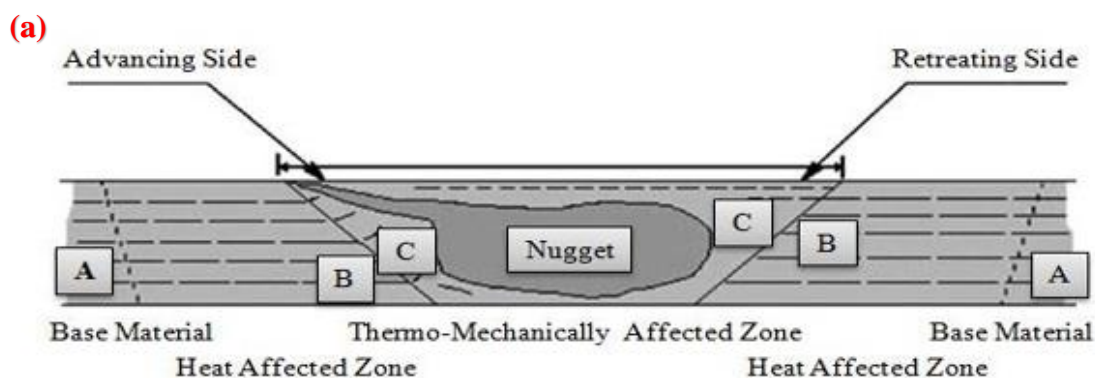
Thermal cycle is experienced by the material in this region and it lies closer to the weldment. Due to the intensive heat mechanical properties and microstructures are modified. However in the region of heat affected zone there is no plastic deformation occurs. At local level coupled with the temperature rise and the region undergoes plastic deformation having temperature rise less than the weld nugget. The preferred name is ‘Heat affected zone’ because of the region direct parallel to the heat.

Thermo-mechanically affected zone (TMAZ):

By the FSW tool, the material has been plastically deformed, the material is also effected by the heat produced during FSW process. Without recrystallization it is possible to get substantial plastic strain in case of aluminium. Generally, we can differentiate between the boundaries of deformed zones and recrystallized zone of the TMAZ.

Weld Nugget:

Generally. Recrystallization temperature in the area of TMAZ called as nugget where intermixing of two materials occurs. The area below the tool shoulder where pin is inserted into the material is weld nugget. The mixing and solidification of metals occurs at joint is generally called as weld nugget. It is due to the continuous dynamic re crystallisation.



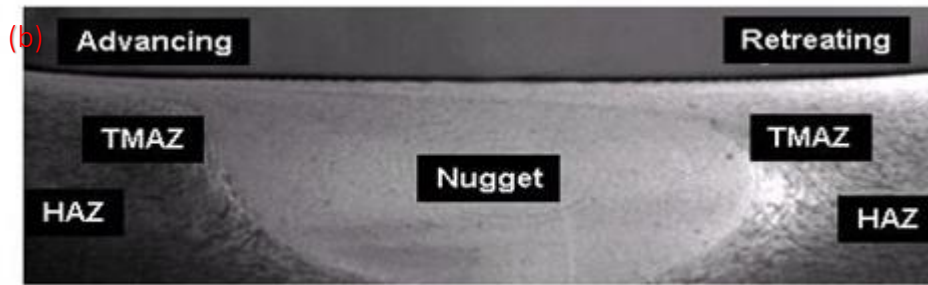


Fig: 1.2 (a) Schematic diagram of micro structural zones in friction stir welds in Al (Threadgill, 2007)

(b) Micrograph showing various micro-structural zones (Threadgill, 2007)

1.4 Vertical milling machine/Friction stir welding machine

We can perform friction stir welding on vertical milling machine. Rotational speed, feed, speed can be varied according to the material. We are using vertical milling machine because of its stability and low vibrations and features of varying parameters. Fixture is mounted on the vertical milling machine. Thus first step is proper testing of machine, fixture, tool and providing proper lubrication to the machine in order to avoid vibrations.



Fig 1.3 Vertical milling machine



Fig 1.4 Modes

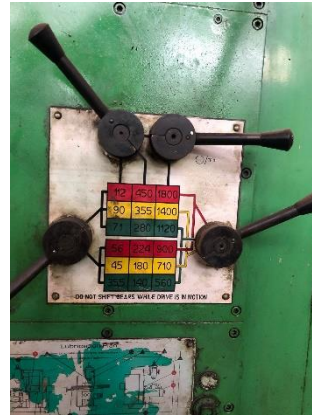


Fig 1.5 Parameters

Table 1.1 HMT FN2V Milling Machine

| | |
|--------------------------------|-----------------|
| Power operated table traverses | 800 mm |
| Number of speed | 18 |
| Speed range | 35.5-1800 rpm |
| Main motor | 5.5/1500 kW/rpm |
| Feed motor | 1.5/1500 Kw/rpm |

1.5 Material and work piece

Cu-Cr-Zr has a significant use in the nuclear industries while stainless steel grade SS304 has unique properties of high temperature resistance, good magnetic properties, excellent forming and welding characteristics. Thus, friction stir welding of Cu-Cr-Zr/ SS304 can give us properties that can be very useful. 3 mm Cu-Cr-Zr and 3 mm stainless steel plates are taken for the process of friction stir welding.

Advantages of friction stir welding:

- I. Dis-similar metal can be welded.
- II. The process is environment friendly because no fumes are generated thus no shielding gas is required.
- III. The friction stir welding process uses non-consumable tools.
- IV. There is no constraints of any position can be used in any direction i.e. vertically, horizontally and overhead.
- V. Very low shrinkage is observed in this process because the temperature rises during the welding is below the melting point.

- VI. Tool life is greater in the friction stir welding thus can be used for various materials and distance up to 1000m.
- VII. No Fusion or filler metal are required.
- VIII. No formation of slag in it.
- IX. Superior weld quality with fine structures and having excellent mechanical properties weld are obtained in this welding.
- X. The process is cost effective because the process can easily performed in vertical milling machine.
- XI. Chances of automation.

1.6 Friction stir welding applications

I. Aerospace industry:

For the welding of proto-types parts friction stir welding process is using in the aerospace industry. In the military and the civilian aircraft where two dis-similar metals are required FSW process is used. Friction stir welding provides benefits over riveting that can reduces weight and saves manufacturing cost.

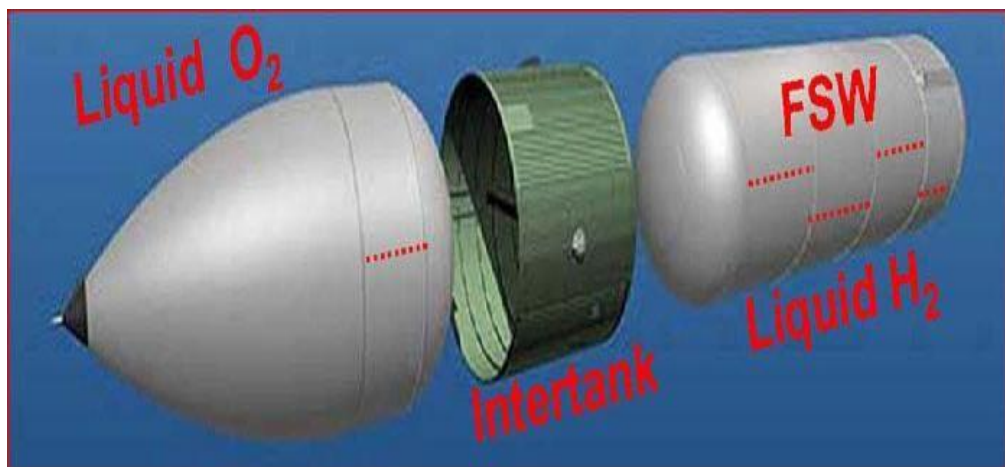


Fig 1.6 Rocket fuel tanks fabricated by FSW (Rowe and Thomas, 2006)

II. Railway industry

In high speed trains making of container body, underground garages, railways tankers and good wagons are generally made up from friction stir welding.



Fig 1.7 Japanese railway rolling stock fabricated from aluminum by FSW (Rowe and Thomas, 2006)

III. Shipbuilding and marine industries:

For the commercial applications the ship building and the marine industries have adopted the friction stir welding process. For marine and transport structures, helicopter landing airports, panel for decks and floor are generally constructed by FSW process.



Fig 1.8 Aluminum ships constructed in Japan using FSW (Rowe and Thomas, 2006)

IV. Thermal power plant:

Specific application of Cu-Cr-Zr /SS304 LNin reactor prime vessel system, however in thicker section. Its feasibility need to investigate in solid state welding process like friction stir welding. Hence 3 mm thick material are conducted for investigation.

V. Electrical industry:

In Electrical connectors. And Electric motor housings the electrical industry shows increasing interest in the application of FSW.

Limitations of Friction stir welding

- I. Due to pin of the tool at the end of weld plate there is a keyhole.
- II. Welding speeds are moderately slower.
- III. For defect free welding, fixture must be kept tight.
- IV. Design and development of fixture is required.
- V. Backing plate is required.
- VI. Due to the variations in the thickness of welding materials pin tools having different length is required.

1.7 Development of fixture

The main purpose of the fixture of friction stir welding is to hold the work piece in position during welding. Fixture must be designed in such a way that heat must be dissipated quickly through it and provide enough area between tool shoulder and work piece. Its main aim is to prevent motion and remain stationary with the bed of the machine.

Main parts of fixture:

- Base plate
- Fixed plate
- Side supporting plate
- C clamps
- Backing plate

1.8 MATERIAL OF BACKING PLATE:

Table 1.2: Parts used in fixture

| | PART LIST | MATERIAL | NUMBER |
|----------|--------------------------|-----------------|---------------|
| A | Base plate | M.S | 1 |
| B | Movable supporting plate | M.S | 1 |
| C | Fixed side plate | M.S | 1 |
| D | C clamp | M.S | 2 |
| E | Backing plate | M.S | 1 |

| | | | |
|----------|--------------|------|---|
| F | Nut and bolt | C-30 | 9 |
| G | Hinges | S.S | 2 |

CHAPTER 2

LITERATURE REVIEW

Thomas *et al.* [9] the welding institute [TWI] discussed the problems rises in fusion welding of aluminium alloys. They developed a method of Friction stir welding in order to avoid formation of surface oxides and prevent base metal. The main advantage of this process is that it uses non-consumable tool which rotated at some rpm and when it comes with the contact of work piece produces heat by friction which results in joining of metal. Success in welding occurs by excessive deformation of material below its melting point at room temperature. That's why friction stir welding is also called as solid state welding.

Steuwer *et al.* [10] worked on the residual stresses developed in AA5083-AA6082 by changing various parameters and revealed that as compared to the rotational speed and the transverse speed for friction stir welding, rotational speed of the welding tool is more effective than the transverse speed of the residual stresses especial in AA5083.

Martins *et al.* [11] the joining of copper to stainless steel by friction stir welding is very difficult because of different chemical composition and thermal physical properties. There is deterioration of mechanical properties due to the formation of inter metallic phases. The advantage of friction stir welding is minimal detrimental effect on both material in this lap joint is produced by changing various parameters.it concluded that the shear strength of the lap joint is affected by material thickness.73.8% of joining efficiency were achieved.

Ogura *et al.* [12] lap joint was welded by friction stir welding of Stainless steel / aluminium alloy and mechanical properties were characterized. In AA303 Aluminium alloy /SUS304 steel lap joint it is concluded that the retreating side has the lesser strength as compared to the advancing side. Sound joint is produced when amorphous layer is formed owing to the mechanical alloying effects.

Yilmaz *et al.* [13] by diffusion bonding and soldering copper and stainless steel 304 were joined at different bonding conditions. It concluded that at the interface, formation of phases limited the inter diffusion of elements across the bonds. The correlation of temperature and electrical resistivity of the interface change with the polynomial function obtained by soldering

and diffusion bonding. Various phases are formed $\gamma_{Cu} + \alpha_{Fe}$, $\gamma_{Cu} + \alpha_{Fe} + \gamma_{Ni}$, $\alpha_{Fe} + \sigma$, $\alpha_{Fe} + \gamma_{(Fe,Cu,Ni)}$, $\gamma_{(Fe,Cu,Ni)}$ also called as ternary equilibrium diagram.

Hwang *et al.* [14] pure copper under goes friction stir welding and butt joint were produced in order to explore thermal properties of workpiece.at different locations in the work piece, temperature was noted with the help of K-type thermocouple. The temperature with ranging 460 °C and 530 °C is found to be appropriate for friction stir welding. Observed that temperature is slightly higher on the advancing side rather than retreating side. TMAZ has the tensile strength and hardness of 60% of base metal. The elongation can reach up to 3 times of base metal.

Celik *et al.* [15] at different rotational and traverse speed of (630, 1330, 2440rpm), (20, 30,50mm/min) friction stir welding process is performed on the material of pure copper and 1050 Al alloy. Best welding performance of 89.55% with a rotational speed of 1330rpm having 20mm/min traverse speed was calculated. At the stir zone region, a fine layer of copper particles distributed over the Al material because of the higher tensile strength. Cylindrical pin tool provides effective results in friction stir welding. In low tensile strength specimen, copper content found to be very less in the weld zones. Homogeneous composite structure have higher strength values. During phase analysis, intermetallic phases of $CuAl_2$ and Al_4Cu_9 were found in weld region.

Jafari *et al.* [16] friction stir welding process used to weld stainless steel SS304 to copper. The characterisation of joints resulted that grain size become smaller in weld region for each base metal as compared to TMAZs, HAZs, and base regions. As welding pass number increases ductility and strength of friction stir welded samples decreases. Growth in HAZ grains on the side of copper because of heat made during the subsequent drag. As steel has higher rigidity then copper, 1 mm of pin shifted towards the steel. TMAZ and HAZ has lower hardness value as compared to the weld regions. SEM –EDS analysis resulted that there is a diffusion of nickel from 304L steel to the copper side.

Chen *et al.* [17] optimum operating conditions of frictions stir welding of AA6061 aluminium alloy and SS400 low carbon steel were observed. In significant parameters of FSW, low transverse speed and operational speed were observed. For the best quality of impact values transverse speed of 0.9 mm/s and rotation speed of 550 rpm provides acceptable quality of

tensile strength. At 150 degree angle specimen remains unbroken and considered as best quality specimen.

Keivani *et al.* [18] to measure the temperature of the work piece numerical method can be used. The effect of pin angle and pre heating corresponds to temperature distribution studied. The analysis shows that in the weld line there is an enhancement of temperature by increase of pin angle. There is no effect in temperature distribution by preheating.

Rathod *et al.* [19] with ERNiCr-3/ENiCrFe-3 consumables, fill-pass welding and buttering deposition varied in welding process. There is a development of more favourable properties by adding NI-Fe layer in it. Buffer layer has a consequent effect on mechanical and metallurgical properties. There is control of carbon migration and lesser angular distortion was achieved. By buttering, weld metal chemistry changes by increasing in solubility. Wider dendrite arms are formed as compared to without buffer layer

Joshi *et al.* [20] heating and cooling corresponding to the friction stir welding was studied where pre heating current was varied. The parameters set are 20, 40 and 60 A. In case of cooling corresponds to FSW varied in compressed air flow rate as 15 -30 PSI. Water cooling applied at 75ml/min. it is resulted that surface oxides are formed in case of pre heating and elimination of surface oxides cooling by compressed air. On the increase in cooling conditions, the distortion of tensile strength increases. In Normal FSW highest tensile strength was noted as in case of preheating. Pre heating assisted FSW gives poor macro structure. SS particles diffused inside Cu particles reported as a major defects.

Soni *et al.* [21] different types of defects are formed in FSW and thus effected the weldment. Change in the micro structural and mechanical properties were observed. By optimizing the parameters we can overcome with the defects. In case of AA508 and pure copper channel defect was produced near sheet interface because of low welding temperature. In nugget zone, various defects were identified at lower rotation speed of 400 rpm. Good metallurgical bonding are observed between copper and aluminium at higher rotational speed of 800 and 1000 rpm.

Zhang *et al.* [22] examined that design of tool affects the tool forces and the temperature in the welding direction. In tool forces, temperature rise plays significant role. Increase in welding temperature is observed as increase of rotational speed and tool shoulder size. At 500 rpm, 140 mm/min the maximum rise in temperature is 430°C. In case of conical pin, slightly decrease in maximum temperature of 426°C was observed. Decrease in maximum temperature was observed from 384°C to 344°C when tool radius changed from 10mm to 8mm. As the rotating

speed is increased from 550rpm to 600rpm, the maximum temperature also increased from 393°C to 400°C. With the decrease of temperature, Frictional stress can be changed.

Cabibbo *et al.* [23] different grain morphologies of microstructure in thermos mechanically affected zones was observed. There is a fairly elongation and bent grains were found in the advancing side. Narrow grains and huge elongation was found in the retreating side. Tensile test resulted that as compared to the parent metal yield and ultimate strength gets lower across the weld while reduction in ductility is also seen in weld element. Minimum of hardness achieved as increase in frictional heat. In case of TMAZ, aspect ratio increases and in case of HAZ aspect ratio deformed very slightly.

Sun *et al.* [24] 5 mm thickness of magnesium alloy AZ61 plates were welded at different ω/v ratio. The average ultimate tensile strength of all weld conditions satisfying a ω/v ratio. s, it was determined by electron backscatter diffraction analysis and scanning electron microscope that grain orientation changes in the interface between the thermomechanical affected zone and the stir zone and it is also the weakest part. The retreating side region because the grain orientation whereas the advancing side region was relatively weaker.

Chandrashekar *et al.* [25] the quality of tool depends on the flow conditions of the mixing of the material. Critical issues in the FSW process is to design tool and selection of variables. Tool performance is dependent on material selection and design. In stir development zone, there is an important role of shoulder to pin ratio. Shape and size of tool and shoulder, effects plastic flow and heat generation. Threads on the tool also play an important role in material flow.

Chiteka *et al.* [26] (steel) on the aspect of microstructure and mechanical properties of the joint it was determined that carbon content, welding speed and rotational speed plays significant role. In case of carbon steels micro structures and mechanical properties are greatly affected by welding parameters. Phase transformation occurs in carbon steels due to higher rotational speed. Sound joint in steels also produced when peak temperature and cooling rate also decreases than critical cooling rate. In the SZ of carbon steels friction stir welding has grain refining effect. The improvement in mechanical properties by FSW was seen as compared to the BM. Homogenous micro structure are found in the joints and ductility improved in the joints.

Ogura *et al.* [27] by friction stir process slices of aluminium alloy/stainless steel dissimilar lap joint was produced and characterized. It also indicated that advancing side have higher strength

than retreating side. Due to the formation of amorphous layer sound joint was produced that fractured at the base metal. Various intermetallic compounds were formed in it. Micro tensile test shows slightly deformation of the interface and resulted that tensile strength increase with aging at 433k.

Babu *et al.* [28] friction stir welding performed on Al-Cu alloy to stainless steel AA 2219-T87. WC-Co tool with different tool profile were carried out for welding experiments. Tensile strength of 250 MPa was successfully produced. Rugged and some fragments of stainless steel found in stir zone. Intermetallic layers are observed having FeAl, Fe_3Al and $AlCrFe_2$ phases in SS/Al interface.

Marstatt *et al.* [29] analysed the intermetallic monolayers of aluminium and copper joint interface using SEM and STEM. The two metals were joined using friction stir welding (FSW). It was noted that the process temperature is directly related to the rotation speed. The temperature increases up to 390°C. Also the tensile shear strength of joints is correlated with the rotational speed. An intermetallic double layer consisting of AlCu and AlCu₃ forms during FSW process. It was seen that there is well defined columnar growth of intermetallic compounds at bonding interface of material. Arrhenius equation was used to show the relation between interlayer thickness and temperature. This layer was formed as a result of interdiffusion between Al and Cu phase after the oxide layer was being removed by rotating probes.

Shokri *et al.* [30] obtained the microstructure and studied mechanical properties of joined copper alloy and duplex stainless steel (DSS). It was revealed that the thickness of interface was directly dependent on temperature. The mechanical properties of the interface were noted at different temperatures. It was concluded that at low temperature, metallurgical bonding is very weak and hence, insufficient mixing occurs. But at high temperature, a thick interface layer is formed which results in causing stress at interface and hence premature failure. The highest tensile strength was obtained to be 279 MPa at intermetallic layer thickness of 55 µm. This value of highest tensile strength was obtained at rotational speed of 1200 rpm, travel speed of 30 mm/min and tool offset of 0.5 mm.

Chiteka *et al.* [31] discussed how to select tool material so as to obtain good welding quality as result. The foremost requirement for the selection of tool material is that it should not result in contamination in interface joint. The parameters determining the best tool material are rotational speed, transverse speed, the type of material to be welded and the material

characteristics. It was seen that the soft materials can be easily welded using hard tool such as steels materials but the hard materials requires more harder materials (such as carbide based materials) for effective welding.

Kumar *et al.* [32] welded different combinations of steel, copper and aluminium with 3 mm thickness using FSW. Different combinations that are: copper and copper, copper and aluminium, aluminium & stainless steel, stainless steel & copper were obtained after welding. During FSW the rotational speed was maintained at 1120 rpm and attachment speed within the limit of 14-112 mm/min. Microstructure of these welded materials and parent materials were observed. It was concluded that aluminium and aluminium, copper and copper, aluminium and copper can be easily welded and mixed. For stainless steel to be welded, the rotational speed should be kept high and tool withstanding capability should be more than 60. Due to which the material with stainless steel possess higher hardness and hence difficult to melt.

Rathod *et al.* [33] analysed the structural and mechanical characteristics of four types dissimilar metal weld joints obtained between low alloy steel (SA508Gr.3Cl.1) and stainless steel (SS304LN). Ni-Fe alloy was used as intermediate layer in buttering as a graded composition in welding process. The microstructure of the joints is governed by the diffusion between carbon and graded compositions, which affects the mechanical properties of welded materials. The variations in welding process obtained due to introduction of Ni-Fe alloy layer affected the distortion behaviour of joints.

Chandrashekar *et al.* [34] discussed the effect of tool geometry (straight cylindrical, taper cylindrical, triangular, square, trapezoidal and hexagonal tool etc.), its rotational speed, and weld speed on the welding joints formed. Ratio of tool shoulder to pin diameter greatly effects the development of stir zone. The pin diameter is equal to the thickness of the parts to be welded and its length is slightly shorter than the thickness of the part. The welding quality, tool performance depends upon the mechanical properties of tool that are: strength, fracture toughness, hardness, thermal expansion coefficient etc. The shape and size of tool shoulder and pin affects the amount and rate of heat generated during welding process. Defects generated in final material are also dependent on the type of tool design chosen.

2.1 Conclusion from literature review

Following are the points which can be concluded from the literature review:

- FSW occurs below the melting point of work piece and for high temperature process cylindrical tool with circular pin can be used.

- Redistribution of reinforcement particles by the stirring action of the FSW tool.
- Welding of Copper alloys and stainless steel has a nuclear grade application.

The ultimate area of present work is twofold: to produce repeatable, robust having excellent mechanical and metallurgical properties.

2.2 Gaps in the literature review

Some of the gaps in the literature review are given below:

- Less amount of work is reported on FSW on copper alloys and SS 304.
- Optimum parameters are very critical to find (Hit and trial method is applied).
- Very less information about tool failure.

2.3 Problem formulation

Copper holds excellent conductivity while SS 304 has good mechanical properties. Welding of both the metals can give us a combination of good mechanical and physical properties. Optimum parameters can be found by hit and trial method so the present work is to study the different process parameters of the process. The main objectives of proposed work listed below:

- Preparation of welding plate (optimum parameters)
- Sample extraction
- Evaluation of mechanical and metallurgical properties.
- Cross comparison between welded zones to parent metal.
- Micro structure analysis.

3.1 Problems Associated with Dissimilar Metal Welds [22, 26, 33]**Cyclic thermal stresses:**

In dissimilar metal welding the coefficient of thermal expansion plays an important role. Tensile stress and compressive stress are developed in the adjacent materials because of large difference in the values of coefficient of thermal expansion. During welding hot cracks are induced by the tensile stress whereas cold cracks are produced by mechanical and thermal effects. In the cyclic temperature mode where joints operate at high temperature this type of issues are common. Due to the dissimilar weld each weldment region has distinct Coefficient of thermal expansion values. The fluctuations of CTE values generated cyclic thermal stresses. Tangential, radial and longitudinal stresses are produced whenever there is different coefficient of thermal expansion due to heating and cooling of joints. Failure occurs when residual welding stress, external loads and internal steam pressure superimposed with cyclic thermal stresses.

Carbon Migration:

In the weld interface carbon migration plays a significant role for dictating the life of weldment. Carbon enriched and carbon depleted zones are formed in it. In the heat affected zone carbon from one metal transfers from one metal to adjacent metal. Carbon enriched zones are hard zones whereas carbon depleted zones are soft zones. Generally, mechanical and metallurgical properties are degraded by carbon migration. Across the narrow zones significant changes occur because of soft and hard zones present adjacent to one another. In general oxidation occurs and due to local stresses cause the oxide to crack.

Metallurgical Deterioration:

In the weldment region degradation of mechanical properties occurs as because of carbon migration. During carbon migration soft zone was left behind from where carbon is migrated and hard zone is created where carbon is deposited. Low tensile and creep strength was exhibited by carbon depleted zone whereas due to the formation of phases and some hard phase's carbon enriched zone shows higher in hardness. Due to the change of properties to adjacent side is considered as unfavourable and thus mismatch in mechanical and metallurgical properties. Thus heterogeneous and complex microstructure are projected in these regions. Sometimes there is a mismatch of crystal structures also projected to failure.

Fixture for force balancing offsetting of tool for heat control

Generally, fixture is made up of mild steel having less heat dissipation. Also, it is quite difficult to offset the tool and fix the tool in the centre of the plates. Due to different thermal expansion, we need offset toward copper side because Cu-Cr-Zr has high thermal expansion than SS 304. Due to offset in Cu-Cr-Zr, the mixing increases in the region but it is quite difficult to maintain same offset along full length.

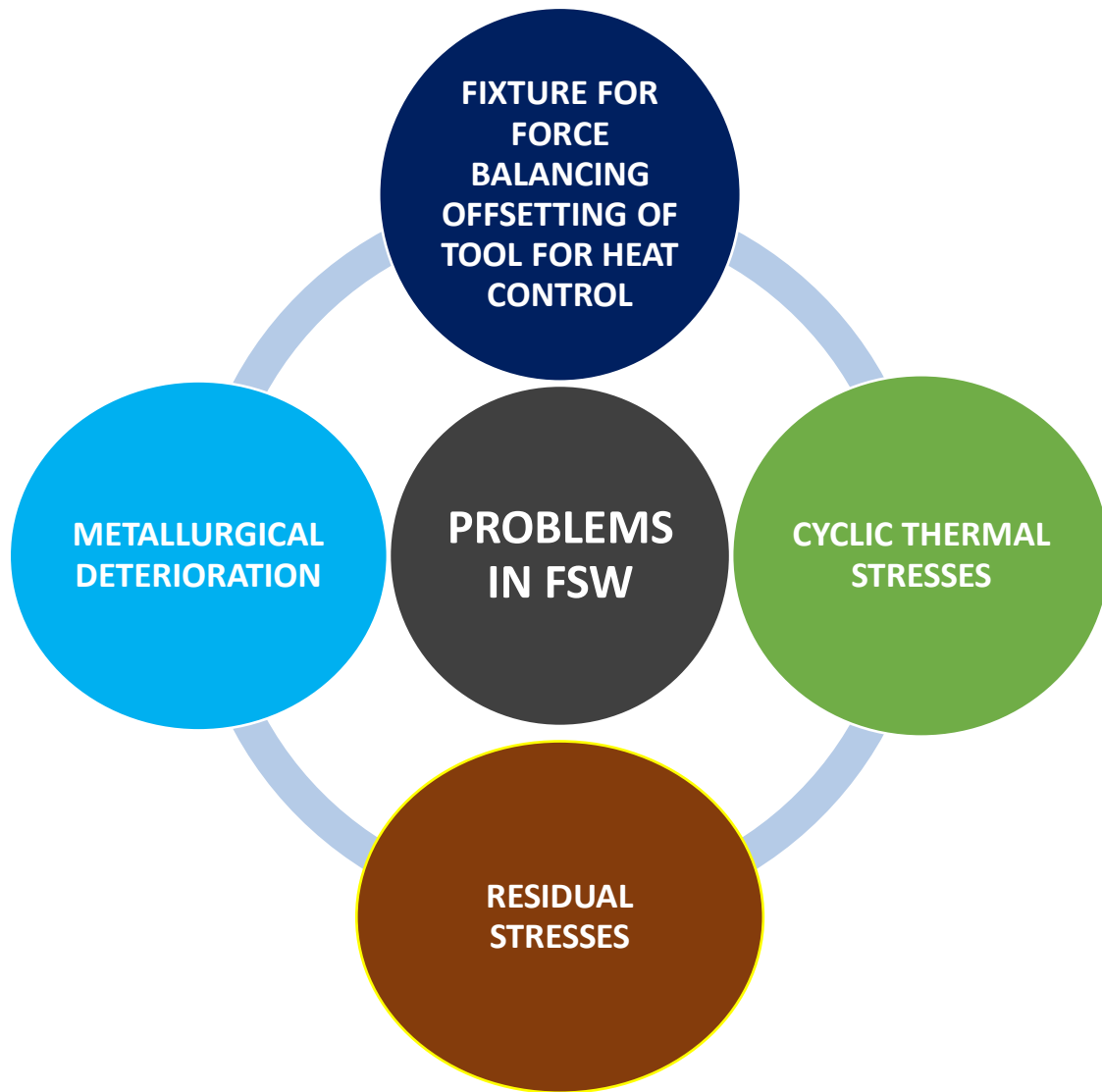


Fig: 3.1 Problems in FSW

3.2 Techniques to resolve problem

In adjacent zones formation of hard and soft zones causes major problems by changing the mechanical properties. The intensity of failure due to carbon migration is higher as compared to other factors.

Intermediate butter layer to control carbon migration:

As a filler metal, Nickel is added to the hard side of the metal also called as buttering. Generally Nickel content alters carbon solubility thus 2 to 3mm thickness of layer is sufficient. Due to buttering diffusivity changes and carbide become less stable. In nickel base filler metal also contains chromium (18-21%) and carbide such as Niobium and tantalum (3-4%). Carbon shows high affinity towards chromium and chromium considered as strong carbide former. The 'graded' transition joint or spool piece which feasts out the disparity effect can be used to control carbon migration. In dissimilar weld cladding formation of soft and hard zones are controlled by nickel base interlayer. The rate of formation of soft zone become slow due to formation of carbide by employing one or more intermediate buffer layer.

Welding processes and consumable variations:

Thermal cycle consists of rapid rise in temperature to thousands degree Celsius during welding followed by rapid cooling to ambient temperature. In weld metal there is a change in the micro structure and in heat effected zone in base metal is dependent on heating and cooling rate. These all are dependent on heat input which is a function of arc energy, type of welding process, travel speed and efficiency. In the work piece the nature of heat flow is complex and non-linear conductive due to localized intense heat input. Different welding processes requires different nature of heat flow. Temperature gradient as well as carbon activity is affected by varying nature of flow heat during rapid heating and rapid cooling of work piece.

4.1 Vertical milling machine/ Friction stir welding machine

We can perform friction stir welding on vertical milling machine (HMT FN2V). Rotational speed, feed, speed can be varied according to the material. We are using vertical milling machine because of its stability and low vibrations and features of varying parameters. Fixture is mounted on the vertical milling machine .Thus first step is proper testing of machine , fixture ,tool and providing proper lubrication to the machine in order to avoid vibrations.



Fig 4.1 Vertical milling machine



Fig 4.2 Modes in machine



Fig 4.3 Feed and speed parameters

4.2 Preliminary testing

In order to check the machine efficiency and to optimize various parameters preliminary testing of specimen is required. If any type of failure detected must be corrected before final welding. Greasing and oiling of vertical milling machine is required in order to protect machine from unwanted vibrations. According to the size of tool, proper collet is chosen and in order to transmit all forces exerted by tool to spindle a support is made of same diameter as that of tool. Aluminium plates are chosen for preliminary testing because aluminium is considered as best metal to join with friction stir welding because at 300-350°C temperature and 600-1000 rpm can join easily. Two aluminium plates of thickness 3mm and length of 50mm are taken and friction stir welding process performed in it. At different parameters success is achieved and both plates are welded through this process.

Preliminary testing is required in order to check machine efficiency, defects, vibrations, spindle check and to check working conditions. If any defect arises in any stage of preliminary testing, must be corrected.

- **Vertical milling machine test:** Machine is tested by free running of the machine for few minutes and check any type of vibrations.
- **Fixture testing:** Fix the fixture with the bed of the machine check any type of clearance defect or vibrations.
- **Spindle rotation:** Fix the tool in the collet thus check its rotation about the axis.
- **Tilting angle:** In friction stir welding, tilting angle is generally 2-3 degree. The reason is that, it helps in the flow of material in inwards direction. Thus proper setting of angle is required.



Fig 4.4 Setup



Fig 4.5 Before welding



Fig 4.6 After welding



Fig 4.7 Welded plates

4.3 Preliminary investigation carried to decide pathway

- Process parameters are established
- Vibrations are detected thus proper maintenance required.
- Can be used for hard materials.
- Vertical milling machine is suitable for friction stir welding.
- Tool supporter is needed in order to transfer tool load because tool has no contact to the spindle.

Tool supporter/ load transfer:

There is a gap between the tool in collet and spindle .Thus tool can be damaged if load is not transferred to the spindle. Thus a support is required of enough much strength that can transfer the load to spindle without breakage.



Fig 4.8 Tool



Fig 4.9 Tool in collet



Fig 4.10 Gap remain in collet after tool insertion



Fig 4.11 Tool support that transmits load to spindle

4.4 Requirement of backing plate

It is used as a platform for friction stir welding. In order to avoid any deformation /defect of fixture from intense heat backing plate is required. Due to sudden high temperature , sometimes fixture welds with the work piece thus to prevent from any damage a backing plate is used made up of mild steel having thickness of 10mm. Backing plate is grinded by both sides in surface grinder in order to get smooth surface finish. Smooth surface finish is required to reduce unevenness thus to get welding accuracy.



Fig 4.12 (a) (b) Backing plate from different angles



Fig 4.13 Backing plate weld with work piece

4.5 Hit and trial solution

8mm thickness and 50mm width of mild steel backing plate is taken but due to sudden rise in temperature gets welded to the work piece thus defect arises. In second trial, 10mm thickness and 50mm width is taken and thus results in defect free welding.

Screw supports:

A support is required in order to push backing plate and aluminium plates in downward direction thus there will be no movement of the plates during friction stir welding process. Four screw plates are made and joined in four corners and thus tightened by nuts.



Fig 4.14 Screw supports

4.6 Selection of material

Due to high electrical conductivities of copper alloys usage increases rapidly in industries. Generally conventional copper alloys have high electrical conductivity but limited strength. Stainless steel have good corrosion resistance and having higher strength. Thus joining these dissimilar metal become technically important. Cu-Cr-Zr and SS304 are excellent alloys possesses desirable combination of high strength and excellent electrical conductivity. It is very difficult to join copper alloy to stainless steel SS304 because of larger difference in their physical properties. Due to differ properties it is clear fusion welding would not be appropriate thus solid state joining techniques like friction stir welding and diffusion welding expected more consideration. Different chemical composition and thermos physical properties makes it quite difficult in joining. Additionally they easily forms intermetallic phases and deteriorate the mechanical strength of the joint. Most widely used non-magnetic, austenitic stainless steel is SS304. Another name of SS304 is 18/8" stainless steel because of the composition .it contains 18 percent chromium and 8 percent nickel. This alloy has good forming and welding properties

as well as strong corrosion resistance and strength. The ultimate tensile strength of Cu-Cr-Zr is more than 530MPa whereas ultimate tensile strength of stainless steel SS304 is 505Mpa. Due to good balance of electrical conductivity and strength can be used in international thermos nuclear experiment reactor.

Table 4.1 The chemical composition of Cu-Cr-Zr and SS 304

| Analysis no. | C | Mn | P | Cu | Si | Cr | Ni | Zr | Fe |
|---------------------|----------|-----------|----------|-----------|-----------|-----------|-----------|-----------|-----------|
| Cu-Cr-Zr (%) | - | - | - | Rest | - | 1 | - | 0.8 | - |
| SS 304(%) | 0.08 | 2 | 0.045 | - | 0.75 | 18 | 8 | - | 67-71 |

5.1 Materials and Methods

In this study dimensions of SS304 and Cu-Cr-Zr plates are 170 mm× 50mm ×30mm.

5.2 Experimental setup

Fixture:

Main function of fixture is to hold the metal plates in positions that can bear the load and pressure exerted during friction stir welding process. Fixture is designed in such a way that can holds up to very high temperature developed during friction stir welding. The fixture should be such that it should withstand the forces and rising temperature during welding processes without distortion in shape. Various clamps are provided in it in order to prevent specimen from any motion while being welded. The quality of weld is also dependent on stability of fixture.

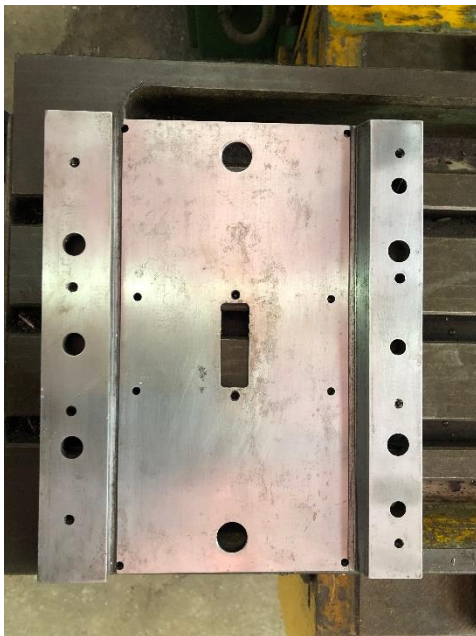


Fig5.1 Front side of fixture plate

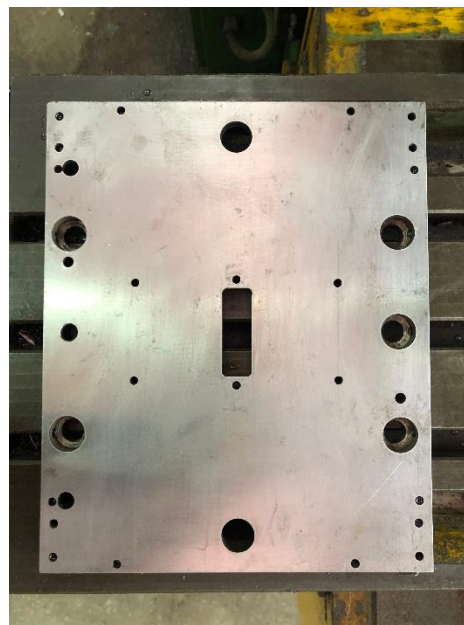


Fig5.2 Back side of fixture plate



Fig5.3 Supporting sides of fixture



Fig5.4 Assembly of fixture

5.3 Influence of tool material geometry on weld quality

Mainly it carries two parts, a probe and body. Another name is given to the tool technology, heart of friction stir welding. The heating, forging pattern of weld element and plastic is dependent on the tool shape and geometry. The determination of tool strength, weld size and welding speed is based on tool shape. Working temperature and rate of heat produce is also dependent on tool material. In case of the copper alloys and stainless steel circular shaped tool profile is suited for the best results.

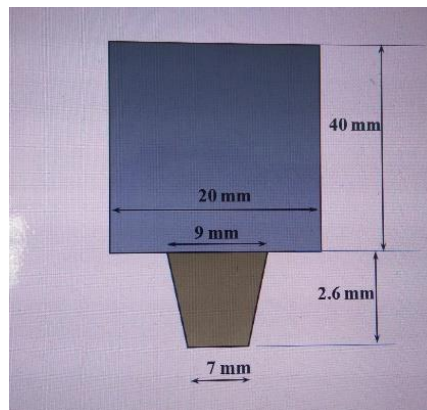


FIG 5.5 Tool dimensions and design



Fig 5.6 Tungsten carbide tool

5.4 Tool material selection

The selection of material generally depends on the hardness of material and thus the wear rate of the tool. In case of aluminium, it is nonmagnetic, silvery white, soft and ductile metal. Due to such properties, tool wear is very less thus tool steels can be used. If we compare it with high melting point materials such as stainless steel and copper alloys, such tools are not worthy.

The thermal conductivity and tool material indicates the rate of heat removal. Flow stress, microstructure and temperature field is affected by this. At high temperature wear rate of tool increases thus we required tool that can withstand up to 1800 degree Celsius. Tungsten carbide tools has a success history of wear resisting and a good combination of high hardness and high fracture toughness. Carbide tool has an advantage of having low coefficient of friction between tool and the work piece.

5.5 Various tools can be used in friction stir welding [26]

Polycrystalline cubic boron nitride: Skeletal matrix is formed by the combination of small cBN particles and cBN is the second hardest material after diamond having a high hardness number on pcBN (>30GPa). Main feature is to maintain high hardness at high temperature. pcBN has high thermal resistance at higher temperatures. Generally, this tool has 80 to 90(%) of pcBN content and others metallic binders are used and result to higher fracture toughness. Tool have higher diffusion wear if pcBn content is low. On the other hand, the tool of pcBN is very expensive and not suited in budgeted projects.

Tungsten carbide: melting point temperature up to 2900°C and high hardness of 27GPa at room temperature makes it favourable to the friction stir welding. However, tungsten carbide is brittle in nature and possesses 2-4Mpa.m^{1/2} fracture toughness. To intensify further properties i.e. mechanical properties and sinter ability another material such as metallic binders, ceramic binders, cubic boron nitrides.

Ultra high temperature ceramics: Transition metal diborides (TiB_2 , ZrB_2 , HfB_2 , etc.) considered as ultra-high temperature ceramics as they have melting temperature above 3000°C .

Tungsten Rhenium (W-Re): the advantage of adding rhenium to tungsten is **conversion of** ductile to brittle transition temperature. The addition of rhenium not only affects transition temperature but also effects the ductility above transition temperature.as the rhenium content increases, the ductility also increases.

Table 5.1 Mechanical properties at ambient temperature

| Material | Hardness (GPa) | Fracture toughness[$\text{MPa}\cdot\text{m}^{1/2}$] | At high temperature |
|-----------------|----------------|---|--|
| W-Re | 7 | - | unknown |
| pcBN | 23-27 | 4-6 | Remains required hardness and strength |
| ZrB_2 | 15-23 | 3-4 | Ultra high temperature ceramics |
| Ti B_2 | 25-27 | 6-8 | ultra high temperature ceramics |

5.6 Welding procedure

The experimental study of friction stir welding includes firstly butt joining of $170 \times 50 \times 3$ mm plates to set proper parameters of the machine. After setting the parameters, observe the alignment of the plate's weather fully straight or not if it shows some alignment withdraw all the plates and check any irregularities in base of the fixture. After resolving all irregularities place the plates so that middle of the tool and the adjoining boundaries of the plates are co-centric.

5.7 Tilt angle

Tilt angle of 2 – 3 degree is given by tilting the spindle head of vertical milling machine.it exerts inward force to the flowing metal and push metal to flow in downwards direction.

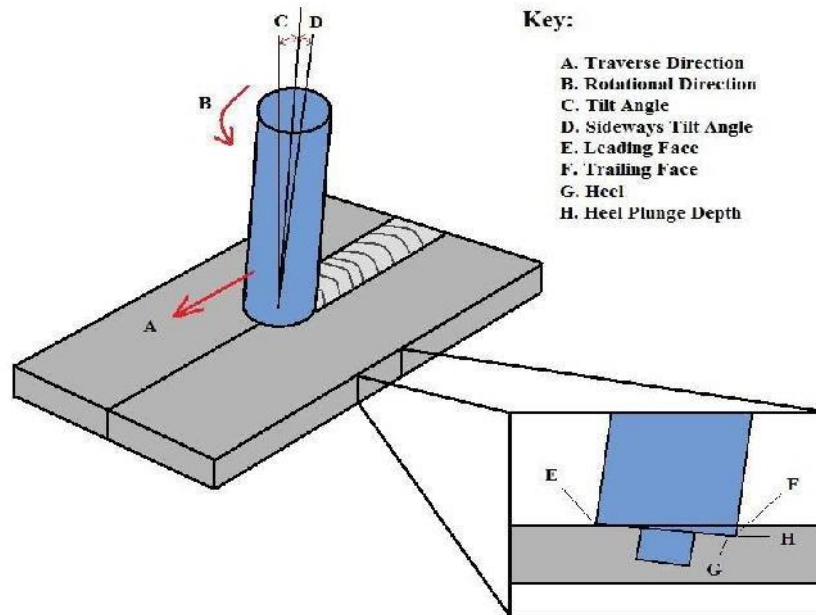


Fig 5.7 Diagram of FSW Tool Terms (Threadgill, 2007)

For aluminium plates preliminary investigation

Table 5.2 Process parameters

| | Feed (mm/min) | Angle (degree) | N (rpm) | Tool No. | Material | Dimensions |
|-------------|--------------------------|---------------------------|--------------------|---------------------|--------------------|-------------------|
| Exp. | 50 | 2 | 900 | 20 | SS304/Cu- Cr-Zr | 170mm×50mm×3mm |

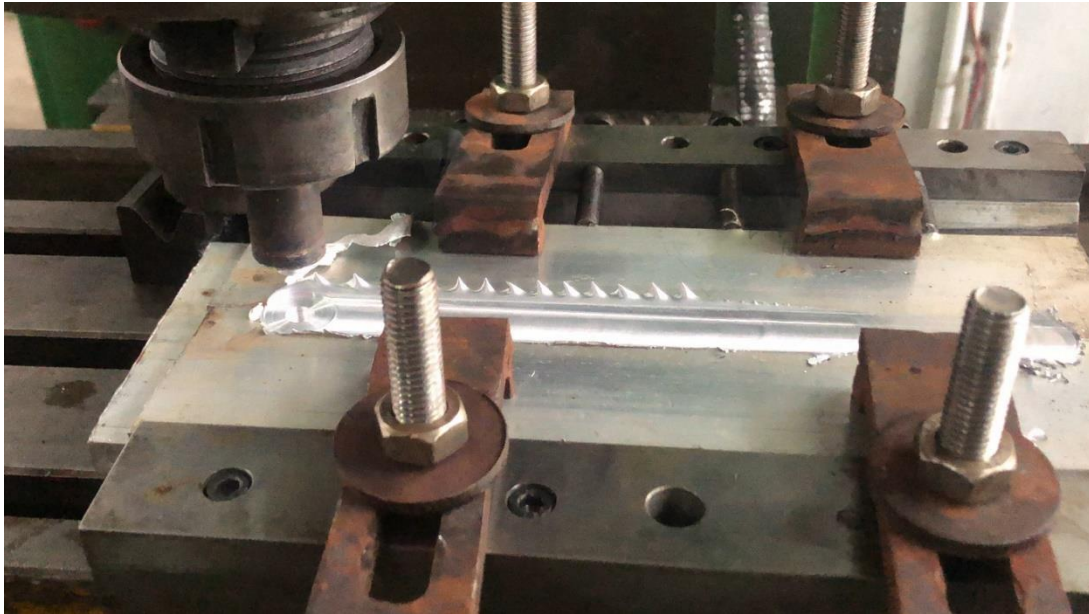


Fig 5.8 Friction stir welding process of aluminium plates at 2 degree tilt angle



Fig 5.9 Friction stir welding of SS 304/Cu-Cr-Zr at 2 degree of tilt angle

Experiment 01:

Cu-Cr-Zr plate was placed in the advancing side

Table 5.3 Parameters for friction stir welding

| | Feed (mm/min) | Angle (degree) | N (rpm) | Tool No. | Material | Dimensions |
|-------------|--------------------------|---------------------------|--------------------|---------------------|--------------------|-------------------|
| Exp. | 50 | 2 | 900 | 20 | SS304/Cu- Cr-Zr | 170mm×50mm×3mm |

Result:

Defects in welding due to less intermixing of material.



Fig 5.10 Defect in welding at 900 rpm

Experiment 02:

Cu-Cr-Zr plate was placed in the advancing side and speed increases to 1200 rpm.

Table 5.4 Process parameters

| | Feed (mm/min) | Angle (degree) | N (rpm) | Tool No. | Material | Dimensions |
|-------------|--------------------------|---------------------------|--------------------|---------------------|--------------------|-------------------|
| Exp. | 50 | 2 | 1200 | 20 | SS304/Cu- Cr-Zr | 170mm×50mm×3mm |



Fig 5.11 Defect in welding at 1200rpm

Result: Defect in welding due to intermixing

Experiment 03:

SS 304 plate was placed in the advancing side

Table 5.5 Process parameters

| | Feed (mm/min) | Angle (degree) | N (rpm) | Tool No. | Material | Dimensions |
|-------------|--------------------------|---------------------------|--------------------|---------------------|--------------------|-------------------|
| Exp. | 50 | 2 | 1200 | 20 | SS304/Cu-Cr- Zr | 170mm×50mm×3mm |

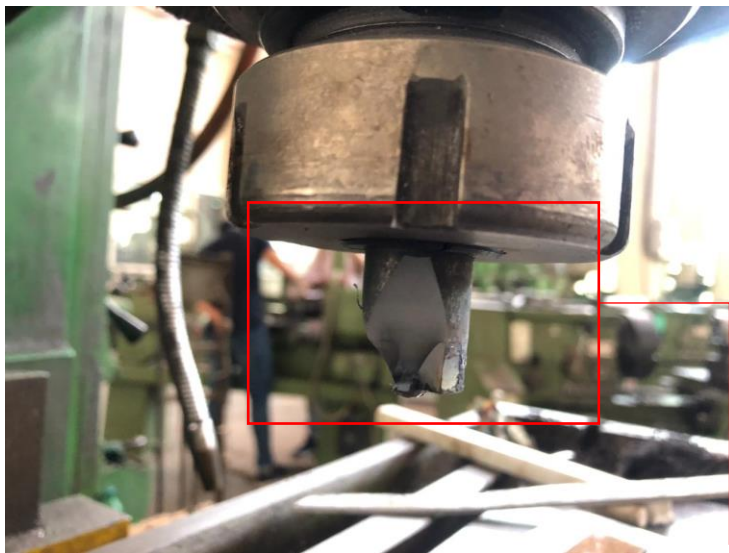


Fig 5.12 Tool failure

Result: Failure of tool due to high load and low speed.



Fig 5.13 Tool crack due to impact force



failure

Fig 5.14 Tool failure

When the tool is plunged between the plates , An upward force acts on the tool which is very high. Any type of irregularities result in tool failure.

Experiment 04:

Cu-Cr-Zr was placed in advancing side

Tool dia decreases to 16 mm

Rotational speed changes to 1400 rpm

Table 5.6 Process parameters

| | Feed (mm/min) | Angle (degree) | N (rpm) | Tool No. | Material | Dimensions |
|-------------|--------------------------|---------------------------|--------------------|---------------------|---------------------|--------------------|
| Exp. | 50 | 2 | 1400 | 16 | SS304/ Cu- Cr-Zr | 170mm×50m m×3mm |

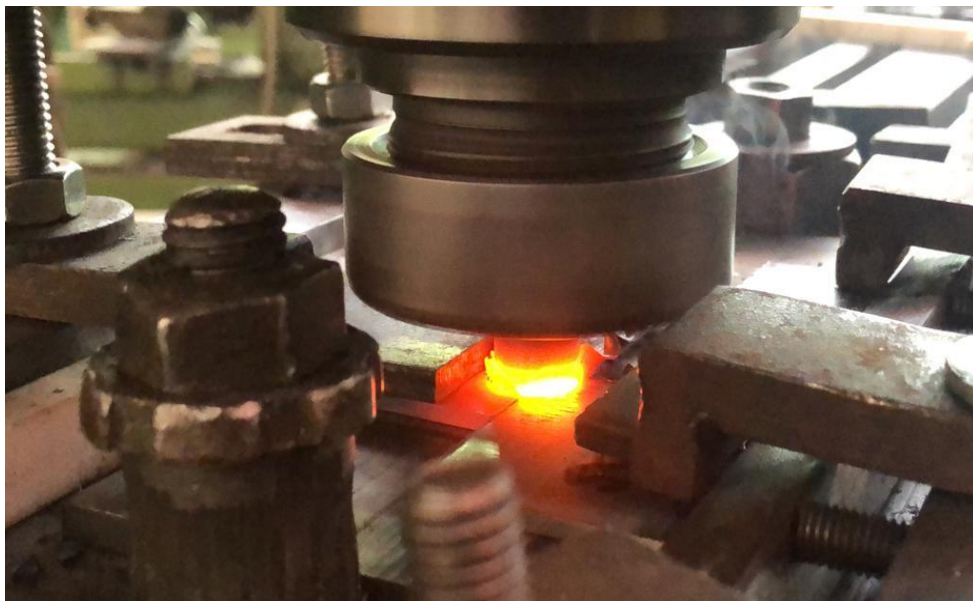


Fig 5.15 High heat generation causes tool failure

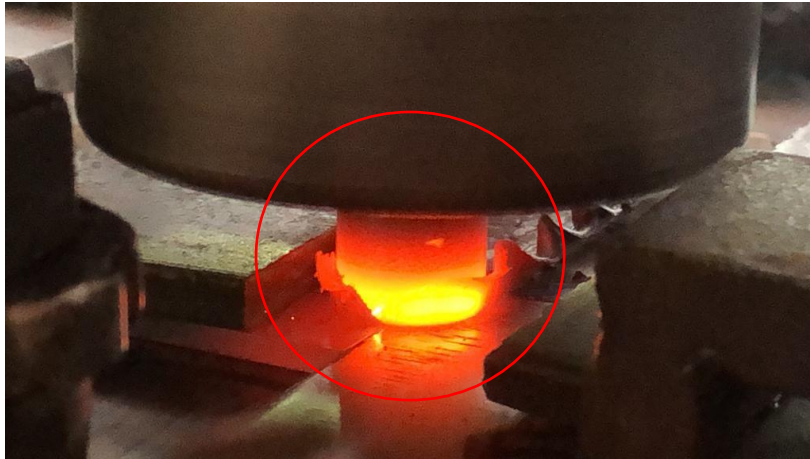


Fig 5.16 Tool pin damaged grinded due to high temp/pressure/forces



(a)



(b)

Fig 5.17 Tungsten carbide tool



Fig 5.18 deformation in tool due to heavy heat and force produced



Fig 5.19 Distorted tool pin

Experiment 05:

Cu-Cr-Zr plate was placed on advancing side

Tool diameter to 15 mm

Rotational speed kept 1400 rpm

Table 5.7 Process parameters

| | Feed (mm/min) | Angle (degree) | N (rpm) | Tool No. | Material | Dimensions |
|-------------|--------------------------|---------------------------|--------------------|---------------------|--------------------|-------------------|
| Exp. | 50 | 2 | 1400 | 15 | SS304/ Cu-Cr-Zr | 170mm×50mm×3mm |

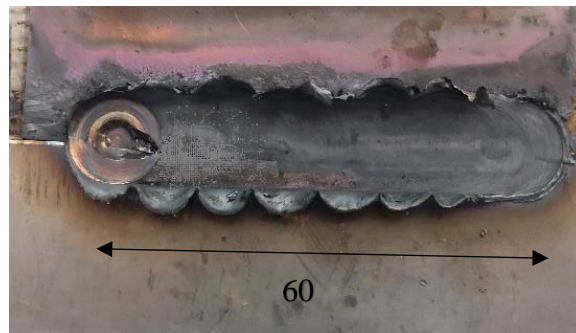


Fig 5.20 Continuous welding

Result: At 1400 rpm , Feed rate of 50 mm and having tool diameter of 15 mm the Cu-Cr-Zr and SS 304 plates are welded.

6.1 Micro hardness testing

Another name of micro hardness testing is indentation hardness testing. This process involves indentation at various loads up to 500 g and thus indentation depth ($70 - 100 \mu\text{m}$) gives the value of hardness at particular point in the specimen. Generally, Vickers hardness testing machine is used in it. In Vickers hardness testing machine a diamond indenter having square based pyramidal shaped. The ratio produced by diamond shaped indentation between the long and short diagonals is 7:1. With the help of optical microscope resultant indentation is measured. The optical microscope is calibrated as to the indentation size. Large load as possible is used in it. Test needs to be carefully controlled and strain free specimen is used in it. The diamond indenter is indented at maximum load and lowered slowly at rate of $<1 \text{ mm/min}$.

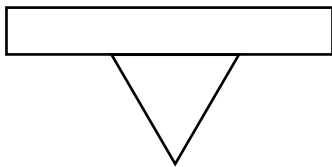


Fig 6.1 Diamond indenter

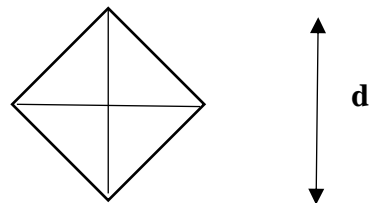


Fig 6.2 Pyramidal indentation



Fig 6.3 Vickers hardness tester

6.2 Cross weld tensile test

It is also called as ultimate strength or tensile strength of a material. Generally, it gives the relation between the loads and elongation. The capacity of material to tolerate loads tending to elongate. In simple words, tensile strength resists tension. While being pulled at maximum stress the material can withstand up to a value, called as ultimate tensile strength. Brittle failure occurs when material breaks sharply without undergoes any plastic deformation whereas when material experiences some plastic deformation, then the chances of ductile failure increases. Engineering stress and engineering strain graph is very helpful to know the characteristics of the material. In the stress strain curve highest point gives the value of the ultimate tensile strength. It is independent on size of specimen thus it is intensive property. A tension force is generated by applying a pulling force in opposite direction by vises. The specimen withstand to the ultimate tensile strength.



Fig 6.4 Cross weld tensile testing

6.3 Optical microscopy

Optical microscope is also called as light microscope. It uses visual light and the main function of the optical microscope is to magnify images of small objects by using a systems of lenses. A photosensitive camera is used to generate micrograph. Camera can produce digital images. Two types of optical microscope are available one is simple microscope and other is compound microscope. For magnification one uses only a single lens such as magnifying glass is called

as simple microscope. To enhance the magnification of the object several lenses are used is basically a compound microscope.

Components:

1. Eyepiece
2. Turret
3. Objective lenses
4. Focus adjuster (coarse and fine arrangement)
5. Stage (to hold specimen)
6. Light source
7. Diaphragm and condenser
8. Mechanical stage

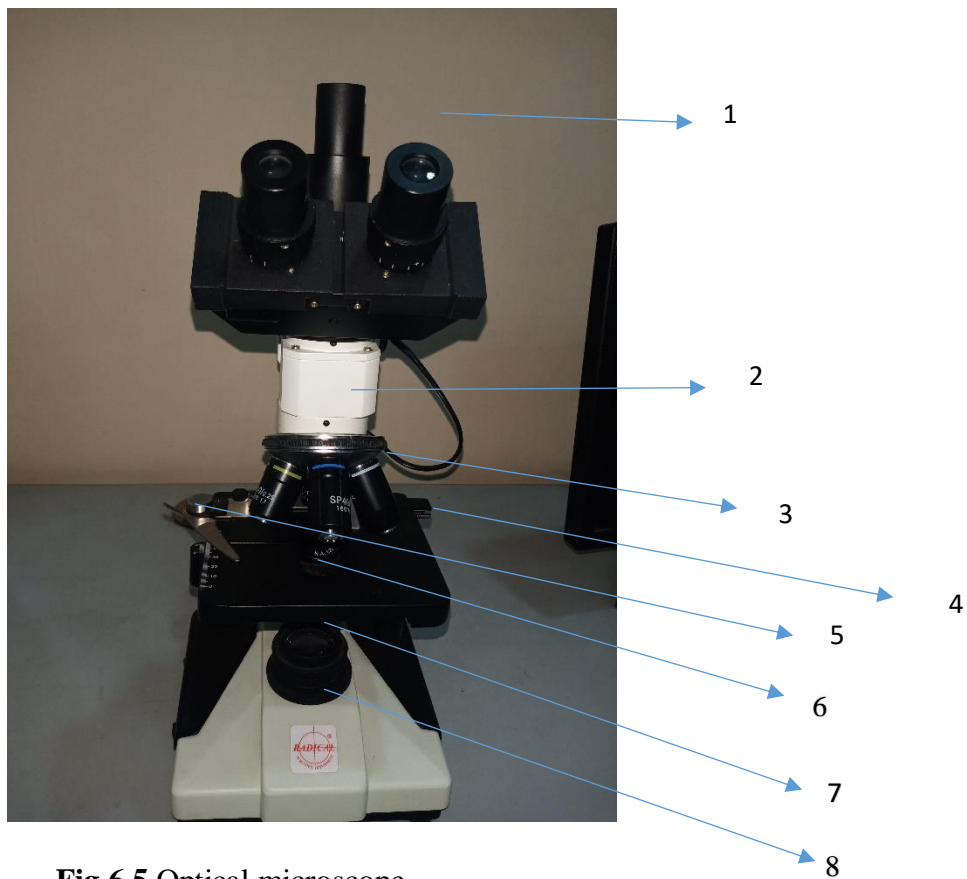


Fig 6.5 Optical microscope

6.4 Spectroscopy

This is generally called as electron spectroscopy. The study of the interaction between matter and electro-magnetic radiations called as spectroscopy. The investigation of composition, physical structure and electronic structure of matter at the atomic scale is done by spectroscopy. It refers to the measurement of radiation intensity as a function of wavelength. Elemental composition is denoted by the spectroscopy. A beam of electron or laser is incident on the Sample then reflect back according to the wavelength thus denotes the elemental composition of the alloy or metal. Machine is directly attached to the computer thus all the reading are shown in it.



Fig 6.6 Spectrometer

6.5 Scanning Electron Microscopy (SEM)

The image of the sample is produced by simple scanning of surface with a focused beam of electrons. In the sample electrons interact with the atoms produces signals that contains information regarding the surface topography and composition of the sample. In a raster scanned pattern the electron beam is scanned. The intensity of detected signal is combined with

the position of the beam to produce an image. Signal intensity depends on the topography of the specimen. Up to 1 Nano meter of resolution can be achieved by the Sem. The environmental conditions of SEM required high vacuum and elevated temperature.

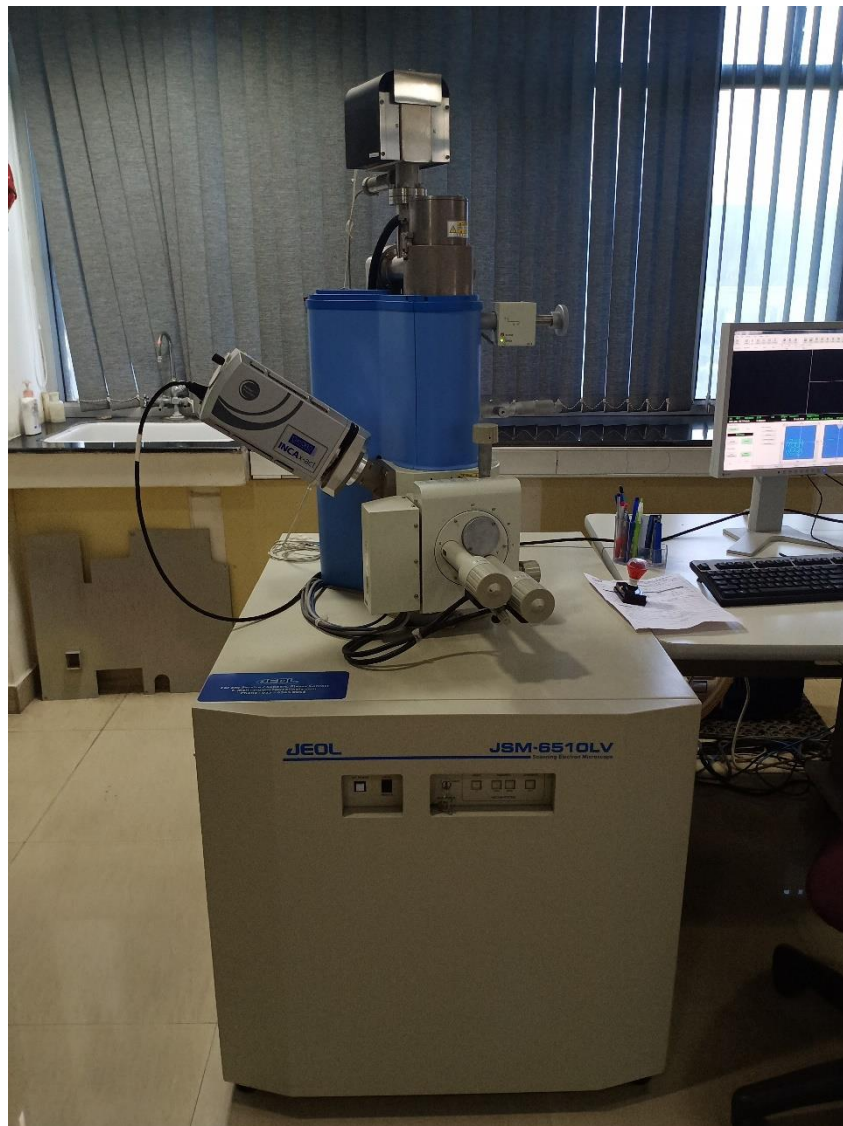


Fig 6.7 Scanning electron microscopy

6.6 Disc polisher

Disc polisher machine is used to polish the surface and reduce the scratches from the specimen. Disc polisher rotates at some required rpm. In disc polisher there are two types of discs where in one disc emery paper is placed to remove the pits by grinding the specimen (can use various sizes of emery papers) and second disc is coated with soft cloth for diamond polishing to obtain mirror like polishing. The rpm of machine is set according to the material of specimen. After polishing specimen is used to observe and measure metallography structure of specimen under microscope.



Fig 6.8 Disc polisher

6.7 Wire cut electric discharge machining

Wire cut Edm process is a metal machining process in which thousands of sparks are generated by tool instead of cutting the work piece electric discharge machining melts the work piece and vaporise it and thus provide accurate finish. Little debris is produced in it. We can also cut very hard materials from wire cut machining process. The wire cut EDM process uses a cathode tool that disperse the electric current, the work piece is anode through which cathode runs along the metal piece. Wire cut Edm equipment is a Computerised numerically controlled machine which can control wire in 3 dimensional axis. Cutting model is designed in the prescribed software at particular constant velocity depends on the shape, size and strength of the material. Generally copper wire is used in it because of its high electrical conductivity and availability. We can also re-use the copper wire until it totally destroyed.



Fig 6.9 Wire cut EDM

6.8 Surface grinder

A flat surface is produced with a smooth finish by grinding process through surface grinder. For abrasive machining process it is widely used. Material is removed from the work piece in form of chips when spinning wheel comes in contact with the work piece. The spinning wheel is covered with the rough particles. The composition of surface grinder contains an abrasive wheel, a work holding device and reciprocating table. Work piece is placed over the table as it is magnetic. Due to high magnetic strength it bears all the forces developed during grinding process. If a non-magnetic material is used then design of fixture is required. Material removal rate of surface grinder is in microns thus having good accuracy and precision. Due to the generation of spark i.e. heat coolant is also added during operation.



Fig 6.10 Surface grinder with magnetic chuck

6.9 Specimen extraction from welded region

From a welded plate sample is extracted by wire cut EDM process. The accuracy and precision of wire cut EDM machine is very high. Various zones are created in the weldment region and they are affected by heat input. Thus in order to protect from any distortion, wire cut EDM process is chosen. Wire cut EDM process does not produce enough much heat that can distort any sample because of submerged into water and use of high electric current.



Fig 6.11 Welded specimen

Hardness testing specimen Extraction

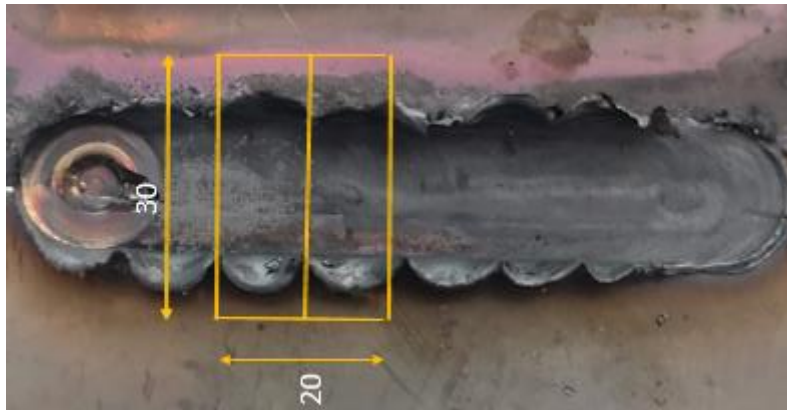


Fig 6.12 Drawing of sample extraction from specimen

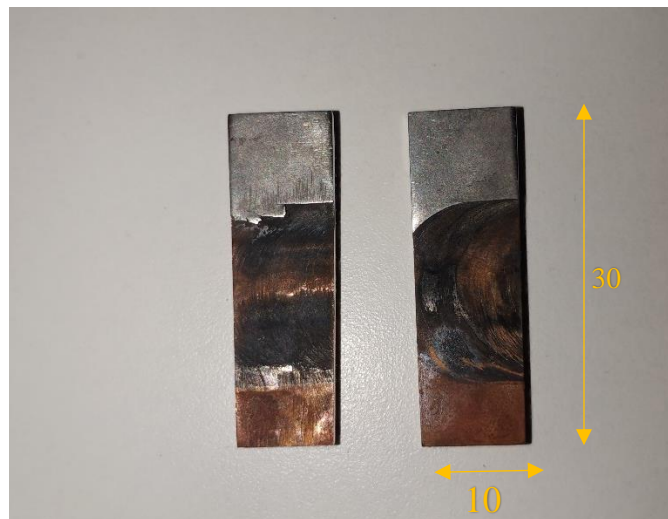


Fig 6.13 Top view of Samples extracted from wire cut EDM for hardness testing, SEM, Optical microscopy

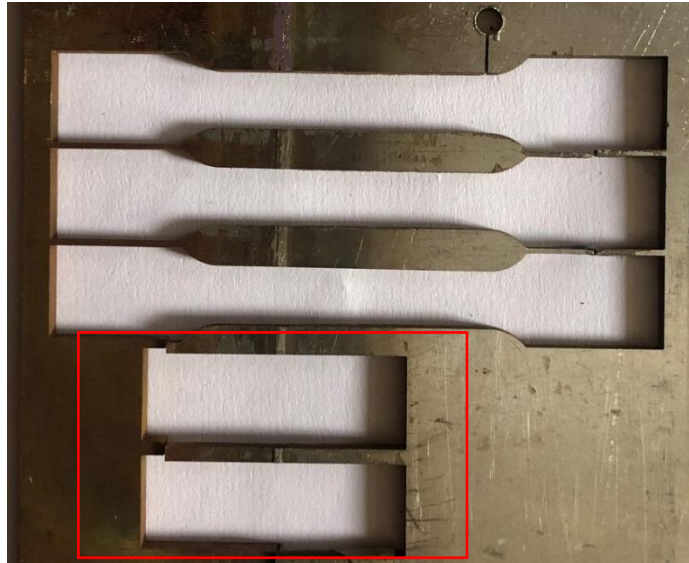


Fig 6.14 Skeleton of specimen extraction



Fig 6.15 Side view of hardness testing samples

Extraction of tensile testing samples

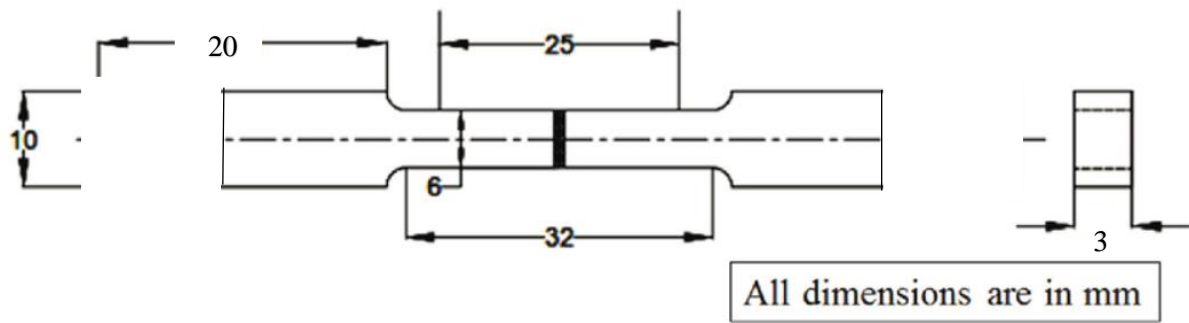


Fig 6.16 Dimensions of sample for cross weld tensile testing

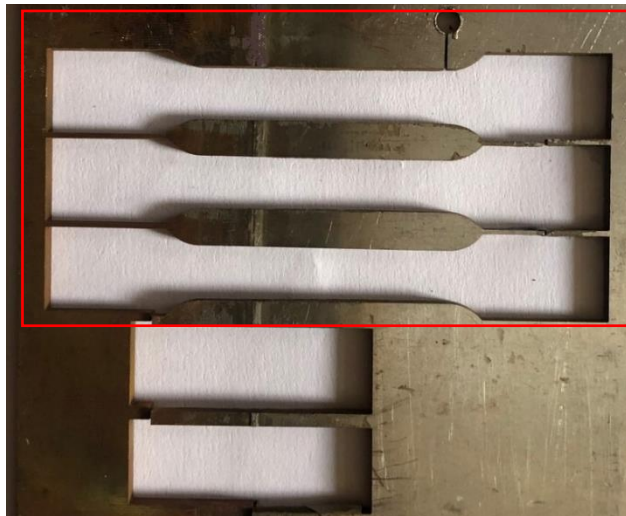


Fig 6.17 Skeleton of specimen extraction for cross weld tensile testing



Fig 6.18 Cross weld tensile testing specimen

7.1.1 Hardness testing

Irregularities of the sample surface is cleared by using emery paper. Set of emery papers are used in it starting from grade 200 to 2000. All type of pits, scratches and irregularities can be clear by this process and the sample is tested in particular Vickers's hardness machine at load of 500g.



Fig 7.1 Shows number of reading taken in direction from 1-15

Table 7.1 Vickers hardness reading by indentation process

| | SAMPLE 1 | SAMPLE 2 | SAMPLE 3 | |
|--------------|-----------|-----------|-----------|----------------|
| Margin 0.5mm | LOAD 500g | LOAD 500g | LOAD 500g | Dwell time (s) |
| 1 | 303.3652 | 323.7909 | 297.9453 | 20 |
| 2 | 292 | 300.1647 | 269.2945 | 20 |
| 3 | 339.811 | 302.4089 | 270.1104 | 20 |
| 4 | 298.9527 | 291.4331 | 251.2867 | 20 |
| 5 | 396.7825 | 293.5085 | 261.866 | 20 |
| 6 | 310.1469 | 275.0752 | 271.5526 | 20 |
| 7 | 347.8854 | 278.5601 | 295.7505 | 20 |
| 8 | 324.4832 | 343.6607 | 273.1278 | 20 |
| 9 | 329.4751 | 114.0528 | 300.1647 | 20 |
| 10 | 88.2208 | 100.5147 | 88.11805 | 20 |
| 11 | 94.63726 | 109.2511 | 95.88394 | 20 |
| 12 | 90.4779 | 93.39519 | 86.45809 | 20 |
| 13 | 91.05511 | 65.93422 | 94.63219 | 20 |
| 14 | 102.4661 | 110.0301 | 110.0301 | 20 |
| 15 | 90.4779 | 100.5147 | 103.3066 | 20 |

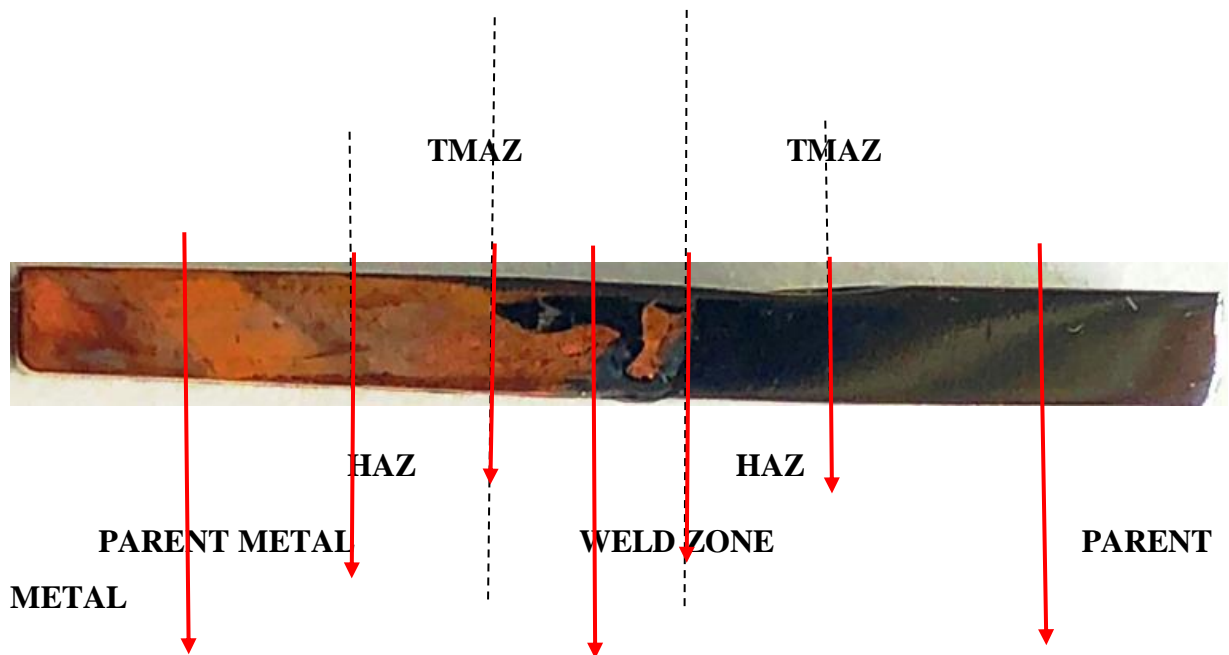
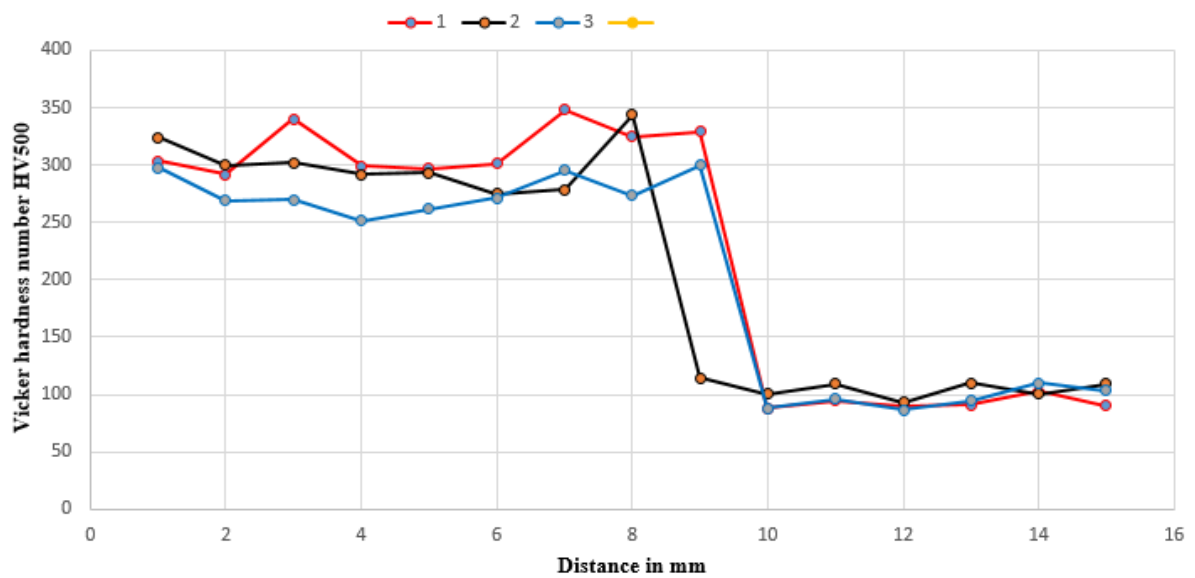


Fig 7.2 Different zones in welding samples



Micro Hardness variations across weldment regions

Fig 7.3 Micro hardness variations across the welding regions (red line indicates readings of sample 1, Black line indicates sample 2, blue line indicates sample 3)

7.1.2 Hardness analysis

The hardness variation the Cu-Cr-Zr side to SS 304 is shown in the Fig 7.3. In the welded condition, the stir zone showed highest hardness of about 347.88 HV₅₀₀. In the TMAZ of copper region there is a drop in the hardness value of about 94.63 HV₅₀₀ as calculated. In case of SS304 parent metal shows hardness of 297.94 HV₅₀₀ and as we move towards TMAZ of SS 304 it shows decrease in hardness of about 251.286 HV₅₀₀. Thus TMAZ of Cu-Cr-Zr has lowest hardness value among all regions. To summarize the variation in hardness, that is mainly due to higher temperature gradient in the TMAZ region effects the grains of the microstructure. Thus due to the grain refinement, it leads to change in the hardness value.

7.2 Macrograph of weldment

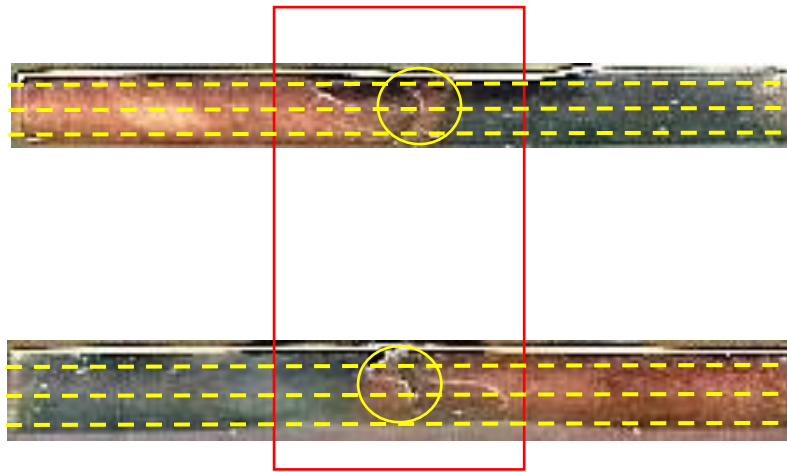


Fig7.4 Micrograph shows mixing zones

7.3 Spectroscopy results

It shows the elemental composition in the weld bead. In macrograph yellow point shows the area of the burn where elemental composition is noted as follows:

Table 7.2 Composition at the weldment

| Elements | Test 1 | Test 2 | Test 3 | Test 4 |
|----------|--------|--------|--------|--------|
| Fe% | 37.4 | 10 | 18.1 | 18.2 |
| C% | 0.552 | 0.467 | 0.383 | 0.345 |
| Si% | 3.14 | 1.06 | 1.41 | 1.12 |
| Mn% | 2.74 | 0.218 | 0.407 | 0.430 |
| P5 | 0.800 | 0.800 | 0.800 | 0.800 |
| S% | 0.150 | 0.150 | 0.150 | 0.150 |
| Cr% | 24.8 | 8.37 | 10.2 | 10.2 |

| | | | | |
|------------|--------|--------|--------|--------|
| Mo% | 0.0741 | 0.155 | 0.246 | 0.424 |
| Ni% | 4.69 | 1.99 | 4.17 | 8.11 |
| Al% | 0.256 | 0.104 | 0.0298 | 0.0310 |
| Co% | 0.112 | 0.451 | 0.152 | 0.248 |
| Cu% | 8.50 | 8.50 | 8.50 | 8.50 |
| Nb% | 0.141 | 0.0760 | 0.155 | 0.189 |
| Ti% | 0.169 | 0.0534 | 0.800 | 0.474 |
| V% | 0.125 | 0.0470 | 0.0664 | 0.0885 |
| W% | 6.06 | 4.87 | 1.54 | 3.01 |
| Pb% | 0.350 | 0.350 | 0.350 | 0.350 |

7.4 Cross-weld tensile test: It is weld qualification test

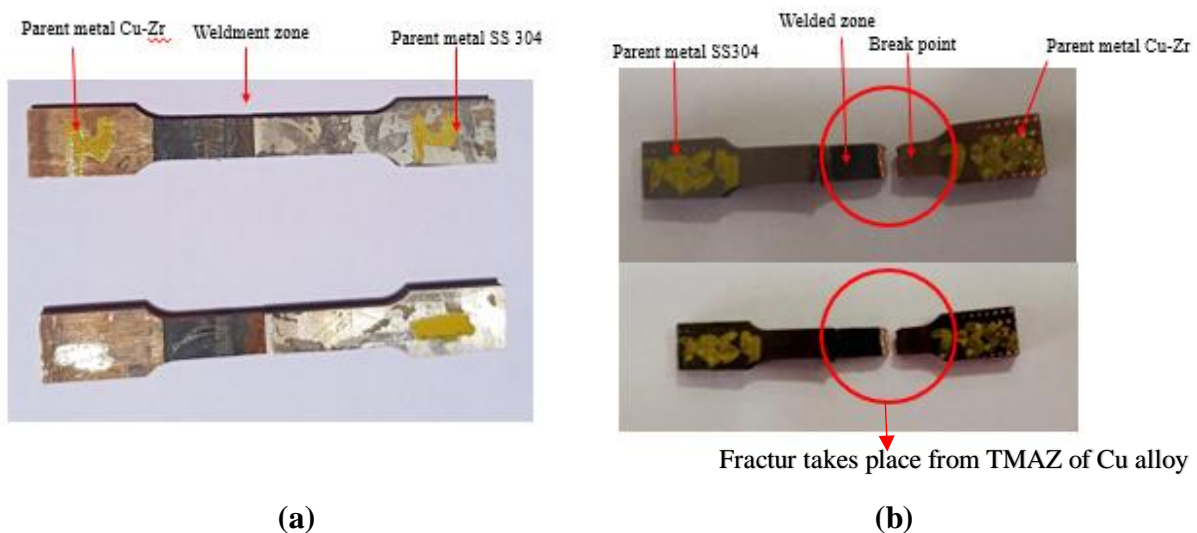


Fig: 7.5 (a) Cross weld tensile test specimen before test

(b) Cross weld tensile test specimen after test and its fracture location

7.4.1 Result of cross weld tensile test

The specimen is placed under tensile testing machine. As we can see from the figure, the specimen is break from the TMAZ of the Cu-Cr-Zr metal. It gives us the positive indication that our weld is strong enough as compared to the parent metal.

For first sample, at 3.734 KN/ 244 kNm⁻² the sample breaks into two pieces from the TMAZ of copper and at the force of 3.796 KN/ 222kNm⁻² second sample breaks. To note down the analysis of failure investigation is required.

7.5 FRACTOGRAPHY

To determine the failure structure and to know about the material properties at the fracture surface fractography is required. To obtain the metals behavioural nature with force, and to examine fracture at a finer scale fractography is required. It can show that the fracture occurs is due to impact forces or ductile forces.

To determine the fracture mode, fracture origin and crack direction close examination of the topology and fracture is required. For proper evaluation and classification of a fracture surface the scanning electron microscope plays an important role. For rough surfaces, SEM allows high resolution image at high magnification. Through SEM, ductile, brittle and intergranular fractures can be easily identified. Some typical features discussed follows: [29-34]

Ductile Failure

High amount of plastic deformation results in the fracture is generally considered as ductile failure. Forming distinct elongated dimples, initiation of voids or inclusions are considered in ductile fractures.

Brittle fracture

By two distinct methods brittle failure of material can occur. Across crystallographic planes, cleavage fractures occur creating ledges or steps often containing river patterns that allow the metallurgist to determine the direction of crack progression.

Intergranular fracture

Due to the influence of the phases or substances low energy failures occur along the grain boundaries (weaken the grain boundaries). Thus susceptible to crack propagation during loading.

7.6 Specimen extraction and results



Fig 7.7 Sample extraction for fractography (SEM)



Fig 7.8 Top, intermediate and bottom points in TMAZ (Cu-Cr-Zr) for fractography

Top surface: From where the tool is inserted and having maximum heat generated in the surface, due to the frictional force generated by the tool shoulders contact.

Intermediate surface: Generated heat flow occurs from top to bottom and thus where heat generated by pin is intermediate surface. Heat generation is less than top surface.

Bottom surface: Heat dissipation occurs through bottom surface. it transfers heat energy from material by conduction through the backing plate.

7.7 Results

The SEM results as follows

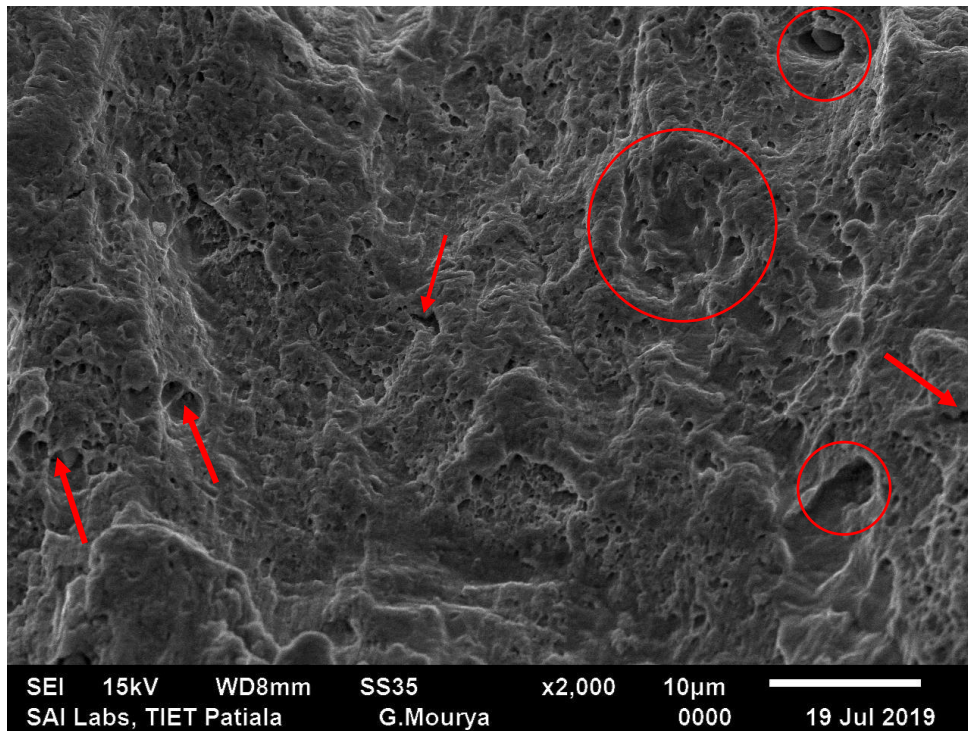


Fig 7.9 Top surface of sample 1 (Red arrows shows dimpled cleavage like structure)

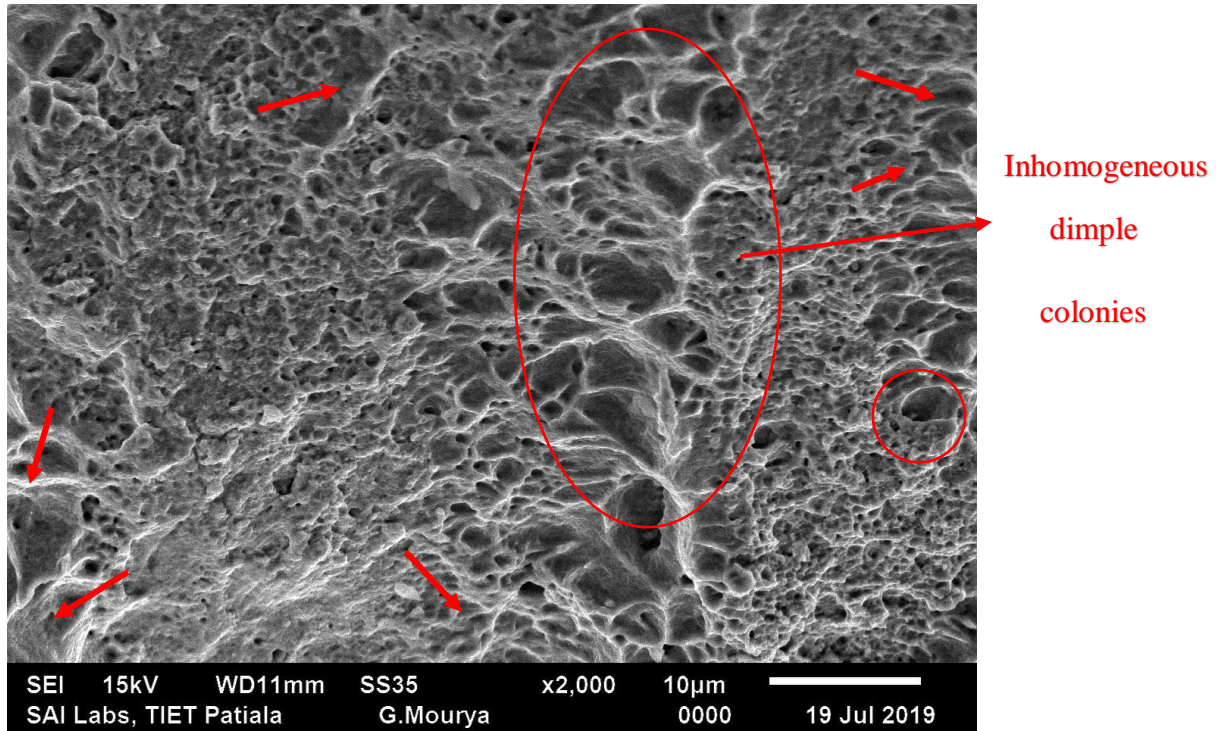


Fig 7.10 Top surface of sample 2(pure ductile failure) dimpled cleavages structure

RESULT:

Dimpled cleavage (tear like structure) indicates ductile failure.

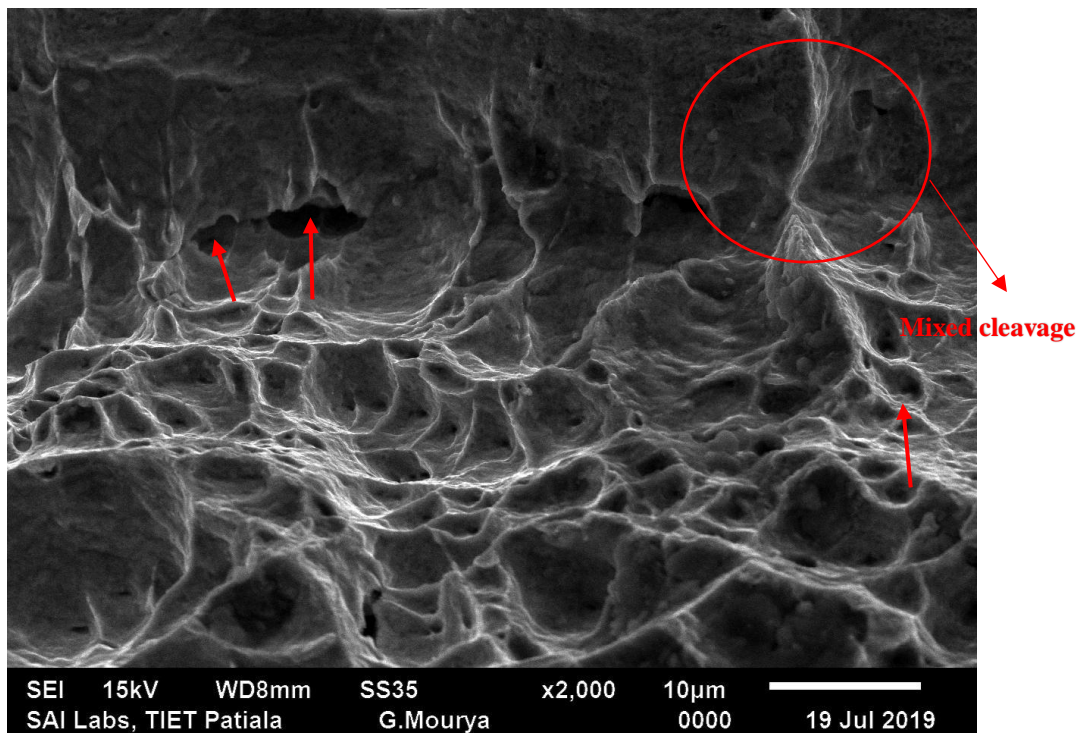
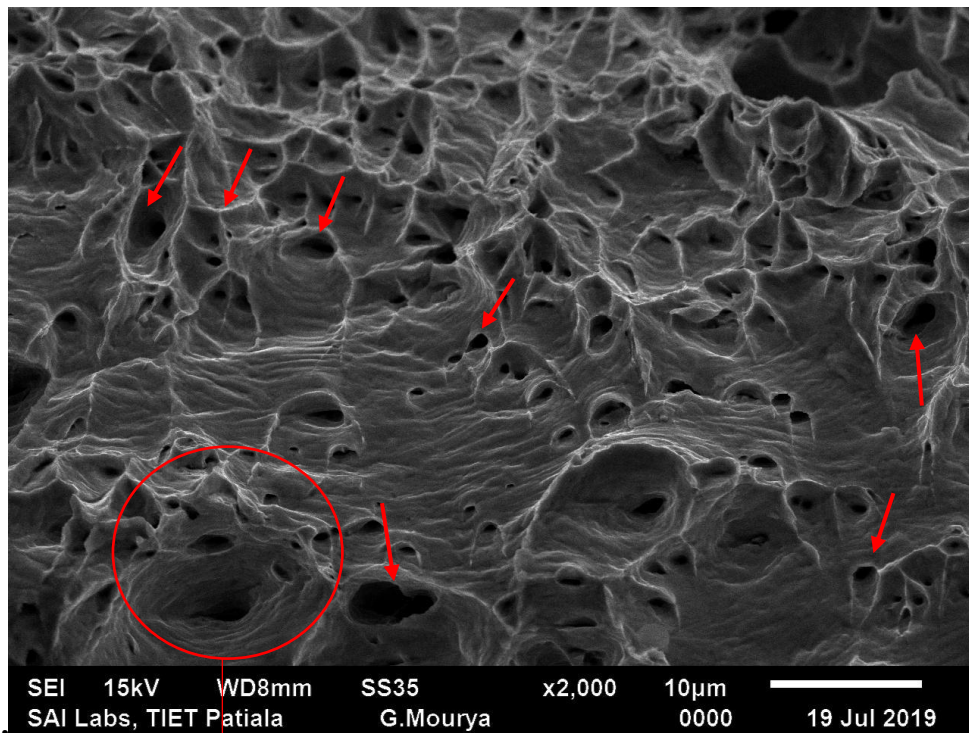


Fig 7.11 Middle surface sample (red arrows shows dimpled cleavage like structure)

RESULT: Red arrows indicates the dimpled cleavage (tear like structure). Thus the failure is ductile.



Inclusions nucleated voids

Fig 7.12 Bottom surface sample 1 (red arrow shows dimple like structure)

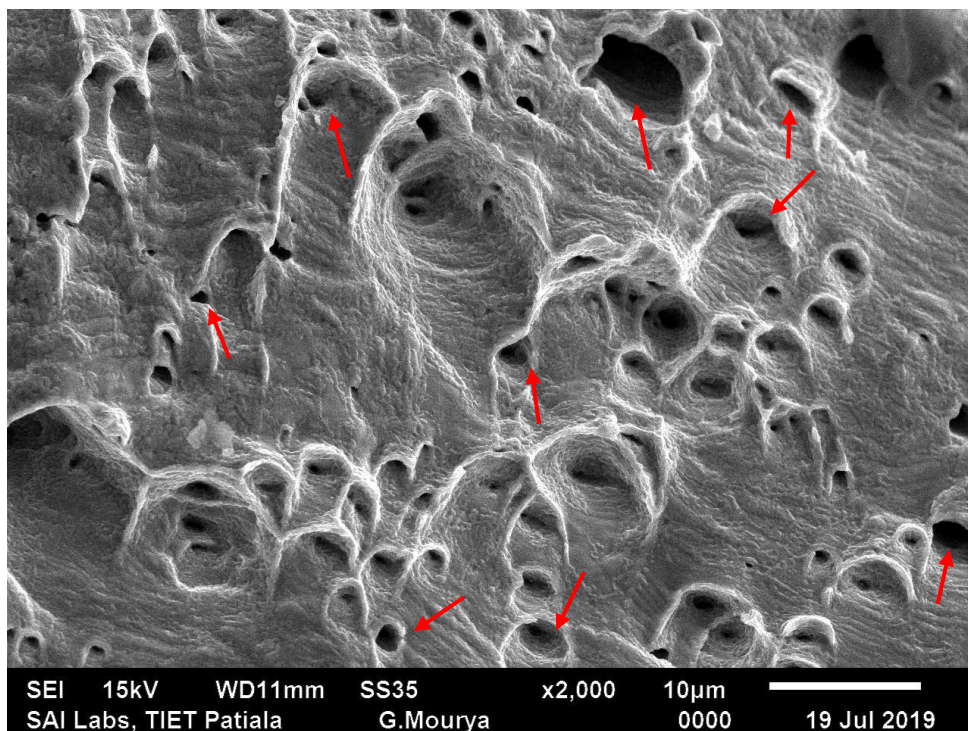


Fig 7.13 Bottom surface sample 2 (red arrows shows dimple like structure)

RESULT: Dimple structure shows ductile structure

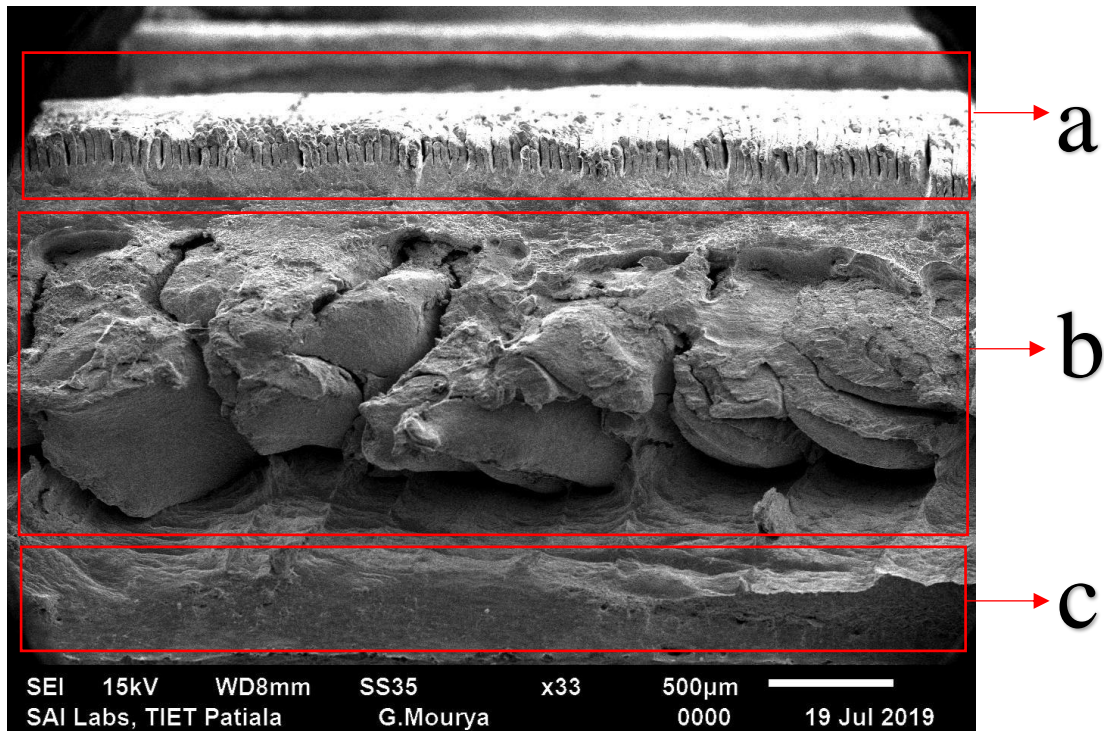


Fig 7.14 Lowest magnification full sample SEM of sample 1

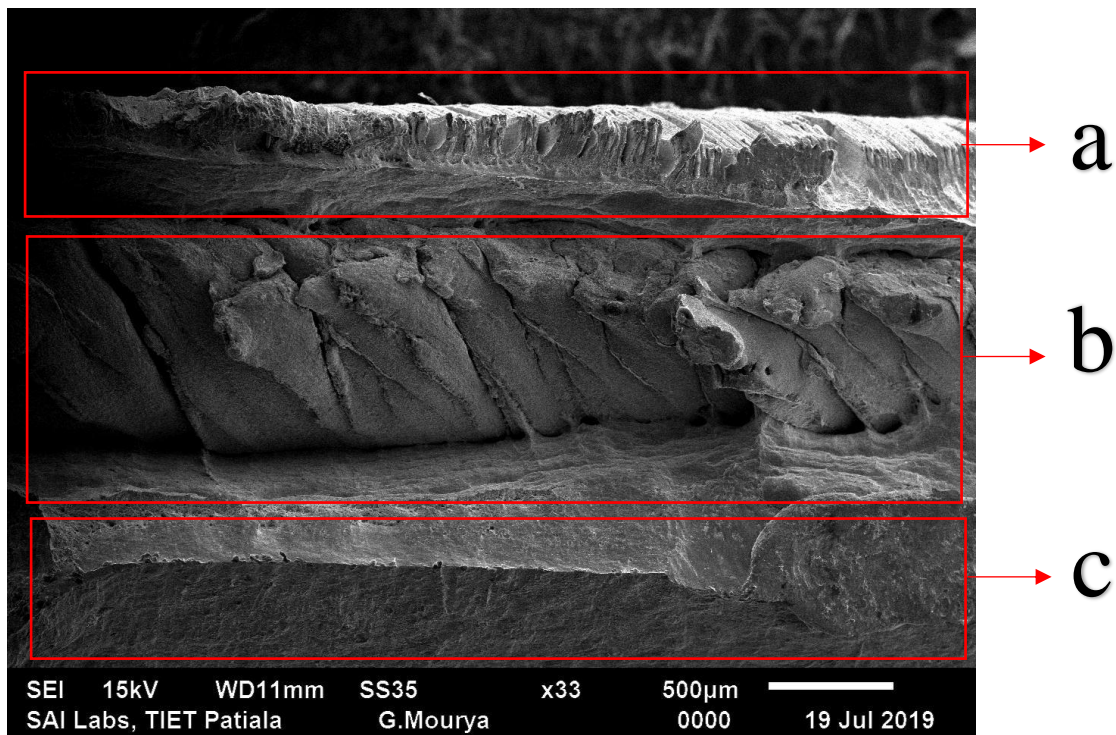


Fig 7.15 Lowest magnification full sample SEM of sample 2

7.7.1 RESULT

Low magnification of TMAZ region does not show any equivalent data for fracture analysis because at low magnification, general properties of fracture surface cannot be analysed, it shows only topographical properties. (Refer Fig 7.14, Fig 7.15)

(a) Clearly indicates that the shoulder of the tool is plunged up to “a”. Deeply penetrated serrations. Effect of high heat and surface contact with tool material.

(b) Represents the area where mixing occurs at very high temperature and due to thermal gradient changing in downwards direction. In this region, heat transfer through conduction (to backing plate) takes place in the downward direction.

(c) Region where low heat is produced as compared to region (a), (b).

In case of metallic material fracture surfaces, investigation is carried out according to their deformation of failure. Sliding or cleavage are the factors causes of the catastrophic failure. Ductile failure generates less serious problems than brittle fractures under operating conditions. When variable load at high amplitudes are applied then metallic materials are subjected to the fatigue failures. Fatigue failure is the combination of the brittle and ductile failures. SEM clearly indicates about the fracture type. [29-31]

7.8 Microstructure Analysis

Small scale structure of a material is called as microstructure. Microstructure of a material strongly influence the physical properties such as strength, toughness, ductility, corrosion resistance, hardness, wear resistance etc.



Fig 7.16 TMAZ Cu-Cr-Zr (Ultimate tensile break point)

Sample breaks from The TMAZ from the Cu-Cr-Zr thus it is necessary to analyse the TMAZ of Cu-Cr-Zr. Microstructure of Cu-Cr-Zr as follows:

Microstructures

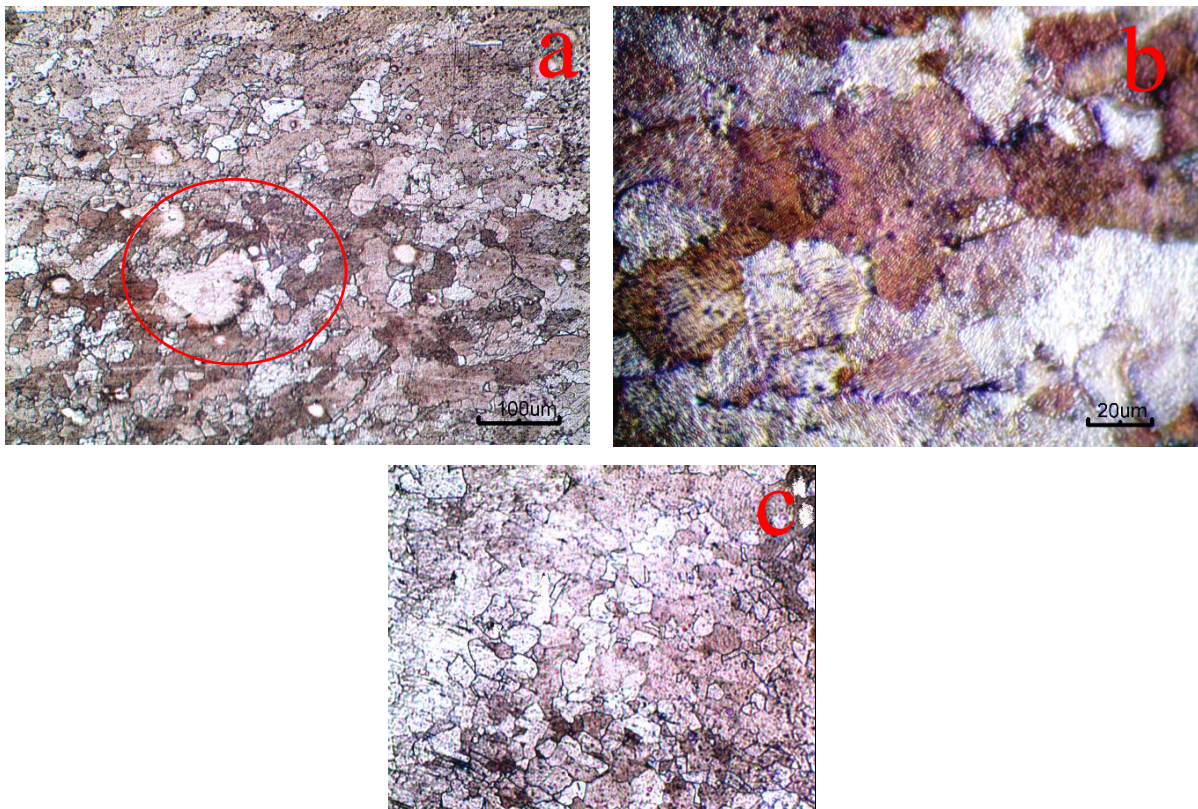


Fig 7.17 Microstructure of TMAZ of Cu-Cr-Zr (a 100X, b 400X magnification) and c pure copper

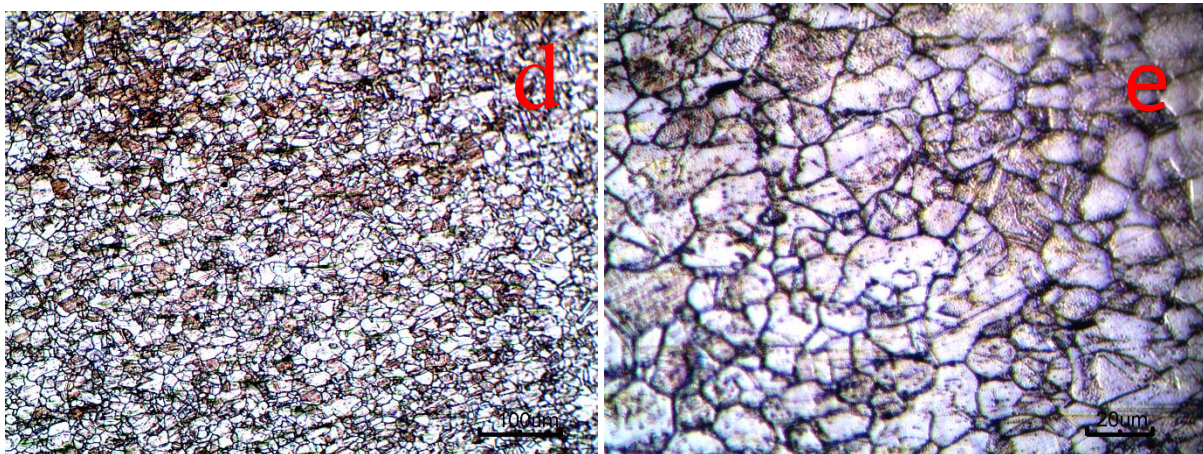
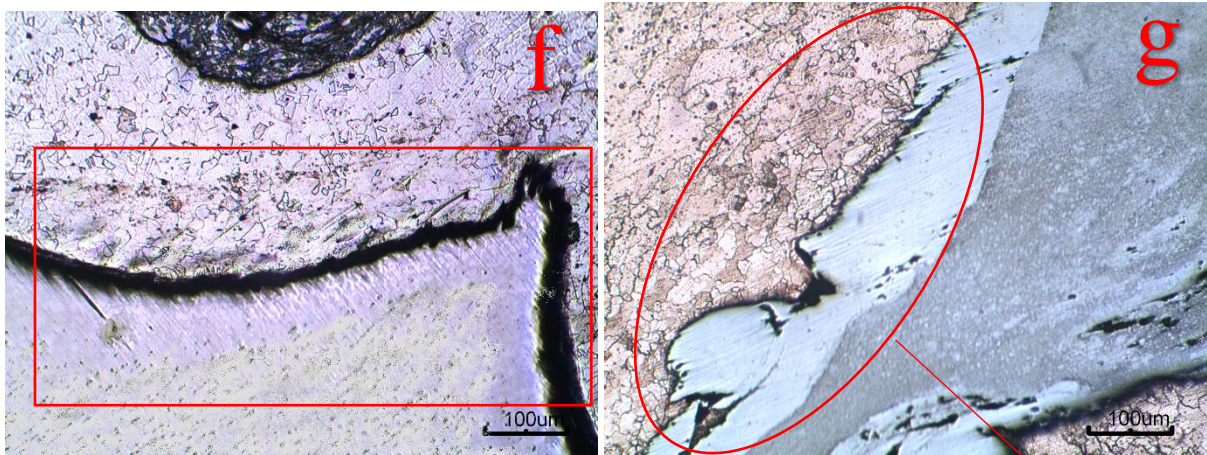


FIG 7.18 Microstructure of SS304 at d 100 X, e 400 magnification



Mixing region (clearly shows Cu-Cr-Zr mixes with SS304)

Fig 7.19 Microstructures at the weld bead of Cu-Cr-Zr/SS 304

7.8.1 Microstructural analysis

In FSW, process to evaluate the microstructural evolution is quite complicated. In case of parent metal microstructures of Cu-Cr-Zr is shown in fig(c). The grains are generally inhomogeneous in nature having fine grain size and showing the hardness of about 103.30 HV. As compared to the TMAZ region of the Cu-Cr-Zr, micro-structural grain size increases due to the effect of high heat generation. Due to high thermal gradient at TMAZ of Cu-Cr-Zr there is an elongation of grains as compared to the parent metal. Elongation of grains can also be seen in 400 X resolution. Generally, fine grain structure will have higher value of toughness. Toughness is the ability of a material to withstand and absorb the maximum energy applied due to external loads up to fracture. So fine grained structure provides more resisting area due to presence of more grains and less interstitial positions present inside it. Whereas for a coarse grained structure, there will be less resisting area due to less grains and more interstitial positions present inside it. So during crack propagation, the crack should follow and shear more grains as in case of fine grains compared to coarse ones. Thus a fine grained structure resists and absorbs more energy up to fracture and thus it is tougher. Thus refinement brings out a change in the grain size in the microstructure and thus effects the hardness in the TMAZ region. Due to these factors during tensile test specimen breaks from the TMAZ of Cu-Cr-Zr side. Fig (d), Fig (e) shows the microstructure of SS304 at 100 X and 400X magnification. It has single phase austenitic structure, with a coarse grain size and irregular grain shape. Fig (f) and Fig (g) shows the microstructures at the bead zone where two materials mixes each other and welded

with each other. Fig (f) shows the microstructures of SS304 at the weld bead while Fig (g) shows the microstructures of Cu-Cr-Zr at the weld bead. As compared to the TMAZ region of SS304 grain size is smaller thus having larger hardness than TMAZ region of the copper.

On the basis of primary deformation bands the base metal grains are splits into different types. Due to the continuous dynamic recrystallization in welded nugget, refinement in microstructures occurs, sub grains growth associated with absorption of dislocation is a typical CDRX mechanism. During FSW, the weld nugget undergoes a serious plastic deformation process. Results in temperature increase and mass friction heat. Heat dissipation is faster from the bottom side. The growth of CDRX nucleation is accelerated due to large thermal heat. The CDRX nucleation instigates growth into new equiaxed grains in the upper layer while some of that growth will be restrained owing to the insufficient thermal energy of bottom layer. At low temperature

7.9 SEM Analysis

In order to know the topology and the morphology of the TMAZ region of Cu-Cr-Zr allow we get various structures in SEM as follows:

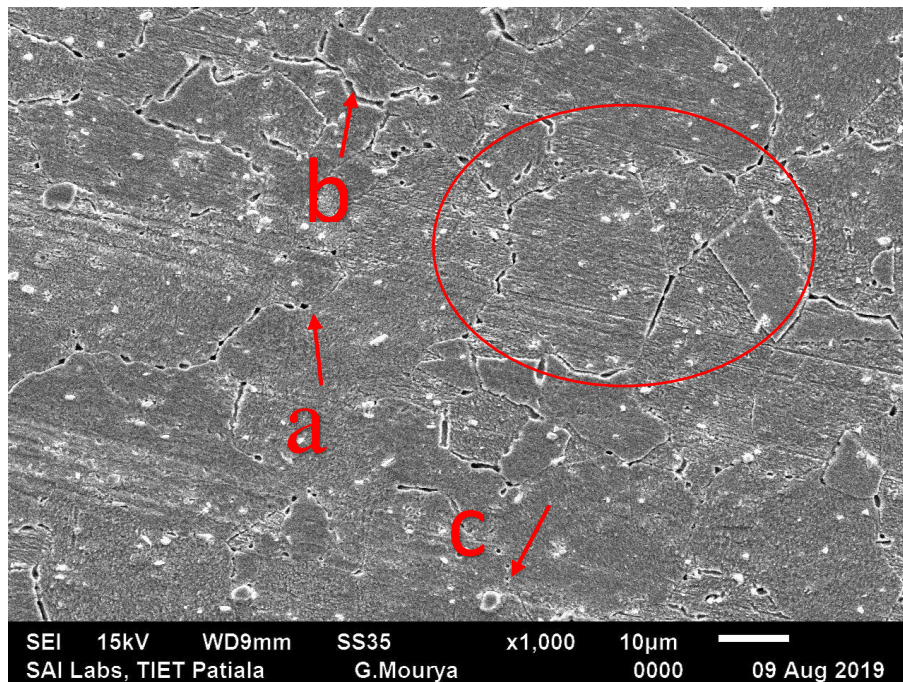


Fig 7.20 SEM grain structure of Cu-Cr-Zr in TMAZ Top region

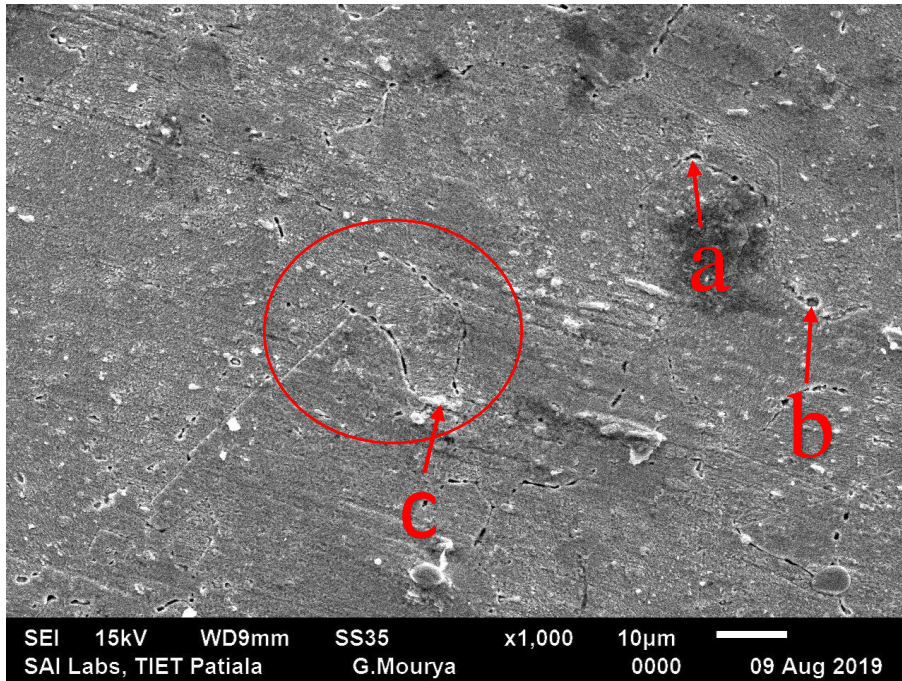


Fig 7.21 SEM grain structure of Cu-Cr-Zr in TMAZ intermediate region

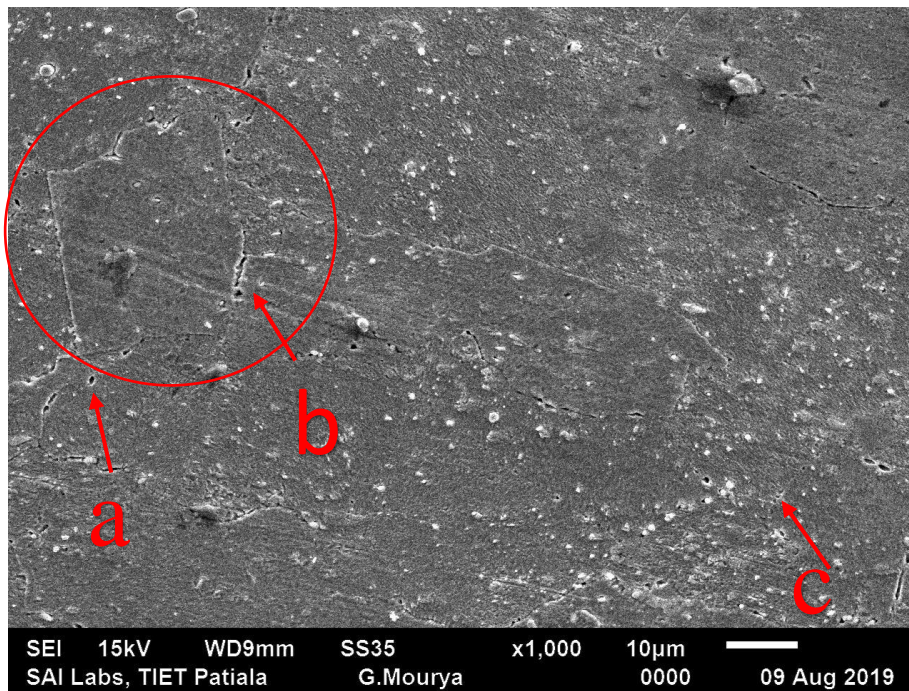


Fig 7.22 SEM structures of Cu-Cr-Zr in TMAZ bottom region

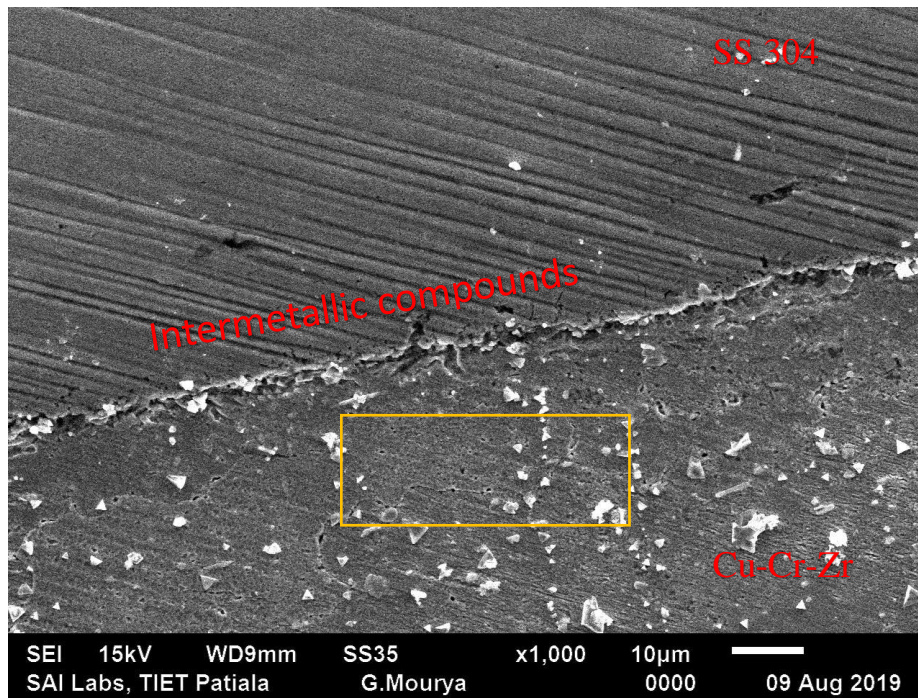


Fig 7.23 SEM structure of intersection of SS304 and Cu-Cr-Zr

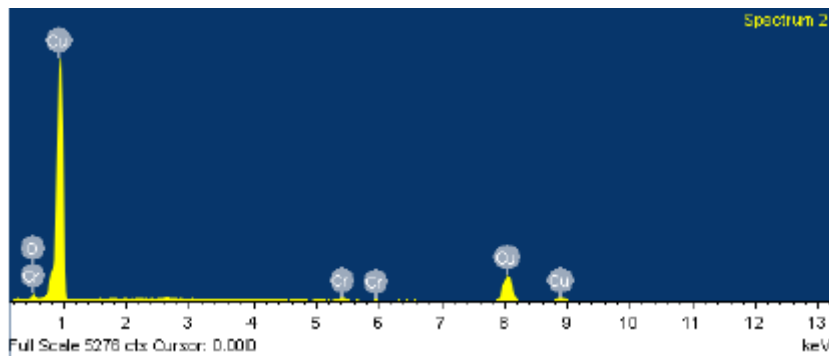


Fig 7.24 Shows the elemental composition in intersection region (yellow rectangle)

Table 7.3 Major elements present in specified region

| Element | Weight % | Atomic % |
|-----------|----------|----------|
| O | 3.01 | 10.91 |
| Cr | 2.26 | 2.52 |
| Cu | 94.74 | 86.57 |

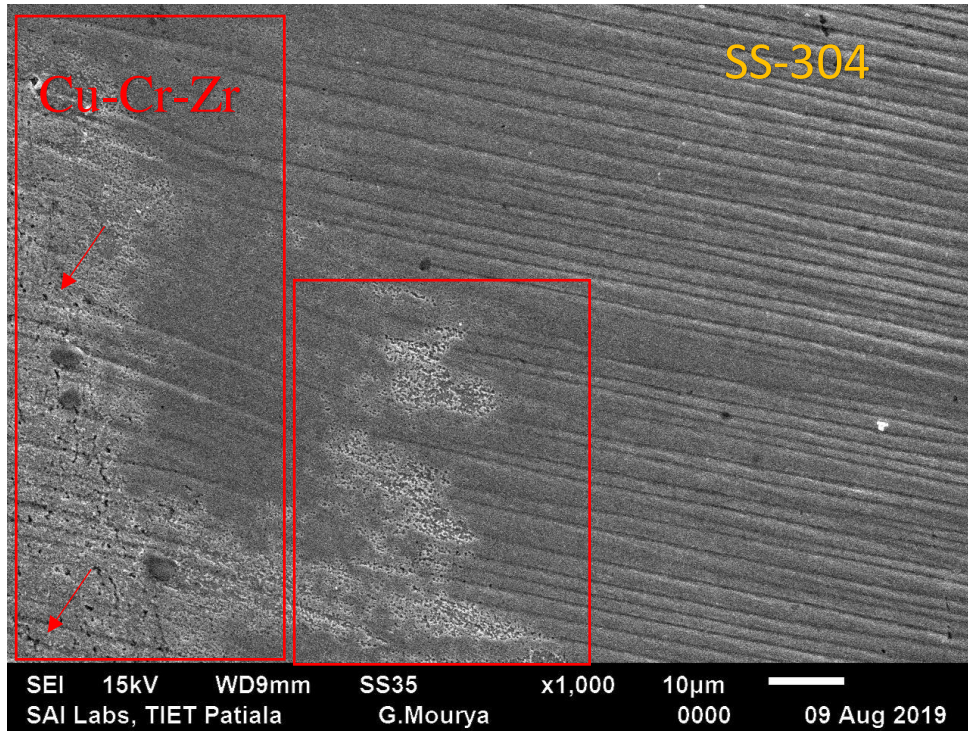


Fig 7.25 SEM image in the bead region shows intermixing

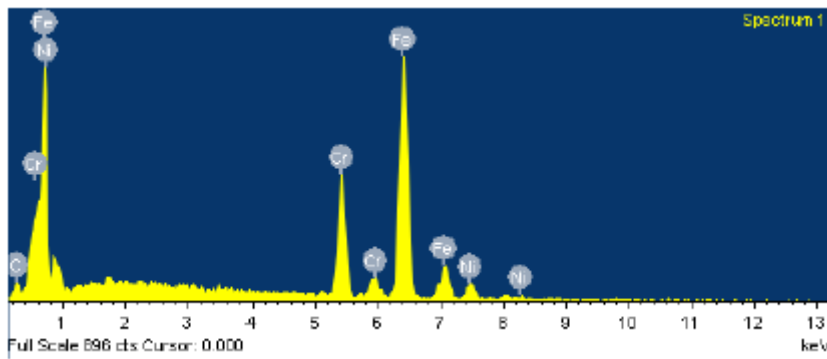


Fig 7.26 Peaks shows the content present in intermixing region

Table 7.4 Major elements present in bead region

| Element | Weight % | Atomic % |
|---------|----------|----------|
| C | 3.16 | 13.05 |
| Cr | 19.66 | 18.75 |

7.10 RESULTS

Image of bead region shows that the mixing of two metals in the bead region. Red boxes clearly shows that the copper is mixed with the SS 304 and the colour difference shows how the copper is shifted towards SS304 side during mixing. Micro cracks are also generated in it as shown in figure because of high heat conditions.

In the intersection region:

- The layers of SS 304 and Cu-Cr-Zr can be easily identified from this image.
- Intermetallic compounds are formed in the intersection of both the layers.
- Due intermetallic compounds hardness decreases.
- The average size of the grain is calculated to be 9.69 μm .
- Due to fine grain size at the intersection, generally effects the Hardness .Thus weld region has higher hardness as compared to the base metal.

TMAZ of bottom region:

- Chromium rich particle “a”.
- Zirconium rich phase’s particle “b”.
- Cr primary phases embedded in Cu_5Zr particle “c”.
- The average grain size of this region is calculated to be 12.55 μm .

TMAZ of top region:

- The average grain size is calculated as 11.25 μm .
- Due to the non-homogeneous elongation in the grain size, generally effects the hardness .Finer grains have high mechanical properties as compared to the coarse grains and also initiates crack propagation.
- Thus under tensile loading the coarse grains start elongating or crack formation initiate.
- Thus, the specimen breaks from the TMAZ of Cu-Cr-Zr.

TMAZ if intermediate region:

- The average grain size is calculated as 9.26 μm .

Table 7.5 Average grain size in different region

| | Average grain size(um) | Range(um) |
|--------------------------|-------------------------------|------------------|
| TMAZ top | 11.25 | 4.765-21.27 |
| TMAZ intermediate | 9.26 | 5.65-13.50 |
| TMAZ bottom | 12.55 | 8.75-17.59 |
| Intersection | 9.69 | 6.20-19.67 |

7.11 Cross comparison of Grain size, fractography and hardness value

7.11.1 TMAZ top:

Under the effect of high heat, the grains possess elongation and convert into non-homogeneous coarse grains. Thus due to coarse grain size, the mechanical properties experience a change. Hardness in this region decreases and thus breaks from the TMAZ region of Cu-Cr-Zr side. SEM testing in this region confirms the elongation in grain size and from the fractography image, it is concluded that the failure is ductile. Serration effect also confirms the elongation in the grains and support ductile failure. (see Fig 7.9, Fig 7.20)

7.11.2 TMAZ intermediate:

The grains are non-homogeneous and coarse in nature. Grain size of intermediate region is slightly less than top region. Thus from fractography image it is clear that the failure is ductile but serration effect in this region is high. Serration effect possesses slightly high strength thus low elongation in this region as compared to the TOP TMAZ region. (Refer Fig 7.11, Fig 7.21)

7.11.3 TMAZ bottom:

Image clearly shows that the failure is pure ductile due to the non-homogeneous coarse grain size. Larger grain size tends to high elongation thus the fracture is pure ductile in nature. (Refer Fig 7.12, Fig 7.22)

High quality welded joints in 3mm thick Cu-Cr-Zr/SS304 is manufactured by friction stir welding process. The weld metal provides a good combination of strength and toughness and appeared to match or exceed the properties of the parent metal. The obtained results are summarized here:

1. The Feasibility of joining of nuclear grade metal Cu-Cr-Zr and SS 304 is quite possible by friction stir welding.
2. The optimum parameters were found to be rotational speed of 1400 rpm, feed rate of 50 mm/min, 2 degree tilt angle and tool diameter of 15 mm.
3. Welded joint is enough much strong that under tensile load testing, the specimen breaks from the TMAZ of Cu-Cr-Zr side.
4. Due to high thermal gradient in TMAZ of Cu-Cr-Zr side, elongation in grain size can be seen clearly. Coarse grain size results in the ductile failure.
5. At weld nugget, the mixing and bonding of two metal can be easily identified.
6. In fractography, TMAZ of top Intermediate surface and bottom side were having coarse grains that leads to ductile failure.
7. At intersection, average grain size is calculated to be $9.6\mu m$.
8. In stir zone, the hardness was about 347.88 HV₅₀₀. Due to refinement in grain size from coarse to fine.

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