Chapter 3 Materials and Methods

Materials, methods and experiments section is designed under different subsections such as base material and experimental set-up, experimental methods, tool designs and process parameters, and testing methods.

3.1 Base materials and experimental set-up

The dissimilar system of AA6061-T651 and Electrolytic-Tough-Pitch (ETP) Cu, having 6.3 mm thickness, are used as base materials, procured under the sponsored project of NFP/MAT/8A 10/04, Board of Research in Fusion Science and Technology (BRFST), Institute for Plasma Research (IPR)-Gandhinagar. Chemical compositions of base materials are shown in Table 3-1. The experiments are conducted on special experimental set up of FSW (see Figure 3-1), developed under the sponsored project of ISRO E33011/60/2010-V, Indian Space Research Organization (ISRO)-Ahmedabad, which includes tool tilt arrangement, control panel of rotational speed and travel speed, and fixture for placing the workpieces. Stainless steel fixture is used as shown in Figure 3-1 (b) and (c), which can accommodate portable workpieces of size 100 X 100 mm. The use of stainless steel helps to retain the heat at the rear side of the workpiece, due to its low thermal conductivity characteristic. Operating window of FSW machine is presented in Table 3-2.

Table 3-1 Chemical compositions of base materials

Materials	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Impurities	Al
AA 6061-T651	0.56	0.30	0.17	0.12	1.03	0.11	0.08	0.03	0.04	Balance
Electrolytic-Tough-Pitch (ETP) copper	-	-	>99.9	-	-	-	-	-	Balance	-

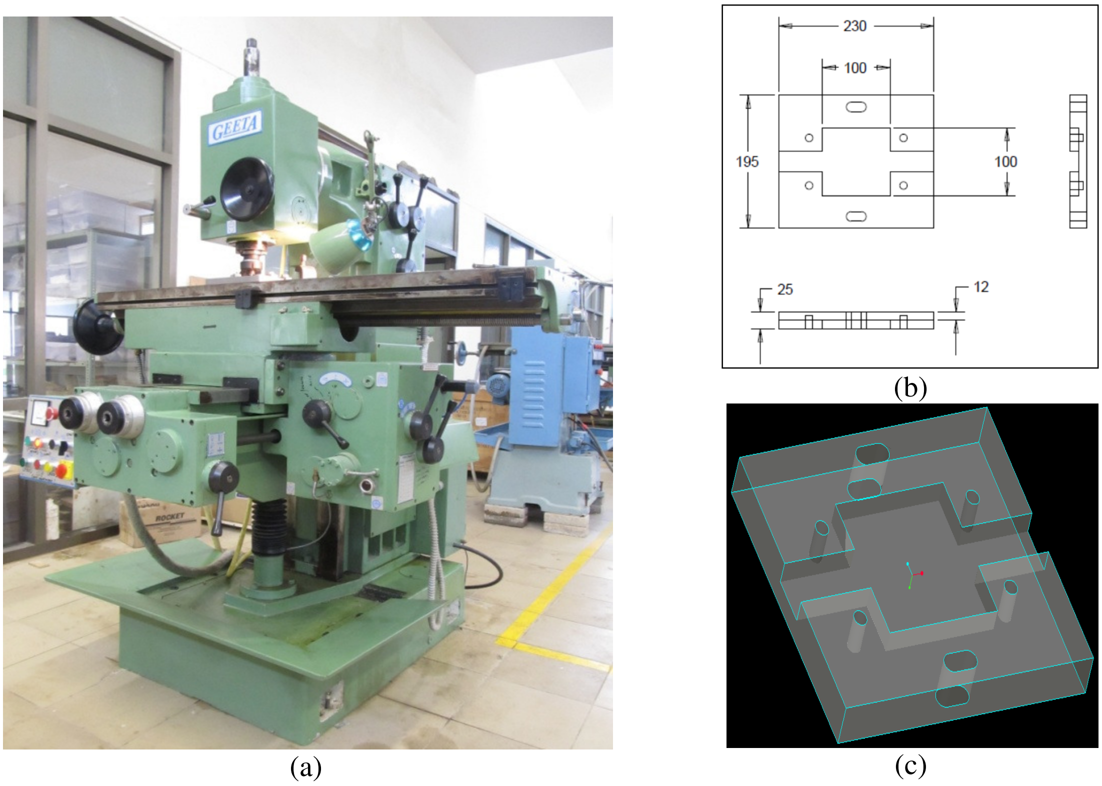

Figure 3-1 (a) FSW machine, (b) and (c) fixture

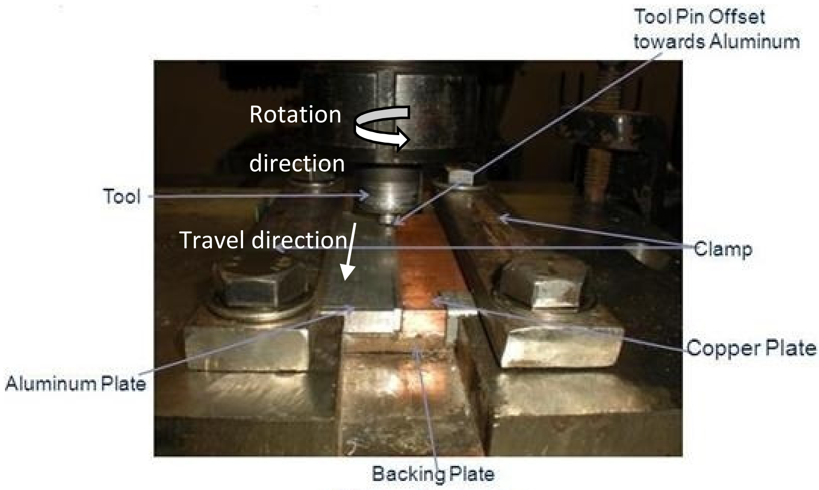

Figure 3-2 Workpiece material positioning

Table 3-2 Operating window of FSW machine

Process parameter	**Operating range**
Rotational Speed	35 – 1500 (RPM)
Welding Speed	20 – 800 (mm/min)
Motor Power	3.0 H.P
Tilt Angle	0° - 90° (in both transverse direction)

3.2 Experimental method

Experiments are carried out with six set of experiments in order to investigate dissimilar FSW for its tool design, process parameters and hybrid approaches. Each set of experiment is performed to analyse the influence of specific process parameters on properties of dissimilar Cu-Al FSW. Individual effect of each parameter is investigated while rest of the process parameters are kept constant. Process parameters such as tool pin design and profiles, tool tilt angle, tool pin offset, welding speed and hybrid FSW are selected for investigation based on the gap identified from the literature review, aforementioned in section 2. 9. Each set of experiment is performed based on the results of previous set of experiments. Table 3-3 shows the summary of methods for six set of experiments.

3.3 Tool designs and process parameters

There are ten different tool designs applied on the dissimilar Cu-Al FSW system, wherein tool material is kept constant at 62 HRC hardened tool steel of M2 grade (see Table 3-4 for chemical compositions). Different tool pin profiles such as taper, polygonal and cylindrical are designed based on the results obtained in each set of experiment. Table 3-5 provides different tool designs used in the present investigation. Selection of specific tool design is explained in **Chapter 4**.
In case of polygonal tool pin profile, the tool is mechanically cleaned after each and every experiments, with the help of finishing tools such as workshop files. Sticking of material at polygonal pin changes the shape of the tool pin, especially for the harder alloys (Mehta et al., 2014).

3.3.1 FSW parameters

Process parameters are investigated as per Table 3-3, wherein specific process parameter is investigated, while rest of the parameters are kept constant. Feasibility trials are conducted initially in order to set the working window of FSW basic process parameters. It is reported from the feasibility trials that, below of 1500 rom rotational speed, and welding speed of 100 mm/min and above are resulted into the tool failure due to extremely cold conditions. Tool failure photographs are evidenced in Appendix I. Therefore, experiments are performed on rotational speed of 1500 rpm and welding speeds below 100 mm/min.

Table 3-3 Summary of methods for six set of experiments

Set of experiment	Investigated parameters	Constant process parameters
1	Tool pin offset, taper tool pin profile and shoulder diameter	Rotational speed, welding speed, tilt angle, workpiece positioning, tool design
2	Welding speed, cylindrical tool pin profile and shoulder diameter	Rotational speed, tilt angle, workpiece positioning, tool pin offset, tool design
3	Tool pin diameter for cylindrical tool pin profile	Rotational speed, welding speed, tilt angle, workpiece positioning, tool pin offset
4	Polygonal pin profile features	Rotational speed, welding speed, tilt angle, tool pin offset, workpiece positioning
5	Tool tilt angle	Rotational speed, welding speed, workpiece positioning, tool pin offset, tool design
6	Preheating current of heating assisted FSW, and Cooling medium of cooling enhanced FSW	Rotational speed, welding speed, tilt angle, workpiece positioning, tool pin offset, tool design, distance between FSW tool (parameters of normal FSW), Preheating torch, electrode extension, gas flow rate, electrode diameter, torch angle and voltage (parameters of heating assisted FSW), Distance between cooling source and FSW tool, flow rate or pressure of cooling medium (parameters of cooling enhanced FSW)

Table 3-4 Chemical compositions of tool material

Element	C	Cr	V	W	Mo	Co	Cb
Percentage (%)	0.80	4.00	2.00	6.00	5.00	_	_

Table 3-5 Different tool designs used in the present investigation

Tool No.	Tool design		Tool No.	Tool design
1	SD: 19 mm PRD: 6 mm PTD: 3 mm PSp: Taper left hand threaded (1 mm pitch) SPR: 2.6:1 PL: 6.1 mm		2	SD: 26.64 mm PD: 8 mm PSp: Cylindrical left hand threaded (1 mm pitch) SPR: 3.33:1 PL: 6.1 mm
3	SD: 26.64 mm PD: 6 mm PSp: Cylindrical left hand threaded (1 mm pitch) SPR: 4.44:1 PL: 6.1 mm		4	SD: 26.64 mm PD: 10 mm PSp: Cylindrical left hand threaded (1 mm pitch) SPR: 2.66:1 PL: 6.1 mm
5	SD: 26.64 mm PSp: Triangular PES: 10.77 mm SAP: 50.22 mm^2 DAP:121.15 mm^2 PL: 6.1 mm		6	SD: 26.64 mm PSp: Square PES: 7.09 mm SAP: 50.26 mm^2 DAP: 78.85 mm^2 PL: 6.1 mm
7	SD: 26.64 mm PSp: Hexagonal PES: 4.4 SAP: 50.29 mm^2 DAP: 60.82 mm^2 PL: 6.1 mm		8	SD: 26.64 mm PSp: Triangular PES: 6.93 mm SAP: 20.78 mm^2 DAP: 50.27 mm^2 PL: 6.1 mm
5	SD: 26.64 mm PSp: Square PES: 5.66 mm SAP: 32 mm^2 DAP: 50.27 mm^2 PL: 6.1 mm		10	SD: 26.64 mm PSp: Hexagonal PES: 4 mm SAP: 41.57 mm^2 DAP: 50.27 mm^2 PL: 6.1 mm
*SD: shoulder diameter, PRD: pin root diameter, PTD: pin tip diameter, PD: pin diameter, PSp: pin surface profile, SPR: shoulder to pin diameter ratio, PL: pin length, PES: polygonal edge size, SAP: static cross sectional area of pin, SSP: static cross sectional surface of pin, DAP: dynamic cross sectional area of pin, DSP: dynamic cross sectional surface of pin				

Workpiece material positioning is an important process parameter reported in most of the literatures of dissimilar Cu-Al FSW, which is kept constant as Cu at advancing side and Al at retreating side. Specific values of process parameters of FSW for the six set of experiments are presented in Table 3-6. Selection of these values is explained in **Chapter 4**.

3.3.2 Hybrid FSW

Novel approaches of assisted heating and cooling are implemented in a dissimilar Cu-Al FSW system as shown in Figure 3-3, wherein process parameters of FSW are kept constant as per Table 3-5. In order to evaluate the influence of individual effect of assisted heating and cooling, the FSW parameters are kept constant at their favourable values based on the previous set of experiments. The external tungsten inert gas (TIG) torch is kept in front of the FSW tool at the Cu side as shown in Figure 3-3 (a) in case of heating assisted FSW (HFSW) approach. Experiments of heating assisted FSW are conducted at different preheat currents of 40, 80 and 120 amps. The other process parameters of HFSW, namely, the distance between tool and torch, electrode extension, gas flow rate, electrode diameter, torch angle and voltage are kept at 25 mm, 4 mm, 10 litre per minute, 2.9 mm, 45° and 14 V respectively. The heating is applied at the time of welding phase just after the plunge phase.

Another approach of external cooling is applied through pipe behind the FSW tool as shown in Figure 3-3 (b). Two different cooling sources of compressed air and water are used individually in case of cooling enhanced FSW (CFSW). The distance between FSW tool and cooling source is kept at 20 mm. Pressure of compressed air is set to 25 PSI, while the water flow rate is kept at 100 ml per minute.

Table 3-6 Process parameters of FSW used in present investigation

<table>
<tr><th>Set of exp.</th><th colspan="3">Tool pin offset (mm)</th><th colspan="4">Welding/ Travel speed (mm/min)</th><th colspan="5">Tool tilt angle (˚)</th><th>Rotational speed (rpm)</th><th>Workpiece material positioning</th><th colspan="6">Tool No.</th></tr>
<tr><td>1</td><td>1</td><td>2</td><td>3</td><td colspan="4">30</td><td colspan="5">0</td><td>1500</td><td rowspan="7">Cu at advancing side and Al at retreating side</td><td colspan="6">1</td></tr>
<tr><td>2</td><td colspan="3">2</td><td>40</td><td>55</td><td>70</td><td>95</td><td colspan="5">2</td><td>1500</td><td colspan="6">2</td></tr>
<tr><td>3</td><td>1</td><td>2</td><td>3</td><td colspan="4" rowspan="5">40</td><td colspan="5">2</td><td>1500</td><td colspan="2">2</td><td colspan="2">3</td><td colspan="2">4</td></tr>
<tr><td>4</td><td colspan="3" rowspan="4">2</td><td colspan="5">2</td><td>1500</td><td>5</td><td>6</td><td>7</td><td>8</td><td>9</td><td>10</td></tr>
<tr><td>5</td><td>0</td><td>1</td><td>2</td><td>3</td><td>4</td><td>1500</td><td colspan="6">2</td></tr>
<tr><td>6</td><td colspan="5">2</td><td>1500</td><td colspan="6">2</td></tr>
<tr><td>7</td><td colspan="5">2</td><td>1500</td><td colspan="6">2</td></tr>
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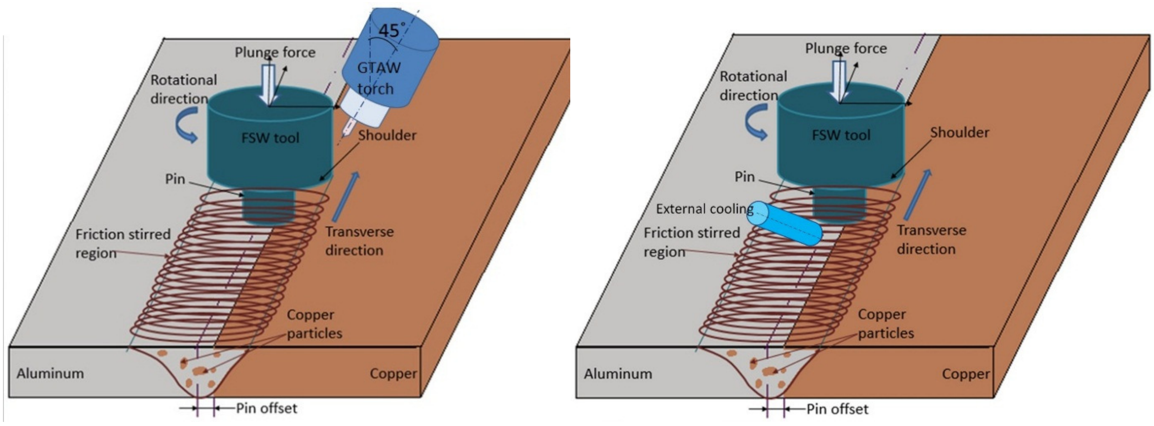


(a) (b)

Figure 3-3 Hybrid Cu-Al FSW, (a) TIG assisted FSW and (b) external cooling enhanced FSW

3.4 Testing methods

The welded coupons are extracted for visual observation, macrograph examination, mechanical testing and metallurgical characterization such a way that it comprised various regions of the weld joint. Start region and key-hole region are excluded from the mechanical and metallurgical testing. However, visual examination of key-hole region is carried out to find internal defects at initial stage.

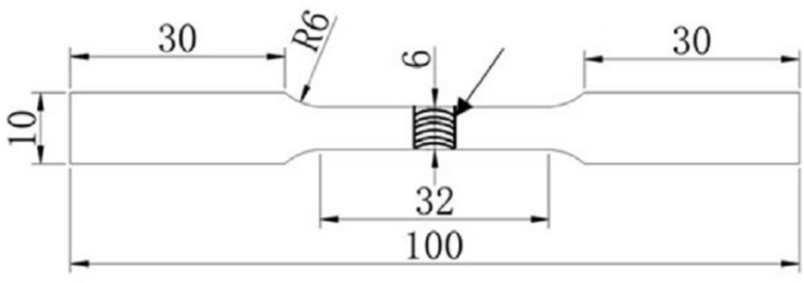
3.4.1 Visual, macro examinations and temperature profile

High resolution camera is used to capture the photographs of welded samples as well as macro samples in order make close analyses. Visual defects such as surface lines, surface tunnel, voids, macro-cracks, oxide formation, etc. are identified and analysed based on the visual and macro examinations. Macro examinations are conducted after metallographic procedure so that interpretation of macrostructure and defects can be easily made. Temperature measurement of the workpiece is carried out through K-type thermocouple at the transverse centre of the work piece on advancing side (AS) as well as retreating side (RS). Drilling of 1.5 mm is performed at 50 mm transverse length and 30 mm away from tool centre wherein K-type thermocouples are inserted. Results of temperature profiles are presented in Appendix II.

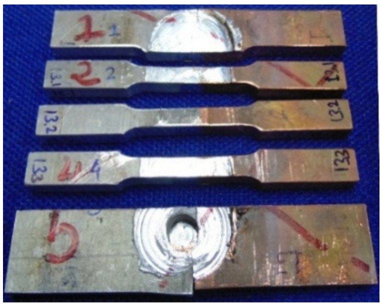
3.4.2 Tensile testing

Tensile testing and hardness analysis are carried out as a part of mechanical testing. There are three samples prepared for tensile testing of each condition in order to see the repeatability and uniformity. The tensile testing is performed on 6 mm thick mini transverse specimens. These specimens are prepared as per the guidelines of ASTM E8 (as shown in Figure 3-4). Tensile testing is performed at loading rate of 1.5 mm/min. These specimens are surfaced before tensile testing in order to obtain results through uniform specimens.

After the tensile testing, the fractured location is identified by visual inspection. Furthermore, the surface topography of fractured tensile specimens is examined by means of macrographs and scanning electron microscopy (SEM) images in order to find the exact reason for the failure.



(a)



(b)

Figure 3-4 Mini tensile specimen preparation (a) as per ASTM E8 standards, (b) prepared through transverse section

3.4.3 Hardness testing

Variations of Vickers Hardness Number (VHN) are evaluated from the mid thickness portion across the transverse cross section of the specimens. The indentation is made along the transverse cross section by load of 500 grams for 20 seconds dwell time at 1 mm intervals.

3.4.4 Microstructures and advanced characterizations

The metallographic investigation, microstructural study followed by SEM, Energy dispersive X-ray spectrographs (EDX) and X-ray diffraction analysis (XRD) are performed on mechanical grinded, polished and chemically etched specimens. Mechanical grinding and polishing is done on 120, 320, 800, 1000, 5000 grit silicon carbide emery papers along with diamond paste. Further, chemical etching of these samples is carried out through solutions made of FeCl + HCl + H2O on Cu side and Keller's reagent made of 5 ml HNO_3 + 3 ml HCl + 2 ml HF on Al side.

SEM analysis is performed at the interface between Cu-stir zone as well as inside the stir zone. Spot EDX is carried out at dark and light area of stir zone in order to find chemical compositions inside the stir zone. XRD analysis is performed on hybrid FS welded specimens and best condition of FS welded sample to evaluate the phases of intermetallic compounds and its amount based on the peaks.

3.5 Summary

The information about base materials used, experimental set-up, tool designs, process parameters, step by step experimental procedure, hybrid FSW and its procedure, and testing methods are explained. This section summarises experimental methodology performed under different process parameter and tool designs. Implementation of hybrid FSW is presented along with its procedure. Standard testing and characterization procedures followed to evaluate joint quality are mentioned and discussed.