CHAPTER – IV

EXPERIMENTAL PROCEDURE

4.1 INTRODUCTION

In the experimental investigation the temperature distribution and residual stresses of AA 5059 grade aluminium alloy joints fabricated using Gas Tungsten Arc Welding (GTAW) and Friction Stir Welding (FSW) processes were measured.

The experimental work was planned in the following sequence:

(i) Evaluation of chemical composition and mechanical properties of base metal.
(ii) Fabrication of square butt joints using GTAW and FSW processes based on RSM for optimized conditions.
(iii) Measuring the temperature distribution on the welded sample in the transverse direction by using Lab VIEW software.
(iv) Measuring the residual stresses of welded AA 5059 grade aluminium alloy joints by using X-ray diffraction method.
(v) Evaluation of tensile and hardness properties of welded AA 5059 grade aluminium alloy joints.
(vi) Micro structural analysis by optical microscopy of AA 5059 grade aluminium alloy joints.
(vii) Studying the fractography of tensile specimen after fracture of welded AA 5059 grade aluminium alloy joints.
(viii) Measuring the residual stresses of welded AA 5059 grade aluminium alloy joints by using X-ray diffraction method.
The detailed experimental procedures involved in each stage of the experimental work are briefed in the following section.

4.2 EVALUATION OF BASE METAL PROPERTIES

The base metal (BM) used in this study was 4 mm thick cold rolled, AA 5059 grade aluminum alloy plates. The chemical composition of the base metal is presented Table 4.1 was obtained using a vacuum spectrometer (Make: ARL USA, model 3460). Table 4.2 shows the mechanical properties of the base metal. Tensile specimens were prepared as shown in Fig. 4.1 to obtain the base metal properties. ASTM E8 M-04 guidelines were followed for preparing test specimen. Tensile test were carried out in 100 kN, electro mechanical controlled Universal Testing Machine (Make: FIE-BLUE STAR, India: Model: UNITEK- 94100). The specimens were loaded at the rate of 1.5 kN/min as per ASTM specifications, so that tensile specimen undergoes deformation. Vicker’s micro hardness testing machine (Make: Shimadzu, Japan and model: HMV-2T) was employed for measuring the hardness of base metal with 0.05 kg load. The optical micrograph of as received base material is shown in Fig.4.2.

Table 4.1 Chemical composition (wt. %) of base material (Spectrometry results)

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Si</th>
<th>Cu</th>
<th>Mn</th>
<th>Mg</th>
<th>Cr</th>
<th>Zn</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight in (%)</td>
<td>0.041</td>
<td>0.003</td>
<td>0.933</td>
<td>5.21</td>
<td>0.5-0.6</td>
<td>&lt;0.001</td>
<td>0.489</td>
<td>Remain.</td>
</tr>
</tbody>
</table>
Table 4.2 Mechanical properties of base material

<table>
<thead>
<tr>
<th>Tensile strength (MPa)</th>
<th>0.2% yield strength (MPa)</th>
<th>Elongation in 50mm gauge length (%)</th>
<th>Hardness (HV 0.05 kg@ 15 sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>385</td>
<td>290</td>
<td>16</td>
<td>123</td>
</tr>
</tbody>
</table>

Fig. 4.1. Un notched tensile specimen

4.3 FABRICATION OF JOINTS

The rolled plates of AA 5059 aluminium were machined to the required dimensions (150mm x 75 mm x 4 mm). Square butt joint configuration as shown in Fig.4.3 was prepared to fabricate the joints.

The plates to be joined were mechanically and chemically cleaned by acetone before welding to eliminate surface contamination. Figure 4.4 (a) displays
the GTAW machine used in this investigation to fabricate the joints. Photograph of Friction Stir Welding machine which was developed by R.V.S. machine tool, Coimbatore is shown in Fig.4.4 (b). The direction of welding was normal to the rolling direction. Necessary care was taken to avoid joint configuration and the joints were made by securing the base material in clamps rigidly. Single pass welding procedure was applied to fabricate the joints. The welding conditions, process parameters and tool parameters used in this investigation are presented in Table 4.3(a) & (b). Non-consumable tool made from high speed steel which was used to fabricate the FSW joints. Tool tilt angle 2.5° was maintained in this work [Arakare et al (2013)]. Tool tilt angle by 2.5° such that rear of the tool is lower than the front, to assist the forging process. The tool is tilted at a certain angle and is rubbed against the surface of the work piece with the pressure which increases the heat and results move stirring of the work piece material. The tool having the ratio of shoulder diameter to pin diameter (D/d) as 3 has been chosen for this study because it is having good joining properties among various pin configurations [W.M. Thomas et.al (1999)]. In this investigation, taper threaded tool pin used, which facilitates the stirring action from the tip to the collar and avoid the turbulence compared to cylindrical thread tool, thereby this tool profile is effective for getting defect free welds. Better stirring action from the tip to the collar and better consolidation of the material are the reasons for choosing the threaded tool profile. The photograph taken during gas tungsten arc welding and friction stir welding of AA 5059 graded aluminium alloy is shown in Fig. 4.5 (a) and 4.5 (b) respectively. The fabricated joints are shown in Fig.4.6. The welded joints were sliced using a power hacksaw and specimens were machined to the required dimensions for further testing.

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(a) GTAW machine

(b) FSW Machine

Fig. 4.4 Welding machines utilized for fabrication of the joint
Figure 4.5 Photograph taken during welding

(a) GTAW Machine

(b) FSW machine

Figure 4.5 Photograph taken during welding
Fig. 4.6 Photograph of some fabricated joints of AA 5059 Aluminium alloy

Table 4.3.a Welding Conditions and process Parameters (GTAW)

<table>
<thead>
<tr>
<th>Parameters</th>
<th>GTAW process</th>
</tr>
</thead>
<tbody>
<tr>
<td>Welding machine</td>
<td>Lincoln, USA</td>
</tr>
<tr>
<td>Tungsten electrode diameter(3mm)</td>
<td>3</td>
</tr>
<tr>
<td>Current (Amps)</td>
<td>110</td>
</tr>
<tr>
<td>Shielding gas</td>
<td>Argon</td>
</tr>
<tr>
<td>Gas flow rate(lit/min)</td>
<td>6</td>
</tr>
</tbody>
</table>

Table 4.3.b Welding Conditions and process Parameters (FSW)

<table>
<thead>
<tr>
<th>Parameters</th>
<th>FSW process</th>
</tr>
</thead>
<tbody>
<tr>
<td>Welding speed (mm/min)</td>
<td>25</td>
</tr>
<tr>
<td>Rotational speed (rpm)</td>
<td>950</td>
</tr>
<tr>
<td>Axial force (kN)</td>
<td>3.4</td>
</tr>
<tr>
<td>Tool pin profile</td>
<td>Taper Threaded</td>
</tr>
<tr>
<td>Shoulder diameter(mm)</td>
<td>12</td>
</tr>
<tr>
<td>Pin diameter(mm)</td>
<td>4</td>
</tr>
<tr>
<td>Pin length(mm)</td>
<td>3.7</td>
</tr>
<tr>
<td>Tool tilt angle(Degree)</td>
<td>2.5</td>
</tr>
</tbody>
</table>
4.4 TEMPERATURE MEASUREMENT

Experiments were carried out under different welding parameters in order to use the measured temperature data for verification of the accuracy of the thermal model. The temperature measurement during welding is discussed in this section.

4.4.1 Data Acquisition

Data acquisition (DAQ) is the sampling of any physical phenomenon, to generate data that can be manipulated by a computer. Data acquisition typically involves acquisition of signals and processing the same to obtain desired information. The components of data acquisition systems include appropriate sensors that convert any measurement parameter to an electrical signal, which is acquired by data acquisition hardware. Acquired data is displayed, analyzed, and stored on a computer, using vendor supplied software or dedicated program using programming languages, such as BASIC, C, and Fortran. LABVIEW is one such software, which offers a graphical programming environment optimized for data acquisition.

Data acquisition begins with the physical phenomenon or physical property of an object (under investigation) to be measured. A transducer is a device that converts a physical property or phenomenon into a corresponding measurable signal, such as voltage, current, change in resistance or capacitance, temperature, pressure etc. DAQ also deploys various signal conditioning techniques to adequately modify various electrical signals into voltage that can then be digitized using analog to digital converters.
Signal conditioning may be necessary if the signal from the transducer is not suitable for the DAQ hardware to be used. DAQ hardware is what usually interfaces between the signal and a computer. It could be in the form of modules that can be connected to the computer's ports (parallel, serial, USB, etc.) or cards connected to slots (Peripheral Component Interconnect (PCI), Industry Standard Architecture (ISA) in the mother board. Driver software that usually comes with the DAQ hardware or from other vendors, allows the operating system to recognize the DAQ hardware and programs to access the signals being read by the DAQ hardware.

4.4.2 On-Line Temperature Measurement

To measure the temperature during welding the K Type Chromel-Alumel thermocouple was used [Lu et al (1988), Sadak et al (1995)]. The range of the thermocouple was 95°C to 1260 °C. Another requirement was on the smaller diameter of the hot end of the thermocouple since sensitivity was of a prime concern. The hot end diameter of the thermocouple was 1.5 mm, the cold end was fixed to a thermocouple bank and this was in turn connected to the data acquisition system Lab VIEW [Klobcar et al. (2004)] as shown in Fig. 4.7. Thermocouples are attached to a DAQ system which could measure the data at 15 Hz. The temperature is measured every second and stored in the computer. The time data corresponding to the collected temperature data is also stored in the computer to find the thermal history of the weld specimen during the welding process. Temperature data collection is done through a DAQ system that is attached to the laptop computer running LAB VIEW software.
The user interface was the front panel in terms of a block diagram. The block diagram consists of code, which was the flow chart for the real time temperature acquisition is shown in Fig.4.8. Thermometers were used as indicators to display output values from the program as shown in Fig.4.9. When the DAQ was run, values from controls flow through the block diagram, where they are used in the functions of the diagram, and the results are passed into other functions and indicators through wires.

Fig. 4.7 LABVIEW interface

Fig. 4.8 Block diagram  Fig. 4.9 Thermometer indicators
K type thermocouples made of alumel and chromel with a sensitivity of 41 µV/°C was used for temperature prediction. Layout of position of thermocouple is shown in Fig.4.10. The probe capable of measuring up to 1600 °C range was used. Because of their finite size and response time K type thermocouple have some drawbacks. The drawbacks on drilling holes on the specimen are on thermal aspects, the homogeneity on conduction is affected. The K type thermocouple is more susceptible to electrical resistance. Considering the above two factors the hole dimension was determined based on literature [Fahrenbacher et al (2014), Karunakaran et al. (2011)].

In order to provide the thermocouples, holes are drilled to a depth of 2 mm at three different locations 7 mm, 12 mm, and 17 mm away from the weld centre line and along the mid length at both side of the plate.

![Fig. 4.10 Layout of position of Thermocouple](image)

**4.5 RESIDUAL STRESS MEASUREMENT**

The various methods available for measurement of residual stresses include hole drilling technique, curvature method and diffraction method. In this study X-ray diffraction technique is used for stress measurements. X-ray diffraction has become the one of the standard methods for measuring residual
stress in the past few decades [Matrinez et al. (2003), Hatamleh (2009), Prevey (1986), Cullity (1978), Noyan (1987), Xue et al. (2000), Dolle et al. (1980), Brakman et al. (1988)]. This method is one of the widely used non-destructive techniques for residual stress measurements. Residual stress in the material causes change in the interplanar spacing of the material. The changes in the interplanar spacing “d” can be used with the Bragg’s equation to detect elastic strain “ε” through a change in the Bragg scattering angle Δθ.

4.5.1 X-Ray Stress Measurement

In this study, X-ray residual stress measurements were conducted using AST 3000 X-ray stress analyzer employing CrKα radiation (at DMRL, Hyderabad, India). The instrument uses a pair of solid-state detectors located on each side of the main beam. Diffraction peaks are captured by the individual pixels in the detectors, giving rapid data capture without any mechanical movement. The standard quartz was used for calibration. Data collection and machine control were accomplished through a laptop computer running X3000 software including stress analyzer. The as-welded plates were used for the residual stress measurement, since any processing would alter the levels of stresses induced in the plates. The residual stress was measured for one specimen in each of the two processes (FSW, GTAW) and at the weld bead and at 7 mm and 12 mm from the weld bead centre.

X-ray diffraction measures the strain or the changes in strain, from an unstressed state, by measuring the shifts in the diffraction peak due to an external or residual stress. The measured strains are then converted into a stresses through
Hooke’s Law [(Matinez et al (2003), Prevey (1986)]. X-ray diffraction method employs Bragg’s law to estimate the residual strains present in the atomic plans. The depth of penetration is dependent on the type of radiation, and in practice there are limited types of useful radiation. For example, Cu-Kα radiation, Co-Kα radiation and Cr-Kα radiation are some of the common types of radiation used in laboratory settings. In this experimental study, cobalt tube with Co-K radiation X-ray tube was selected in order to compromise between depth of penetration and Bragg’s angle. The Co-K radiation has higher photon energy than Cr-K radiation. Based on the tube selection, the diffraction plane was decided. In this work, reflection plane {331} was ultimately chosen based on the supporting XRD Win 2.2 software and corresponded to previous reported results. Figure 4.11 illustrates the principle of X-ray diffraction.

A selection of “d vs. sin²ψ” stress measurement process was made in order to get accuracy and precision. Bragg’s Law defines the relationship among the wavelength (λ), diffracted beam angle (2θ) and the interplaner spacing of the lattice (d).

Fig. 4.11 Principle of X-ray diffraction [Cullity B.D. (1978)]
\[ n\lambda = 2d\sin \theta \quad (4.1) \]

Based on the fact that \( \sin \theta \) cannot be greater than unity, the limitations of \( \lambda \), (cannot be too large or too small) and using a first-order reflection (i.e. \( n=1 \)) equation 4.1. becomes equation 4.2.[Hatamleh et al (2009)]

\[ \lambda = 2d\sin \theta \quad (4.2) \]

If the wavelength of X-rays is known, \( d \) can be determined by measuring the angle \( \theta \). From the \( d \) spacing for a given \{hkl\} reflection, the unit cell parameter \( a \), for cubic materials can be calculated using the equation

\[
d = \frac{a}{\sqrt{h^2 + k^2 + l^2}} \quad (4.3)
\]

Note that Co-K radiation has a wavelength of 0.179026nm [Cullity B.D. (1978)] and using the \{331\} reflection of aluminum, which is face centered cubic, the lattice parameter, \( a \), is 0.40497nm and the Miller indices of \( h=3 \), \( k=3 \), and \( l=1 \) resulting in \( 2\theta \) of 148.93 degrees [Cullity (1978)]. The \( 2\theta \) used for all specimen measurements was rounded up to 149 degree. In the presence of residual stresses, \( d \) changes, leading to a shift in X-ray diffraction peaks, which, is a measure of the residual stress. The choice of the diffraction peak, \( 2\theta \), greatly impacts the precision of the stress measurement. The larger the Bragg angle (\( 2\theta \) angle), greater the sensitivity of the X-Ray residual strain measurement and the greater the precision of the stress calculation, so in general, one should use the largest Bragg angle for a given radiation as possible. Once the \( d \) spacing is measured from the centroid of the \( 2\theta \) angle, the measured strain can be determined. If the residual stresses exist...
within the sample, then the d spacing will be different than that of an unstressed state. This difference is proportional to magnitude of the residual stress. If the surface is in compression, then the interplaner spacing “d” is larger than in the stress free state as a result of poisson’s effect. When the specimen is tilted with respect to the incoming beam new grains will diffract and orientation of the diffraction planes is more nearly perpendicular to the stress direction. As a result of the tilt, the d spacing decreases and the angle 2θ increases. In this case the d spacing acts as a strain gauge. Because of the fact that the interplaner spacing is so small, both micro and macro stresses will effect it. The XRD measures sum of all these stresses.

4.6 MEASUREMENT OF MECHANICAL PROPERTIES

4.6.1 Specimen Preparation

The specimens for the characterization of the joints were extracted from the as-welded plates. For each of the material in the mid portion where the thermocouples were glued was selected for characterizing the micro structure and micro hardness. The area wherein specimen for tensile property measurement was extracted is as shown in Fig. 4.12. Un notched smooth tensile specimens [ASTM-E8 04 (2006)] were prepared to evaluate transverse tensile properties of the joints such as yield strength and tensile strength.
4.6.2 Evaluation of Tensile Properties

The extracted specimens were used for the evaluation of the tensile properties. The specimens were made as per the ASTM E8-04 standards which is as shown in Figure 4.1. The transverse tensile properties of the welded joints were measured using the 100kN, electro mechanical controlled Universal Testing Machine (UTM). The specimens were loaded at the rate of 1.5 kN/min as per the ASTM standards. The 0.2% offset yield strength was derived from the diagram and the percentage of elongation was evaluated. Figure 4.13 shows stress-strain curve of base material.
4.6.3 Fractography

![Fractograph of Base Material](image)

Fig. 4.14 Fractograph of Base Material

The fracture surface analysis was done for all the tested tensile specimens to establish the nature of fracture. The fractured surface of one specimen for each test was analyzed through Scanning Electron Microscope (Make: JEPL India Pvt Ltd, Model: JEM -6610LV). Samples were preserved by coating with commercial rust proof oil. Before examining under SEM, the specimen was cleaned ultrasonically with acetone. The fractography of base material is shown in Fig. 4.14.

4.6.4 Macro Structure

Macro structural features of the transverse cross section of the weld specimen were recorded using Stereo Microscope (Make: Ch-Metco, India; Mould: CM 0646) under a low magnification of 10 X to reveal the width and depth of the weld region.

4.6.5 Microstructure

The rate of heat input during welding and the cooling rate after welding strongly influence the grain size and phase formation. Hence it is imperative to
understand the effect of various levels of heat input and its influence on the micro structure. The specimen extracted from the mid portion of the welded plates of 70 mm x 10 mm dimension was used for the micro structural analysis. The specimen was prepared for using the standard metallographic procedure, according to the ASTM E3-01(2011) standards. The specimens were sectioned to the required sizes, as shown in Fig. 4.12. From the joint comprising base metal, heat affected zone and weld metal region were refined using different grades of emery sheets. Buffing was done using the diamond component (1\(\mu\)m particle size) in the disc polishing machine. The samples for observations were prepared by standard metallographic procedures and etched with Keller’s Reagent (2 mL HF, 3 mL HCl, 5 mL HNO₃ and 190 mL water) to reveal the grain structure of the welded joints. Micro structural analysis was carried using a light optical microscope (MEIJI; Japan; model; ML7100) with the Metalvision MVLx1.0 software which is an image analyzing software.

4.6.6 Micro hardness

Vickers’s micro hardness testing machine (Make: Shimadzu, Japan; Model HMV-T1) was employed with 0.05 kg load for measuring micro hardness across the weld as per ASTM E-384-05 guidelines (ASTM, 2005). The micro hardness profile was done using a measured hardness values across the mid thickness of welded joints. The Vickers hardness number calculation is based on the following formula:

\[
HV = 1.8544 \times \frac{P}{d^2}
\]

where
\[
d = \text{Indentation in mm},
\]
\[
P = \text{load in Kgf}
\]
4.7 SUMMARY

The experimental procedures were followed to conduct the experiments using two different processes. Trial runs were made for FSW and GTAW processes to fix the upper and lower limits of the various parameters of weld. During welding, temperatures were measured using LABVIEW software. After the final experiments were conducted, the residual stress measurement was carried out immediately. Residual stress measurement was carried out at Defense Metallurgical Research laboratory, Hyderabad. Then the specimens were extracted to do the characterization as per the standards. The metallurgical preparations and hardness tests were carried out using the facilities available at the Metallurgical Laboratory of the Centre for Materials Joining & Research (CEMAJOR), Department of Manufacturing Engineering, Annamalai University. The SEM analysis was carried out at the National Institute of Technology (NIT), Trichy and at the Department of Manufacturing Engineering, Annamalai University.