CHAPTER 3

EXPERIMENTAL DETAILS

This chapter details the experiments conducted for this study. The materials, the welding processes and parameters used, and the mechanical properties and chemical compositions of the base materials are described in this chapter. Characterization techniques and the mechanical testing procedures employed to evaluate the welds are also described in detail. The experimental set up of the friction stir welding machine and friction stir welding process are shown in Figures 3.1 and 3.2. The detailed research plan is explained in the flow chart in Figure 3.3.

Figure 3.1 Friction Stir Welding Machine
Figure 3.2 Friction Stir Welding Process

Figure 3.3 Flow Chart of the Experimental Procedures
3.1 CHEMICAL COMPOSITION OF THE BASE MATERIALS

The major part of this study was conducted on 2219-87 and 5083-321 aluminum alloys of thickness 6 mm, to make these welds. Initially, to identify the optimal welding parameters, 5mm thickness plates were used. The chemical compositions of the base materials used in this study are given in Table 3.1. The values presented in the table are the average of a minimum of three investigations using the spectral analysis of X-Rays.

Table 3.1 Chemical composition of the base materials (weight %)

<table>
<thead>
<tr>
<th>Material</th>
<th>Mg</th>
<th>Mn</th>
<th>Fe</th>
<th>Si</th>
<th>Cu</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA5083</td>
<td>4.15</td>
<td>0.73</td>
<td>0.31</td>
<td>0.13</td>
<td>0.025</td>
<td>Remaining</td>
</tr>
<tr>
<td>AA2219</td>
<td>0.01</td>
<td>0.27</td>
<td>0.13</td>
<td>0.01</td>
<td>6.7</td>
<td>Remaining</td>
</tr>
</tbody>
</table>

3.2 MECHANICAL PROPERTIES OF THE BASE MATERIALS

The mechanical properties of the aluminum alloys 2219 and 5083 are given in Table 3.2. The values presented in the table are the average of a minimum of three investigations.

Table 3.2 Mechanical properties of the base materials AA5083 and AA2219

<table>
<thead>
<tr>
<th>Base metal</th>
<th>Ultimate tensile strength (MPa)</th>
<th>0.2% Proof strength (MPa)</th>
<th>% Elongation</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA5083 – H321</td>
<td>303</td>
<td>255</td>
<td>28</td>
</tr>
<tr>
<td>AA2219 – T87</td>
<td>471</td>
<td>387</td>
<td>20</td>
</tr>
<tr>
<td>AA2219 – O</td>
<td>175</td>
<td>163</td>
<td>26</td>
</tr>
</tbody>
</table>
3.3 HARDNESS TESTING

Hardness testing was conducted using a Vickers hardness testing machine (Figure 3.4), using a 100 g load applied for 15 s. Welds were tested for hardness in the as welded condition. The results were averaged over a minimum of 5 tests per sample; more tests were conducted if required to reduce the standard deviation. Any obviously spurious results were rejected and testing was repeated.

![Figure 3.4 Vickers hardness testing machine](image)

3.4 TENSILE TESTING

Transverse tensile specimens with a gauge length of 25 mm and a width of 6 mm (overall length: 100 mm) were prepared from the weld coupons in an as-welded condition. Room-temperature tensile tests were conducted on three samples as per ASTM E8 (2010) on a universal tensile testing machine (Figure 3.5). The wire cut EDM was used to cut the smooth profile tensile specimens. To minimize the machining error (noise), three
specimens were prepared at each level of the designed matrix, and the average of the three results is presented. The dimensions of the tensile specimen are shown in Figure 3.6. The prepared tensile specimens were subjected to the tensile test and their ultimate tensile strengths were evaluated. The yield strength values presented were 0.2% proof strength values as computed by the computer program that controls the machine.

Figure 3.5 Tensile testing machine

Figure 3.6 Dimensions of the tensile specimen (mm)
3.5 PITTING CORROSION TESTS

A software based PAR basic electrochemical system (Figure 3.7) was used for conducting potentiodynamic polarization tests to study the pitting corrosion behavior of the welded zone. The standard calomel electrode (SCE) and carbon electrode were used as the reference and auxiliary electrodes respectively. All the experiments were conducted in 3.5% NaCl solutions, with the pH adjusted to 10. The potential scan was carried out at 0.166 mV/sec with an initial potential of – 0.25 (OC) SCE to the final potential of pitting. The exposure area for these experiments was 1 cm². The potential at which the current increases drastically was considered as the critical pitting potential (E_{pit}). Specimens exhibiting relatively more positive potential, (or less negative potential) were considered as those with better pitting corrosion resistance.

![Figure 3.7](image)

**Figure 3.7 (a) Basic electrochemical system  (b) Electrochemical flat cell**

3.6 GENERAL CORROSION TESTS

The salt fog apparatus (Figure 3.8) was used for testing the corrosion behavior of the welded zone. The experimental set up is shown in Figure 3.8. This apparatus consists of an enclosed glass chamber with square
plastic rods to hang the specimens. Two nozzles, viz., one for air and one for water, are present within the chamber. The air nozzle is connected to the air compressor and the water nozzle is connected to a can containing the NaCl solution. The air compressor supplies the air to the salt fog apparatus at the required working pressure. The nozzles are placed 90° apart from one another, such that a NaCl fog is created within the chamber. The specimens were tied up in the chamber with a non-corrosive polymer thread and hung in such a way that they were held at an angle of 7 to 14° parallel to the flow of the fog from the nozzles. The constant flow rate is set within few minutes the cloud of fog is created, and all the specimens are subjected to a constant flow of NaCl droplets striking their surfaces. Every 16 hours (for 48 hours) the specimens, depending upon their batch were removed from the chamber, and cleaned with acetone to remove any rust that may have been formed on the surface of the specimens.

Figure 3.8  Arrangement of corrosion test specimens in a salt fog apparatus
3.7 **OPTICAL METTALLOGRAPHY**

A detailed weld microstructural examination was carried out, using the optical microscope (Leica, DMLM) (Figure 3.9) on both the top surface and transverse sections of the samples. The samples were polished with emery paper and disc cloth to remove the scratches. The polished surfaces were etched with Keller’s reagent. The microstructures were recorded using a digital camera, and Image analysis software connected to the Metallurgical microscope. The etched specimens were primarily examined for shape and size of grains. The grain sizes were measured with the help of a graduated eye piece.

![Stereo microscope](image)

*Figure 3.9 Stereo microscope*
3.8  **SCANNING ELECTRON MICROSCOPY (SEM)**

SEM micrographs were taken in the secondary electron image mode at 15kV and 20kV. The energy dispersive X-ray (EDX) analysis was carried out to quantify the elements of Cu and Al, and Mg and Al using ZAF software. Scanning electron microscopy (SEM) was done on both the top surface and transverse section of the welds. Samples were metallographically prepared and etched before the SEM-EDX analysis. In the case of fractographs, the fracture surfaces were covered with tape, and kept safe from any physical damage that could occur to the surface.

3.9  **ELECTRON PROBE MICRO ANALYSIS (EPMA)**

The Electron Probe Micro Analysis (Cameca MBX) was used for the qualitative chemical analysis of different phases, by elemental X-ray mapping and by X-ray line scans technique. The line scan technique was also used for studying variations of the solute content (Cu and Mg) across the grains.

3.10  **TRANSMISSION ELECTRON MICROSCOPY (TEM)**

The TEM uses the same principles as the SEM, in replacing light with electrons to increase the resolution. However, in the TEM, the electrons are not measured escaping from the surface of the sample, but are transmitted through it, and projected on to a phosphorus screen below. This requires samples to be thin enough to be electron transmissive, a width of less than a few hundred nanometers. Samples could then be resolved at higher magnifications than was possible using the SEM. In addition, by focusing the beam onto a different plane, diffraction patterns from the samples could be obtained and recorded.
There were several stages involved in the preparation of the samples for the TEM. Sections were cut from the welded zones of the friction stir welds, using the EDM wire cut to the approximate dimension of 15mm x 6mm x 0.3mm. These were then attached by the TEM wax to a block, and ground by hand to ~0.1mm thickness. A Jet Polisher was then used to create a dimple on each side of the disc, which became enlarged and formed a hole surrounded by a thin, electron transmissive region.

The Jet Polisher required a 30% nitric acid solution in methanol, cooled to ~30°C, and was operated at a potential of 4.5V. Care was taken to ensure that the temperature remained at ~30°C; as the solution was allowed to warm, it became potentially explosive. The microscope used for the TEM analysis was the Phillips CM20 with EDX capabilities. The samples were loaded into the machine on a double tilting, low background, beryllium holder. Where appropriate, The EDX analysis was undertaken to find the composition of the specific objects where appropriate. The EDX was operated at ~1000 counts/sec at an operating voltage of 200kV, and with a live time of 100 seconds.