For grain refinement, the grain growth plays significant role in the reduction of the grain size of the SZ produced during FSP. Grain growth phenomena is purely dependant on the process temperature. Schematic illustration of this phenomena is explained in Figure 6-1. The heat input conditions during FSP is mainly dependent on the tool rotation, travel speed, tool tilt angle, and tool geometry. There are two ways to change the heat input during FSP. First, with changing of above mentioned factors but that may affect the stirring action and hence material flow, which may affect resulting properties. Second, the most effective way is to apply cooling medium on the friction stir processed region without changing the process parameters or tool geometry. The use cooling medium will make the low heat input condition during FSP, that will inhibits the grain growth in order to achieve very fine grain size. Since fine grain size is preliminary criteria to achieve superplasticity, the superplastic behavior is enhanced by reducing the grain size.

![Figure 6-1 Schematic illustration of the grain growth under different heat input conditions during FSP: (a) Coarse grains at high heat input or natural cooling FSP, (b) Fine grains at low heat input or hybrid with active cooling approach FSP](image-url)
Therefore, Hybrid FSP can be considered as variant of FSP to enhance grain refinement and superplastic behavior, in which the external cooling is employed during the process. For achieving superplasticity the primary requirement is the fine grain stable microstructure structure. The external cooling can be applied by various means such as underwater or submerging the workpiece in water (Liu and Feng, 2013; Feng et al., 2013; Hofmann and Vecchio, 2005; Pang et al., 2016) and active cooling of the workpiece by throwing the cooling medium onto the processed zone formed during FSP (Liu and Ma, 2008b; Su et al., 2005a). Orozco-Caballero et al. (2013) investigated FSP of AA7075 for an excellent combination of LTSP and HSRS using refrigerated backing anvil.

As per the author’s best knowledge the hybrid FSP approach of using different active cooling mediums need to be explored for obtaining superplasticity in AA7075. Hence, the aim of this study is to investigate hybrid FSP using active cooling approach on the superplastic behaviour of AA7075. For this, the different cooling medium such as compressed air, water, and CO₂ are used to perform hybrid FSP. Cooling rate of each of the cooling medium was also varied to obtain the least possible process temperature. Large series of experiments (series 4) were conducted as described in section 3.2.3 of Chapter 3 and the FSP conditions of all the experiments is tabulated in Table 3-7.

The surface morphologies of all FSP samples is shown in Figure 6-2. From the surface appearance, there was no defects found for all the samples. Surface tunnel or galling are the prominent defects in FSP due to insufficient heat generation (cold conditions), but it was not observed in spite of active cooling during as well as after FSP. Also, surface morphology of all FSP samples revealed the formation of the “onion ring” structure in the processed zone. This surface appearance was clear evidence of effective active cooling approach in hybrid FSP samples. From all hybrid FSP samples, the sample produced at the least processing temperature in each cooling medium was compared against the natural cooling sample.

Sample A2, W3, and C1 from compressed air, water, and CO₂ cooling samples reported least process temperature during FSP. Hence, these samples were compared with natural cooling sample in order to study the effect of active cooling on the grain refinement. Table 6-1 shows summary of the FSP samples produced at minimum process temperature in each cooling
medium category. The highest processing temperature was observed for natural cooling and lowest for CO₂ cooling sample.

Figure 6-2 Surface morphologies after FSP: (a) natural, (b) compressed air, (c) water, and (d) CO₂ cooling
Table 6-1 Summary of samples reported minimum process temperature at each cooling medium

<table>
<thead>
<tr>
<th>Samples</th>
<th>Sample ID</th>
<th>Maximum temperature (T_{\text{max}}), °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>N</td>
<td>N</td>
<td>340</td>
</tr>
<tr>
<td>A2</td>
<td>A</td>
<td>305</td>
</tr>
<tr>
<td>W3</td>
<td>W</td>
<td>275</td>
</tr>
<tr>
<td>C1</td>
<td>C</td>
<td>240</td>
</tr>
</tbody>
</table>

6.1 TEMPERATURE DISTRIBUTION

Figure 6-3 displays the temperature distribution on AS and RS for all the samples. The temperature values were recorded close to the TMAZ, as thermocouples were located 3 mm away from the shoulder surface. Although the temperature distribution in Figure 6-3 do not show the exact temperature in the SZ, the temperature difference among the various cooling medium samples can be understood. No significance difference in temperature was observed between AS and RS for all samples. The process temperature during FSP is dependent on the heat index (ratio of rotational speed to travel speed). Even though HI was kept constant, the difference in the temperature profiles were noted because of the different modes of active cooling. Important observations were made from temperature profile of all four samples.

For Curve N (natural cooling), the temperature gradient for heating is steeper than for cooling. While curve W (water cooling), and curve C (\(\text{CO}_2\)) reported steeper temperature gradient for heating as well as cooling. However, Curve A (compressed air cooling) found that temperature gradient for cooling is less steep in comparison to curve W and C; but more steeper than curve N. The variations in temperature distribution among the samples was attributed due to the different cooling medium used during FSP.
Table 6-2 shows the peak temperature and cooling rate achieved for producing all samples. The peak temperature of 340, 305, 275, and 240 °C found during FSP with natural, compressed air, water, and CO₂ cooling respectively. The lowest peak temperature value was obtained in sample C, while the highest peak temperature was found in sample N. While in case of sample W and A, the temperature of sample W was lower than the sample A. The cooling rate (CR) for all samples have been tabulated in Table 6-2.

<table>
<thead>
<tr>
<th>Samples</th>
<th>T max</th>
<th>T f</th>
<th>T p –T f</th>
<th>t c</th>
<th>CR</th>
</tr>
</thead>
<tbody>
<tr>
<td>N</td>
<td>340</td>
<td>280</td>
<td>910</td>
<td>0.31</td>
<td></td>
</tr>
<tr>
<td>A</td>
<td>305</td>
<td>245</td>
<td>506</td>
<td>0.48</td>
<td></td>
</tr>
<tr>
<td>W</td>
<td>275</td>
<td>215</td>
<td>150</td>
<td>1.43</td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>240</td>
<td>180</td>
<td>158</td>
<td>1.14</td>
<td></td>
</tr>
</tbody>
</table>

T_p = Peak temperature, °C
T_f = Final temperature after cooling, °C
T_p –T_f = Temperature difference, °C
t_c = cooling time, seconds
CR = cooling rate, °C/sec
Figure 6-3 Temperature distribution during process: (a) advancing side, (b) retreating side.
The calculation of CR is based on temperature difference ($T_p - T_f$) and cooling time ($t_c$). The CR of 0.31, 0.48, 1.43, and 1.14 reached for the sample N, A, C, and W respectively. The lowest value of CR was achieved for sample N, while the highest was for sample W. Sample C reported the lowest temperature value but the cooling time taken was almost similar to the sample W as shown in Table 6-2. Therefore the CR for sample W was higher than the sample C. It was well understood that hybrid FSP with active cooling approach can be characterized by the value of cooling rate and the peak temperature during FSP. Figure 6-4 represents the graph of comparison between cooling rate and peak temperature for all samples. This graph indicates that cooling rate can be increased by mainly lowering the peak temperature with the help of different cooling mediums.

![Figure 6-4 Comparison of peak temperature and cooling rate](image.png)

**6.2 MICROSTRUCTURAL BEHAVIOUR EXAMINATIONS**

Figure 6-5 shows the macrostructure of the cross-section of FSP samples. The SZ in form of basin shape was observed with widen top surface because of intense deformation and frictional heat produced from the shoulder contact during FSP.
Figure 6-5 Macrostructure of the FSP samples: (a) natural cooling, (b) air cooling, (c) water cooling, and (d) CO\textsubscript{2} cooling.

The SZ is generated due to the direct interaction of the tool pin and the work material, while TMAZ is not influenced by the tool pin but affected by the plastic strain and thermal history from the neighbouring SZ. Furthermore, the full penetration without any defect or discontinuity was observed in top and bottom surface for all the samples. Further microstructure behavior was studied for the only SZ, the area of interest. The optical microstructure of the as received base metal AA7075 was characterized by pancake shape grains in elongated nature as shown in Figure 6-6 (a). FSP is characterized by extreme plastic deformation with the material flow around the rotating tool pin. The work material also undergoes frictional deformation due to tool and work contact during the process.

Hence, FSP produced the considerable amount heat is generated, which resulted into recrystallized fine equiaxed grain microstructure in the SZ at all cooling conditions as displayed in Figure 6-6 (b-e). No processing flaws such as micro void or cavitation were observed in the SZ microstructure of all samples. This was mainly due to an appropriate material flow which is governed by the suitable process parameters. Process parameters such as tool rotation speed, travel speed, tool geometry, plunge force, and process temperature affect the material flow behaviour and the degree of microstructural refinement.

In current study, the process temperature was varied by using different cooling medium for active cooling during FSP. Therefore, the different level of grain refinement obtained at different processing temperature. It is easy to identify from the SZ microstructure that the grain
refinement of active cooling FSP samples as shown in Figure 6-6 (c-e) is higher than the normal FSP, Figure 6-6 (b).

Figure 6-6 Optical micrographs of the SZ: (a) base metal, (b) natural cooling, (c) air cooling, (d) water cooling, and (e) CO$_2$ cooling.
The SZ of all samples was further examined at higher magnification by scanning electron microscopy (SEM). The SEM images of the SZ at different cooling conditions is displayed in Figure 6-7. The grain boundaries are clearly visible in all samples without any distortions. Also, the orientations of the grain boundaries are very random, especially for active cooling samples as shown in Figure 6-7 (b-d).

The average grain size of the SZ was measured of 4.12, 3.00, 2.64, and 1.96 µm for the sample N, A, W, and C respectively as shown in Figure 6-8. It has portrayed the downward trend of the average grain size while moving in order of N, A, W, and C. The reduction in the grain size was mainly attributed to the peak temperature and cooling rate during the FSP. The sample C was produced at the lowest process temperature of 258 °C, while the sample W was produced at the highest cooling rate of 1.43 °C/ sec (Figure 6-4).

The lower peak temperature and the higher cooling rate during the FSP could hinder the grain growth of recrystallized fine grains and consequently the reduction of the grain size was achieved. This grain refinement is anticipated for the two reasons. First, is to enhance the room temperature mechanical property, as predicted by the Hall-Petch relation (Hall, 1951; Petch, 1953). The second reason is to obtain the better superplastic behavior, as the foremost requirement for achieving structural superplasticity is a fine grain size of less than 15 µm (Mishra et al., 2014). The obtained grain size of all the samples are well below the require size for achieving the superplasticity. However, the key requirement of grain size is necessary but it does not guarantee the superplastic behavior. The fine grain microstructure has to be stable at high temperature, otherwise superplastic elongation could be reduced in significant amount.

Energy dispersion spectrography (EDS) was performed on the SZ to know the presence of the strengthening phase particles. Figure 12 represents EDS analysis of the SZ for all samples. The EDS spectrum was taken at both grain boundary and grain matrix, but no significance difference was observed in obtained peaks. Hence, it was deduced that strengthening phase particles such as Zn and Mg and composed in form of MgZn2 phase were presented on grain boundaries and grain matrix and that would have given higher resistance to the deformation, as this phase is responsible for the remarkable precipitation hardening behavior of the 7xxx series alloy (Conserva et al., 1973).
Figure 6-7 SEM images of the SZ microstructure produced by different cooling medium: (a) natural, (b) compressed air, (c) water, and (d) CO\textsubscript{2} cooling.

Figure 6-8 Summary of the average grain size of the SZ
Figure 6-9 EDS analysis of the SZ produced at different cooling medium
6.3 HIGH TEMPERATURE DEFORMATION IN ORDER TO EVALUATE SUPERPLASTIC BEHAVIOR

The superplasticity in fine grained-microstructure aluminum alloys can be expressed by the constitutive relationship (Mishra et al., 1995):

\[ \dot{\varepsilon} = A D E b \exp\left( -\frac{84,000}{R T} \right) \left( \frac{\sigma - \sigma_0}{E} \right)^2 \left( \frac{b}{d} \right)^2 \] ................ (Eq. 1)

where \( \dot{\varepsilon} \) is strain rate, \( A \) is a dimensionless constant, \( D \) is appropriate diffusivity constant, \( E \) is Young’s modulus, \( b \) is Burger’s vector, \( k \) is Boltzmann’s constant, \( T \) is the absolute temperature, \( R \) is the gas constant, \( d \) is the grain size, \( \sigma \) is the applied stress, \( \sigma_0 \) is the threshold stress. Eq. 1 shows that grain size plays significant role in the enhancement of superplastic elongation at optimum strain rate and temperature. Hence, decreasing the grain size in the microstructure is considered an effective method for achieving the enhanced superplastic behavior.

The appearance of the tensile specimens after superplastic forming at the temperature of 400 °C and strain rate of 3x10^{-2} s^{-1} as shown in the Figure 6-10. Necking was observed at the fractured point in case of all the specimens. CO₂ cooled sample reported dominant behavior of necking at the fractured point among all the samples. Whereas natural cooled sample obtained the little necking at the fractured surface after high temperature deformation.

It was observed that all specimens reported superplastic behavior. Additionally, hybrid FSP samples achieved enhanced superplastic elongation than the natural cooling FSP. Among the hybrid FSP samples, the CO₂ cooled FSP sample obtained the highest elongation. The variation of superplastic elongation with different cooling medium is shown in the Figure 6-11.

The superplastic elongation of 233%, 367%, 467%, and 517% was obtained for natural, compressed air, water, and CO₂ cooling FSP tensile specimens, respectively. The elongation trend is upward, while moving from higher to lower processing temperature during FSP (sample N to C), as it can be seen in Figure 6-11.
This enhanced elongation can be well explained based on the cooling rate during FSP. The cooling rate in hybrid FSP greatly influences on resulting grain size followed by superplastic behavior. Orozco-Caballero et al. (2016) recommended high cooling rates to obtain the finest grain size. The cooling curve form the peak process temperature to room temperature is depicted in Figure 6-12. The hybrid FSP samples (A, W, and C) cooling curve was well behind the natural cooling FSP sample N. Additionally, sample W and C cooling rate curve is much more narrower than the sample A due to rapid and efficient cooling characteristics of water and CO₂ cooling medium.
Chapter 6 Hybrid FSP with active cooling approach

Figure 6-11 Variation of elongations with different cooling medium for FSP AA 7075 at 400 °C and strain rate of \(3 \times 10^{-3} \text{ s}^{-1}\)

Figure 6-12 Cooling rate curve for all FSP samples
The highest cooling rates inhibit grain coarsening more effectively than the lower cooling rate, which led to reduction in the grain size and consequently enhanced superplastic elongation. Therefore, sample C produced at the highest cooling rate achieved the smallest grain size of 1.96 µm and the highest superplastic elongation of 517%. On the other side, sample N produced at the lowest cooling rate obtained the larger grain size of 4.12 µm and the least superplastic elongation of 233%.

The actual stress–strain behavior of FSP samples SZ, produced at different cooling rate in form of relation between strain rate and temperature is represented in Figure 6-13. All the samples have experienced strain hardening during the initial period of deformations. Superplastic deformation can be optimized by minimizing flow stresses or maximum stresses. Flow stress value of ~ 16 MPa was reported for all FSP samples, as the flow stress is generally reduces at high temperature during superplastic deformation. This lower flow stress at higher temperature results to less stress concentration at grain boundaries, which results GBS as a mechanism superplastic deformation.

![Figure 6-13 Flow stress vs. elongation of FSP samples](image)

**6.4 SUMMARY**

Hybrid FSP with active cooling approach can be considered as one of the variant of FSP to enhance grain refinement and the superplastic behavior. The fine microstructure produced at the highest cooling rate resulted into enhanced superplastic elongation.
All hybrid FSP samples were produced at low heat input conditions without changing the process parameters (765 rpm, 31.5 mm/min, 2\(^\circ\)), as process parameters govern the heat input during FSP. The hybrid FSP proved the effectiveness of cooling medium to produce defect free processing zone as well as SZ at low process temperature.

The process temperature of 240, 275, 305, 340 \(^\circ\)C was observed for CO\(_2\), water, compressed air, natural cooling FSP samples, respectively. This was mainly due to rapid cooling characteristic of cooling mediums. The cooling rate of 0.31, 0.48, 1.43, 1.14 \(^\circ\)C/s was obtained in FSP samples, respectively. Cooling medium and rate of cooling rate are the variables, which vary the process temperature and cooling rate, which resulted into enhanced grain refinement. This combination of low process temperature and high cooling rate led to enhance grain refinement.

No influence of cooling approach on the strengthening phase elements was found that ensured hybrid FSP as an effective technique for material processing, as AA7075 alloy is precipitate hardening alloy. This reduced process temperature and increased cooling rate during hybrid FSP resulted into inhibition of the grain growth and consequently reduction in the grain size. Therefore, grain size of 1.96, 2.64, 3.00, 4.12 \(\mu\)m achieved for CO\(_2\), water, compressed air, natural cooling FSP samples, respectively.

The superplastic elongation of 517\%, 467\%, 367\%, 233\% was obtained for CO\(_2\), water, compressed air, natural cooling FSP samples, respectively. Hybrid FSP sample with CO\(_2\) cooling achieved the highest elongation because of the smallest grained microstructure produced at higher cooling rate during FSP.
Chapter 6 Hybrid FSP with active cooling approach