Chapter 06
Summary and suggestions for future work

6.1 Introduction

The chapter illustrated important observations from the thesis and comprised suggestions for future work to rectify some of the issues associated with the current analyses.

6.2 Summary

A detailed study on structure-property correlations of the heat-treated 7075 Al alloy at T4, T6, T73, and T7352 tempers is carried out. The in-depth precipitation behavior was studied at 7352 using the bright field and the dark field TEM imaging. The chemistry was analyzed using the HAADF STEM EDS elemental mapping. The dislocation behavior on account of the partial tensile deformation (respective to 2%, 6%, and 10% partial tensile true straining) is studied using weak-beam dark-field TEM imaging. Stress corrosion cracking (SCC) is studied in T6, T73, and T7352 tempers. The 7075 Al alloy is thermo-mechanically processed, adopting the processing routes of solution quenching (470°C for 1hr) followed by cold deformation to three different amounts (15%, 30%, and 45%), after ageing at 120°C for 24 hrs (SQ+CR+PA), the microstructure evolution and texture characterization are done and flow behaviors were studied. The AA7075T7352 is friction stir processed (FSPed) at 1 pass, and multiple passes (up to 3 passes). The microstructures and textures in different zones (NZ, TMAZ, and HAZ) are studied. The depth-wise dislocation characterization is done. The chapter but some of the significant findings are given below:

6.2.1 Precipitation behavior

The precipitation behaviour of AA7075T7352 displays randomly distributed η' and η phases. Added to the intermetallics of Al_2Cu, Al_2CuMg, and dispersoids of the Al_3Zr. The precipitation process follows the sequences of supersaturated solid solution(α)- η'-η on account of their interfacial energy respective to the matrix. The η' precipitates are separately nucleated. On the other hand, η' and η phases follow both the separated nucleation as well as those of the in-situ nucleation. The free η' and η are separately nucleated. Whereas, η' and η precipitates joining at the edges follow the in-situ nucleation mechanisms. The rod type of η' precipitate displays preferred nucleation along 110 of the α-Al. Whereas the plate type of two intersecting η shows the preferred nucleation along 110 and $1\bar{1}0$ of α-Al. The co-existence of the Al_3Zr dispersoid into some of the 220 spots of α-Al is also observed. The streaking along $1\bar{1}0$ of α-Al suggests the presence of the stacking fault in materials that form during nucleation of the η phase. The dislocation entrapment in η phase at T7352 and the formation of dense dislocation walls (DDWs) are other major observations.

The precipitation was not observed in solution-quenched (SQ) and the naturally-aged (NA) 7075 Al alloy because room temperature (25 °C) and room temperature aging (25°C, for 3 months) are not sufficient for the decomposition of the supersaturated solid solution (α). The peak aged 7075 Al alloy forms the η', and the η precipitates on account of the diffusion-assisted nucleation and the growth process. This also arises because elevated temperature aging (120°C for 24 hours) is sufficient for the decomposition of the supersaturated solid solution (α). The η precipitates are continuously distributed in the grain boundary without any spacing and with very little precipitate-free zone. The over-aged 7075 Al alloy at T73 temper, shows formation of the η' and η phases and the absence of the GP zone, because the thermal stability of the GP zone is limited up to 150°C.

The precipitation behavior of the partially tensile deformed alloy at 2% tensile true strain shows dislocation shear-assisted partial dissolutions of the η' and η. The 6% partial deformation illustrates partial dissolution of the η', but η remains unaffected. On the other hand, 10% partial deformation depicts higher precipitation of the η and partial dissolution of the η' by 10% partial tensile true straining. The thermo-mechanically processed alloy at TMP-1 shows an increased volume fraction of the soft phase. On the other hand, enhanced fractions of the η' and η precipitates are observed in the TMP-2 and the TMP-3 respectively. The coupling effect of dislocation and precipitates, as well as those of the dislocation entrapment in η precipitates, are mainly observed in TMP-2 and TMP-3.

The η, at 2θ = 20° is fully dissolved after 1 pass FSP. On the other hand, *η' and Al_2CuMg* phases are partially dissolved, and growth of the *Al_2Cu (θ)* phase is noticed. The dissolutions occur by the combined effect of the dislocation shear-assisted dissolution and the friction stir-assisted thermal effect. The $Al_{23}CuFe_4$ formation is observed due to the pipe-diffusion and the dislocation sweeping mechanisms. The *η and η'* phases are both partially dissolved after 2 passes of FSP. In contrast, re-precipitation of *η* occurred with a slightly shifted 2θ peak position of 20°, which is noticed with different chemistry but the same crystal structures after the 3 passes. Precipitation of the *η' and η* phases are also observed, but the size and volume fractions of the η' precipitate are reduced. On the other hand, the size of *η* remains unchanged. Such dissolutions and coarsening as well as the growth of precipitates following FSP would likely help to obtain the improved mechanical properties of the alloy. The grain boundary precipitates are also dissolved followed by the FSP. After, 1pass FSP, the NZ shows dynamically recrystallized but equi-axed fine grains. The TMAZ depicts slightly elongated grains and coarse-grain microstructures in the HAZ. On the other hand, the base metal remains unaffected after FSP. Similar microstructural features are also observed after the 2pass and the 3pass of FSP.

6.2.2 Dislocation behaviors

The AR alloy in T7352 temper depicts the dense dislocation walls *(DDWs*), and the dislocation entrapment in the interface of η and α-Al due to 10% compressive deformation. The 2% partial tensile deformation shows forest dislocation and tangled dislocation structures. Partial deformation respective to 6% tensile true strain depicts low and high density of Taylor lattice. On the other hand, 10% partial tensile deformation shows a low and high density of dislocation cell structures. The dislocation re-arranges themselves due to variations of strain on account of the plastic deformation in room temperature. The top of NZ after 1pass FSP shows the dislocation peening and parallel array of dislocation structures. The middle portion depicts the dislocation network, as well as the parallel array of dislocation. On the other hand, the bottom of the processed zone displays sub-grain formation or the dislocation cell type of structures.

6.2.3 Texture characteristics

The heat-treated alloy in SQ, and natural-aging (T4) conditions show the evolution of the α-fiber texture, as well as the Goss {011} <100> texture component with an intensity of 14.5 random. On the other hand, thermo-mechanically processed alloy at TMP-1 shows increased intensity of Goss {011} <100> texture of 20 times. The intensity of Goss {011} <100> texture further increases to 25 times random at TMP-2, and 28 times random in TMP-3. Added to this, Copper {112} <111>, Rotated cube {001} <110>, Rotated goss {110} <110> and S {123} <634> texture components are also observed. Heat treatment does not influence the texture component. Whereas, FSP randomizes the texture components. The texture intensity of base metals (BM) is 28 times random. After 1pass FSP, the texture intensity is 2.5 times random. After 2pass FSP, the texture intensity is 1.6 times random. At the end of 3pass FSP, the texture intensity is 2.1 times random. The Goss {011} <100>, Rotated goss {110} <110>, Rotated cube {001} <110>, and S {123} <634> texture components are another noteworthy feature after FSP. The grain size of base metals (BM) or as-received (AR) alloy is 54±3 μm. There is one order magnitude of grain refinement of 3.8±0.5 μm after 1pass FSP. After 2pass FSP, the grain size is 3.5±0.6 µm. Moreover, at the end of 3pass FSP, grain size further reduces to 2.6±0.4 µm. The low-angle grain boundary fraction (LAGBF), and high-angle grain boundary fractions (HAGBFs) of as-received alloy are 85% and 15% respectively. The HAGBF after 1pass FSP increases to 49% due to dynamic recrystallization. After 2pass FSP the HAGBF is 47%, which further decreases to 30% after 3pass FSP, but it was more than the AR condition. The KAM value of BM metal is 0.52, which increases to 0.56 after 1pass FSP, and thereafter decreases to 0.45 in 2pass FSP. The KAM value further increases to 0.65 for 3pass FSP. The co-incidence site lattice (CSL) boundary fractions (Σ3, Σ5, Σ9) for AR alloy are 0.75, 0.19 and 0.35 respectively. After 1pass FSP, CSL boundary fractions (Σ3, Σ5, Σ9) increase to 1.25, 0.65, and 0.55 alternatively. This further increases to Σ3 =1.35, Σ5 = 0.95, and Σ9 =0.85 after 2pass FSP. On the other hand, CSL boundary fractions slightly decrease to Σ3 =1.0, Σ5 = 0.65, and Σ9 = 0.55 after 3pass FSP.

6.2.4 Residual stress and hardness

The residual stress in SQ and T4 temper is nearly the same (-57±8 MPa) due to the absence of precipitates. On account of the formation of metastable *(η')*, and the equilibrium *(η)* precipitates, the compressive nature decreases to -48±5 MPa, in the T6 temper. After two steps of the aging process at T73 temper, the compressive nature further reduces to -41±9 MPa. The compressive behavior further enhances to -96±7 MPa, at T7352 temper due to 10% compressive deformation. Thermo-mechanical processing further increases the compressive nature of residual stress, -112±7 MPa for TMP-1, -158±3 MPa for TMP-2, on the other hand, -162±5 MPa in the case of TMP-3.

Friction stir processing (FSP) further changes the nature of residual stress. After 1 pass FSP, the RS in NZ is -132±3 MPa, -110±6 MPa in the HAZ, whereas -96±8 MPa in the unaffected base metal region. While increasing the number of passes, after 2 passes of FSP, the compressive nature of RS increases to -153±7 MPa, due to further dynamic re-crystallization assisted grain refinement. After 3 passes of FSP, residual stress further slightly decreases to -148±4 MPa due to re-precipitation of the η. The measured hardness for solution quenched *(SQ),* and naturally aged (T4) alloys are similar, 118±5 Hv, because metastable *(η')* and equilibrium *(η)* precipitates are not formed in these two conditions because room temperature aging is not enough for diffusion assisted precipitation. Upon aging at T6 temper (120°C for 24hrs) hardness increases to 212±8 Hv due to formation of the metastable precipitates. Upon over-aging at T73 temper, the hardness further decreases to 160±4 Hv due to the dissolution of the GP zone, and coarsening of precipitates.

In the T7352 temper hardness was slightly reduced to 151±7 Hv due to over-ageing assisted softening. After thermo-mechanical processing (TMP) the hardness value further increases upto 159±4 Hv for TMP-1. On the other hand, 171±3 Hv, and 178±7 Hv in the case of the TMP-2 and TMP-3 respectively due to deformation-assisted enhanced dislocation density. Following the FSP, variation in hardness value is observed in different regions of the processed alloy and they follow the W shape profile of changes in hardness after measuring the hardness in 5mm intervals from the center of the nugget zone to the base metal region of both the advancing as well as the re-treating side. The average hardness at the top of the nugget zone (NZ), after 1 pass FSP is 155±4 Hv. After the 2 passes and 3 passes of FSP, average hardness changes to 193±7 Hv, and 180±5 Hv alternatively.

6.2.5 Mechanical properties and Flow behavior

The YS and UTS values for T4 are 384±4 MPa and 468±5 MPa. The respective values for T6 temper are 464±3 MPa and 480±7 MPa. For T73 temper, the YS is 448±5 MPa, and the UTS value is 485 ±9 MPa. On the other hand, the YS value for T7352 is 425±7 MPa and the UTS value is 472±6 MPa. The total elongation value for T4 temper is high (25±2%) due to the absence of precipitates. After peak aging (T6) the ductility reduces to 16% due to precipitation but in T73 temper ductility again increases due to over-aging assisted dissolution of the GP zone and formation of the coarse precipitates. In T7352 temper, ductility further decreases due to the combined effects of the formation of the GP zone, as well as, the dense dislocation walls (DDWs). After thermo-mechanical processing (TMP) strength and ductility both decrease in the lower deformation at 15% CR, while strength increases but ductility decreases at 30% CR. On the other hand, improvement in the strength, as well as ductility are observed after 45% CR at TMP-3, which would help design the new alloys for aerospace structural applications. After 1 pass FSP, the strength decreases but significant enhancement in ductility is observed. After 2pass FSP strength and ductility both decrease due to the formation of the dislocation tangles and dissolution of the strengthening precipitates. On the other hand, strength and ductility both increase marginally but remain close to AS alloy due to re-precipitation of the η with different chemistry but the same crystal structures. After FSP (1pass, 2pass, and 3pass) the alloy depicts the Swift flow behavior, but pre-strain *(ε_0)*, and work hardening exponent *(n)* are different due to changes in the size, and chemistry of precipitates. The AR alloy at T7352 follows the Ludwigson flow behavior. On the other hand, SQ, T4, T6, and T73 depict the best fittings with the Swift mathematical model. The thermos-mechanically processed alloy TMP-1 shows the best fittings with the

Swift curve. Whereas, TMP-2 and TMP-3 depict the best fittings with Ludwigson flow behavior. Friction stir processed alloy after 1pass, 2pass, and 3pass FSP show the best fittings with Swift flow behavior. However, their work hardening exponents *(n_1, n_2)* and strength co-efficient (K_1, K_2) values are different for the Ludwigson fitted curve, and the strength coefficient *(K)* and the pre-strain *(ε_0)* values for the Swift fitted curves.

6.2.6 Stress corrosion cracking

The stress corrosion cracking susceptibility (Iscc) for T6 temper is high (35 %) due to continuous grain boundary precipitates. At T73 temper, the I_{SCC} (16%) reduces because of dis-continuously distributed large size of grain boundary precipitates. Moreover, the Iscc value further decreases to 14% in T7352 temper, due to the entrapment of the η phase by dislocation at the interfaces of η and α-Al matri

9.3 Suggestions for the future work

1. Extensive study of nucleation and growth behavior of precipitates using the HR TEM imaging in STEM mode.
2. Detailed study of stress corrosion cracking (SCC) behavior and depth-wise microstructural evolution after 2-pass, and 3-pass FSP.
3. Depth-wise structural, and dislocation characterization, after 2pass and 3pass FSP.
4. Extensive study of changes in the size and chemistry of precipitates using TEM in various zones after FSP.
5. The flow curve behavior and texture characterization of SQ+FSP+PA, and FSP+PA alloys.
6. Nature of the dislocation in NZ of the processed alloy using the HR-TEM imaging
7. HR TEM imaging in STEM mode in different zones for the atomic contrast correlations.
8. Nature of the grain boundary precipitates after thermo-mechanical treatment and friction stir processing.
9. Detailed study of strengthening mechanisms of heat treated (T4, T6, T73, T7352), thermo-mechanical processing (TMP), and friction stir processed (FSPed) alloy and their correlations with the microstructures