

## MICROSTRUCTURE AND MECHANICAL PROPERTIES OF A FRICTION STIR PROCESSED Al-Zn-Mg-Cu ALLOY

Margarita Vargas<sup>1</sup> and Sri Lathaba<sup>2</sup>

Friction stir processing (FSP) was performed on AA 7075-T6, a heat treatable high strength Al-Zn-Mg-Cu alloy. The two main FSP parameters, the tool rotational ( $\omega$ , RPM) and travel speed ( $v$ , mm/min), were varied systematically in order to understand their influence on the microstructure and mechanical properties of the processed zone. It was demonstrated that the range of FSP parameters used did not significantly influence the nugget zone grain size. However, the mechanical properties of the processed zone varied significantly. For instance, at a given rotational speed, increasing the travel speed increased the microhardness of the nugget (stir) zone; for a given travel speed there appeared to be an optimum rotational speed which resulted in the highest microhardness. For all parameters tested, the processed zone displayed lower tensile strength, but significantly higher ductility when compared to the as-received alloy. The variation in tensile strength with changes in rotational and travel speed followed the same general trend as in the case of microhardness. In all cases, the ductility was within the range 20-25% elongation. The observed mechanical properties could not be explained as a direct consequence of grain refinement, by the Hall-Petch relationship. It is suggested that these are a result of the complex interactions between the FSP thermo-mechanical effects and the processes of dissolution, coarsening and re-precipitation of the strengthening precipitates in this alloy.

**Keywords:** friction stir processing, 7xxx aluminium alloy, mechanical properties.

1st TMS-ABM International Materials Congress, July 26-30, 2010 Rio de Janeiro, Brazil

<sup>1</sup> Postdoctoral Research Fellow, CSIRO Process Science and Engineering, Clayton, Victoria, Australia.

<sup>2</sup> Principal Research Scientist, CSIRO Process Science and Engineering, Clayton, Victoria, Australia.

## 1. Introduction

Friction stir processing (FSP) is a novel solid-state technique which enables targeted local processing of metallic components for microstructural refinement and homogenisation as well as elimination of defects, leading to an enhanced global performance. During FSP, a rotating tool with a specially designed pin and shoulder is plunged into the surface of the work piece and traversed along a path to cover the region of interest. The frictional heat generated between the rotating tool and the workpiece softens the material and with the mechanical stirring caused by the pin, intense plastic deformation occurs within the process zone, resulting in a recrystallised ultrafine-grained structure [1-3].

The two main process parameters for FSP are the tool rotational speed ( $\omega$ , RPM) and the travel speed ( $v$ , mm/min). For a given tool geometry and depth of penetration, it has been observed that the peak temperature generated within the stir zone is a strong function of the rotational speed, while the rate of heating is related to the travel speed, and that the heat input per unit length or line energy is directly related to the ratio  $\omega/v$  [2,4,5]. The temperature distribution within and around the stir zone and the associated thermal cycles influence the microstructure including the grain size and texture, as well as the coarsening and dissolution of precipitates and their re-precipitation in heat treatable aluminium alloys, which, in turn, control the mechanical properties of the processed region [2,4,5]. A knowledge of the relationship between process variables and the resulting microstructure and mechanical properties is highly desirable when using FSP for targeted and tailored microstructural modification in metallic components. Although detailed microstructural examinations of friction stir processed age hardenable aluminium alloys of the 7xxx series have been published, many of these do not provide information on the tool design and process parameters [5-7] and the investigation of the relationship between microstructure and properties of these alloys has continued to generate interest [8,9]. Accordingly, in this paper, we present the results from a systematic investigation of the effect of tool rotational speed and travel speed on the microstructure and mechanical properties of AA 7075-T6 processed by FSP.

## 2. Experimental Program

The material used in the study was AA 7075-T6, supplied as a 3.1 mm thick sheet. The chemical composition (in wt. %) as stated in the manufacturer's certificate was Al-5.7Zn-2.6Mg-1.7Cu-0.27Fe-0.19Cr-0.05Si-0.01Mn-0.02Ti. The ultimate tensile strength of the material, also from the manufacturer's certificate was 565 MPa, the yield strength was 499 MPa and the elongation was 10.3%. Test coupons, 200 mm  $\times$  100 mm, were extracted from the sheet such that the longer dimension coincided with the rolling direction. FSP was performed on a vertical milling machine using a stir tool made of H13 steel (Fig. 1(a)), with a shoulder diameter of 16 mm and a conical threaded pin with a tip diameter of 6 mm and 2.7 mm length. A single stir pass,  $\sim$  160 mm long, was made on each test plate. Three rotational speeds were used: 800, 1000 and 1500 RPM. In each case, three travel speeds were used: 39, 57 and 81 mm/min. Two additional travel speeds, 121 and 171 mm/min, were used at the highest rotational speed of 1500 RPM. The parameters used resulted in defect-free processed zones.

Samples for microstructural examination and microhardness testing were extracted transverse to the stirring direction as indicated in Fig. 1(b), and prepared using standard metallographic techniques. Microhardness testing was conducted at a load of 100 g, with a dwell of 15 s. Hardness scans were made in a grid pattern covering both the stirred area as well as the unaffected base material on either side of the pass; the scan started at 0.5 mm from the top surface of the plate and traversed both the length and the thickness of the section at intervals of 0.5 mm, resulting in a total of over 400 indentations. The samples were etched with Barker's reagent for 2 minutes at 20 V and examined using an optical microscope under polarised illumination.

Two sub-size tensile specimens were machined from each of the test coupons, within the nugget zone, parallel to the stir direction, as shown in Fig. 1(b). The total length of the specimens was 38 mm, and the gage dimensions were 10 mm (L) x 3 mm (W) x ~2.5 mm (T). The tensile tests were conducted at room temperature, at a cross head speed of 1 mm/min.

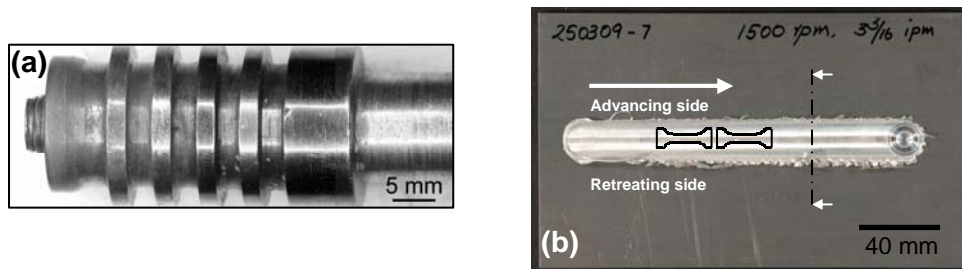


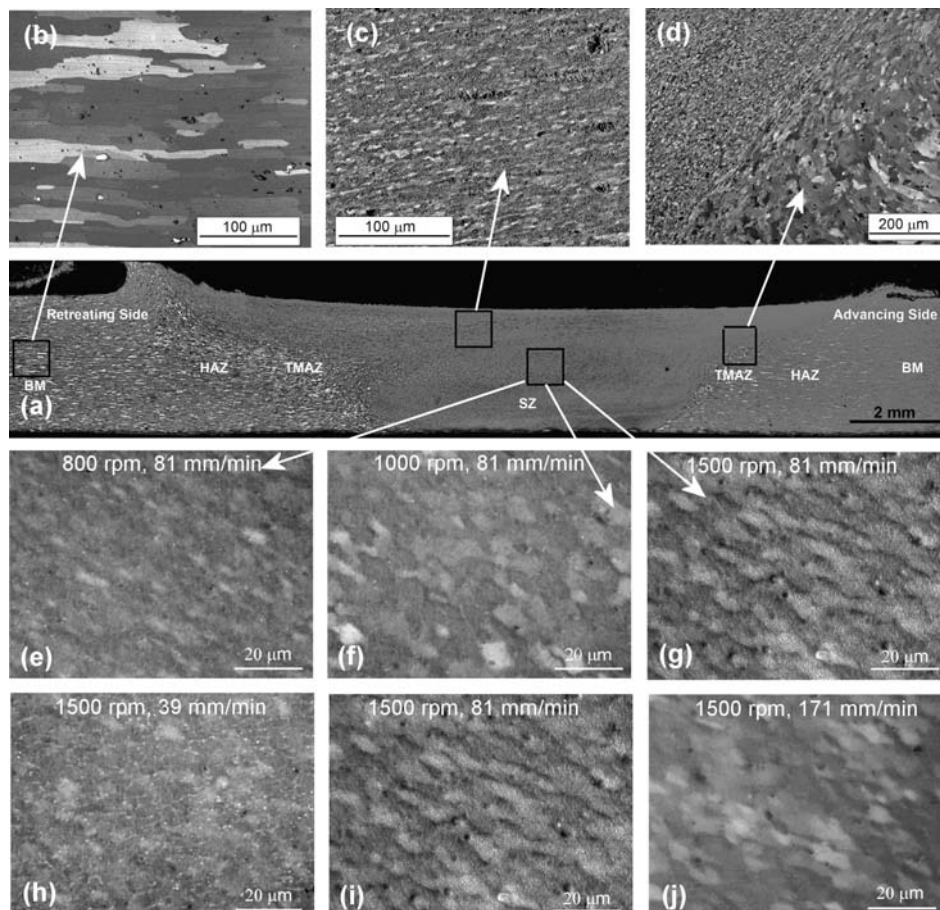
Figure 1. Photographs of (a) the tool and (b) a test plate with a FSP pass made at 1500 RPM and 81 mm/min. The locations of the sample used for microstructural characterisation and microhardness survey (dashed line), and the tensile samples are indicated.

### 3. Results and Discussion

#### 3.1. Microstructural Characterisation

Fig. 2(a) presents the cross-sectional view of a FSP pass made at 1500 RPM and 81 mm/min, indicating the nugget or stir zone (SZ), the thermo mechanically affected zone (TMAZ), the heat affected zone (HAZ) and the unaffected base material (BM) [1-4]. FSP passes produced using other parameters were similar in appearance and all displayed these zones. The higher magnification image of the as-received material presented in Fig. 2(b) shows an elongated, pancake grain morphology with coarse intermetallic inclusions. Although we have not conducted any transmission electron microscopy (TEM) work yet, AA 7075-T6 is a well-characterised age-hardenable alloy and the as-received material is expected to contain three distinct precipitate populations within the grains that cause the strengthening: semi-coherent  $\eta'$  and the equilibrium  $\eta$  phase and GP zones, as well as rod-shaped  $\eta$  precipitates at the grain boundary [4-7]. The complex thermo-mechanical effects associated with FSP mean that the populations of these strengthening precipitates in the SZ, HAZ and TMAZ would be different, see references [4-7].

Fig. 2(c) shows the highly deformed and refined grains in the region that was close to the tool shoulder during FSP. Fig. 2(d) presents the TMAZ region, which experienced both heating and deformation during FSP such that the elongated grains of the base material were deformed in an upward flowing pattern around the nugget zone. The SZ or the nugget had a recrystallised and refined structure. Consistent with other reports in the literature, the grain size varied considerably within the SZ both from top of the nugget to the bottom and laterally from the advancing side to the retreating side [2,5]. Hence, in order to discern the effect of FSP parameters on the SZ grain size, care was taken to ensure that the microstructure from the same region at the centre of the nugget of each sample was compared. Figs. 2(e)-2(g) compare the SZ microstructures of FSP passes made with three different rotational speeds at a constant travel speed of 81 mm/min; Figs. 2(h)-(2(j) present the corresponding micrographs for FSP passes made at a rotational speed of 1500 RPM, for three different travel speeds. It can be seen from these micrographs that while varying the rotational speed from 800 to 1000 RPM resulted in coarsening of the SZ grains (~6 to ~10  $\mu\text{m}$ ), a further increase to 1500 RPM did not have a significant effect. Similarly, it was also evident that varying the travel speed at a constant rotational speed of 1500 RPM did not have a strong influence on the SZ grain size.



**Figure 2.** (a) Low magnification optical micrograph showing the cross-sectional view of a FSP pass made at 1500 RPM and 81 mm/min; Higher magnification optical micrographs of (b) the base material; (c) the region close to the tool shoulder during FSP; (d) TMAZ; (e)-(g), SZ microstructures of three FSP passes made at travel speed 81 mm/min, at rotational speeds 800, 1000 and 1500 RPM, respectively; (h)-(j) SZ microstructures of three FSP passes made with rotational speed 1500 RPM, at travel speeds 39, 81 and 171 mm/min.

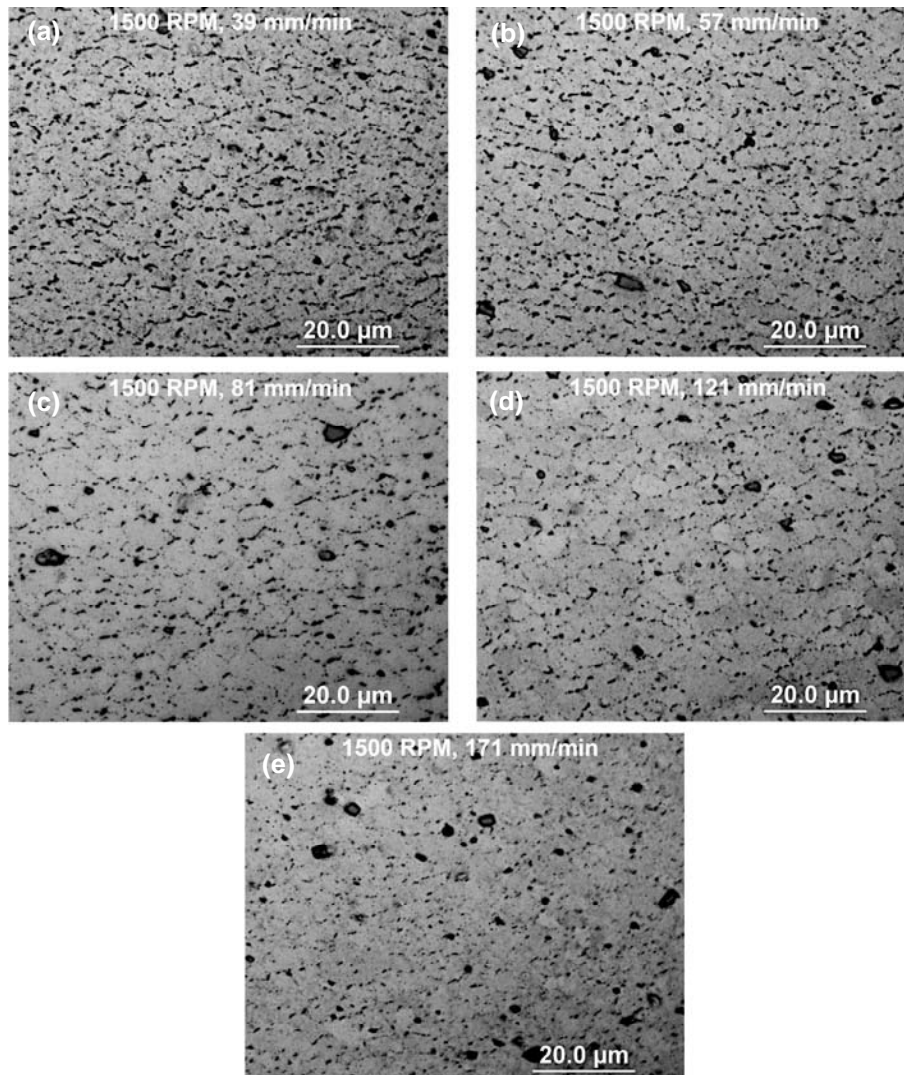
We have noted earlier that the populations of the strengthening precipitates and second phase particles within the various regions of an FSP pass will be different based on the peak temperatures and the thermal cycles associated with those zones. As also noted, the peak temperature generated within the stir zone is a strong function of the rotational speed, while the rate of heating is related to the travel speed [2,4,5]. Accordingly, a detailed analysis of the variation in the appearance and volume fraction of the precipitate and second phase particles within the nugget zone using scanning electron microscopy (SEM) and TEM is currently underway. However, some differences in the second phase particle distribution were already evident from optical microscopy. Figs. 3(a)-(e) show high magnification optical micrographs (using normal illumination) of the nugget zones of FSP passes made with a rotational speed of 1500 RPM, for five travel speeds ranging from 39 mm/min to 171 mm/min. In all cases, two types of second phase particles were evident: coarse, angular particles, randomly distributed in the nugget zone and finer, more globular particles, preferentially formed at the grain boundaries of the equiaxed grains. As seen in Figs. 3(a)-(e), as the travel speed decreased, the extent of precipitation at the grain boundaries increased; at the two slowest travel speeds, Figs 3(a) and (b), the grain boundaries were decorated by a continuous array of these second phase particles. Hassan et al. conducted a detailed analysis of the nugget zone in friction stir welded Al-Zn-Mg-Cu alloy AA 7010 and identified similar coarse second phase particles at the grain boundaries as the  $\eta$  phase [5]. They hypothesised that at high rotational speeds, the solvus temperature of the  $\eta$  phase ( $\sim 440^\circ\text{C}$ ) was exceeded, resulting in its dissolution; however, the slow cooling rate at low travel speeds was insufficient to prevent some re-precipitation of  $\eta$  phase during the cooling cycle of FSP [5]. The observation of increased formation of globular second phase particles in Fig. 3 with decreasing travel speed is in agreement with this hypothesis but needs further detailed analysis for confirmation.

### 3.2. Microhardness Survey

Figs. 4(a) and (b) show the cross-sectional view of an FSP pass made using 1500 RPM and 81 mm/min and the associated hardness contour map. Similar hardness scans were developed for all the samples in the study. The scan reveals the presence of a region of higher microhardness in the SZ, significantly lower than that of the unaffected base material. Between these two regions, there is a region of low microhardness, corresponding to the TMAZ/HAZ. The results are consistent with the more commonly reported w-shaped hardness profile obtained from linear hardness traverses [4-7].

The mean value of all the hardness data from the SZ of each FSP sample was calculated in order to evaluate the influence of the process parameters on nugget microhardness. The results of this analysis are plotted in Figs. 4(c) and 4(d), for variations in rotational speed and travel speed, respectively. As seen in Fig. 4(c), at each of the three travel speeds considered, increasing the rotational speed from 800 RPM to 1000 RPM caused an increase in the mean nugget hardness, but a further increase to 1500 RPM resulted in a lowering of the mean hardness. Clearly, an optimum heat input exists for a given travel speed. In contrast, for all three rotational speeds tested, increasing the travel speed resulted in an increase in the mean nugget hardness. When the data is plotted as a function  $\omega/v$  (Fig. 4(e)), it is evident that the curves for the three travel speeds do not coincide. This suggests that for SZ

hardness, constant values of  $\omega/v$  are not equivalent and that the optimum condition moves to lower values of  $\omega/v$  for increasing travel speed.



**Figure 3.** High magnification optical micrographs of the nugget zones of FSP passes made at 1500 RPM, at different travel speeds: (a) 39 mm/min, (b) 57 mm/min, (c) 81 mm/min, (d) 121 mm/min, and (e) 171 mm/min.

It is interesting to note that an obvious relationship does not seem to exist between the nugget zone grain size and the average microhardness. We have noted that increasing the rotational speed at a given travel speed initially increased the grain size, but a further increase did not affect the grain size significantly, and that at a given rotational speed, increasing the travel speed again had a negligible influence on the grain size, yet both these variations, particularly the latter, significantly affected the microhardness. Although our grain size measurement at this stage is based on optical microscopy and high resolution TEM is required for a more accurate assessment, it is obvious that the Hall-Petch relationship does not seem to apply, which is in agreement with the observations by Hassan et al. [5].

In high strength heat treatable aluminium alloys, the thermal cycles associated with FSP and the peak temperatures attained within the process zone control the level of solution treatment achieved, the amount of solute retained in solution, the

degree of overaging of the precipitates and re-precipitation of second phase particles and the prospect of grain boundary liquation, all of which have a dominant influence on the level of hardness achieved and the ability to recover hardness and strength via post FSP natural aging [2,4,5]. A detailed analysis of the second phase particles and precipitates in the various FSP passes, currently underway, may explain the observed microhardness variations with FSP parameters.

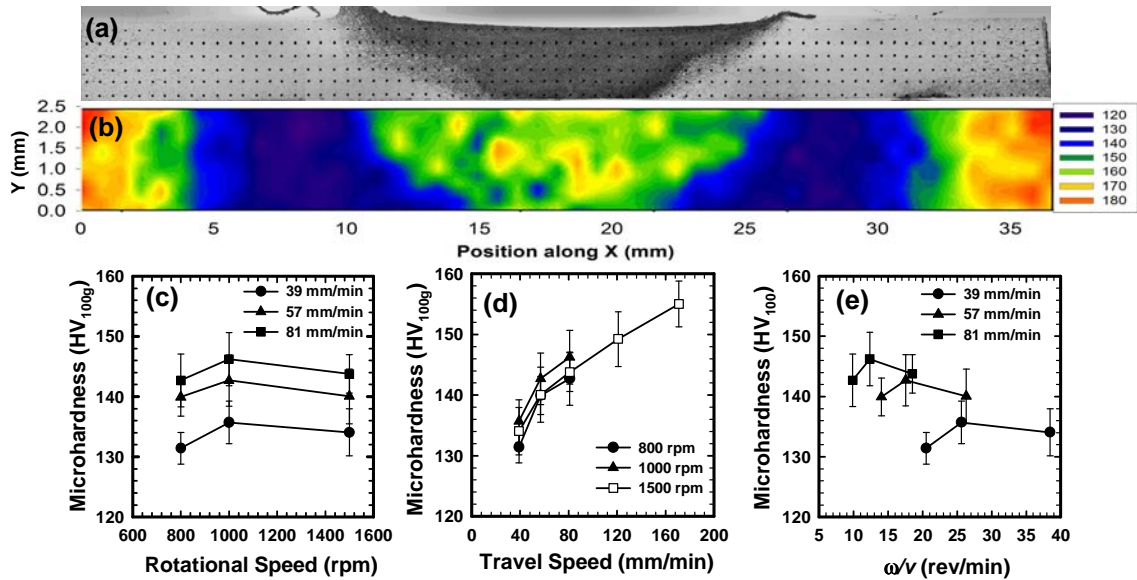


Figure 4. (a) Low magnification optical micrograph of a FSP pass (1500 RPM, 81 mm/min) showing the position of the hardness indents and (b) the corresponding hardness contour map; (c) Variation in mean nugget hardness with rotational speed for travel speeds 39, 57 and 81 mm/min, respectively; (d) Variation in mean nugget hardness with travel speed for three rotational speeds, 800, 1000 and 1500, respectively; (e) Variation of mean nugget hardness with  $\omega/v$ .

### 3.3. Tensile Properties

Fig. 5(a) shows the variation in tensile strength with rotational speed for travel speeds of 39, 57 and 81 mm/min; Fig. 5(b) presents the corresponding ductility data. Figs. 5(c) and 5(d) display the variations in tensile strength and ductility, respectively, with travel speed when the rotational speed is kept constant at 800, 1000 and 1500 RPM. A general observation is that the tensile strengths of the nugget zone of all FSP specimens were significantly lower than that of the as-received material (~565 MPa), while the ductility data (percentage elongation) were significantly higher, about 20-25% compared to ~10.3% for the as-received material. Fig 5(a) indicates an increase in tensile strength with increasing rotational speed at 57 and 81 mm/min travel speeds, but at the lowest travel speed, 39 mm/min, the tensile strength increased when the rotational speed was increased from 800 to 1000 RPM, but dropped to a lower value at 1500 RPM. Fig. 5(c) shows that increasing the travel speed at constant rotational speeds of 800 and 1500 RPM resulted in an increase in the tensile strength. However, at 1000 RPM, the tensile strength dropped when the travel speed was increased from 39 mm/min to 57 mm/min, but increased again when the travel speed was increased to 81 mm/min.

The variation in ductility as a function of rotational and travel speed are presented in Fig 5(b) and Fig 5(d) respectively. In general, the nugget zone of the FSP samples

displayed significantly higher ductility than the as-received material. The FSP nugget zones in all cases displayed ductility in the range 20-25% elongation, however, a correlation with variation in the FSP parameters is not obvious.

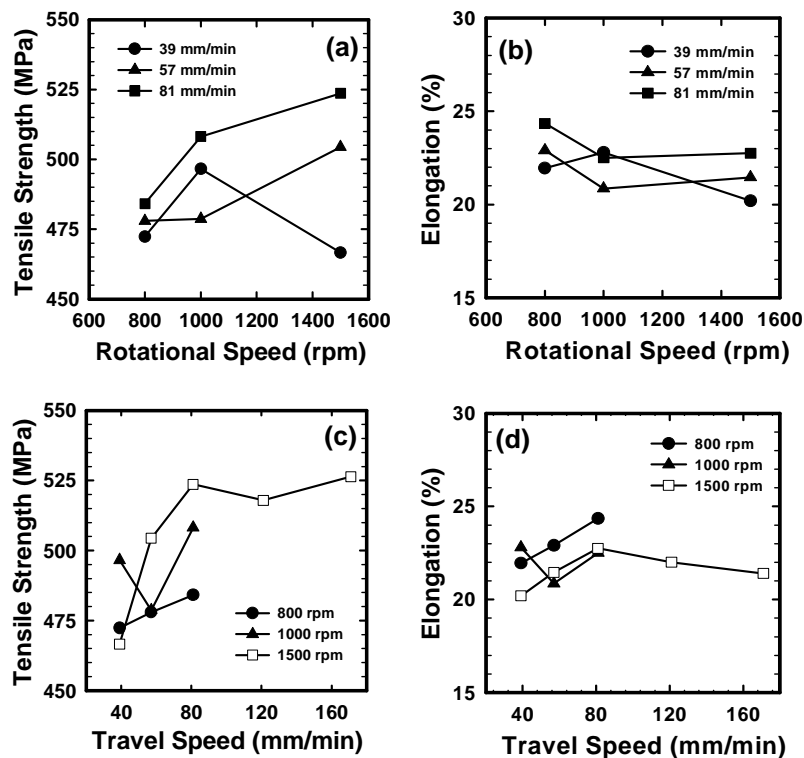


Figure 5. Summary of tensile properties: (a) Variation in tensile strength with rotational speed for travel speeds 39, 57 and 81 mm/min; (b) Variation in elongation with rotational speed for travel speeds 39, 57 and 81 mm/min; (c) Variation in tensile strength with travel speed for three rotational speeds, 800, 1000 and 1500; (d) Variation in elongation with travel speed for three rotational speeds, 800, 1000 and 1500. Each data point corresponds to the mean value of the two tensile test for each FSP pass.

The observed trends in the variation in tensile strength with FSP parameters mirror those obtained for microhardness with some minor variations. Although care was taken to ensure that the tensile samples were extracted exclusively from the nugget zone, it is possible that parts of TMAZ may have been included in the sample, which may account for the observed differences in the trends for microhardness and tensile strength.

#### 4. Conclusions

The effect of the two main FSP parameters, tool rotational speed and travel speed, on the microstructure, microhardness distribution within the nugget zone, and tensile properties of friction stir processed AA 7075-T6 has been investigated. The following conclusions can be drawn:

1. The thermal cycles associated with FSP had a dominant effect on the microstructure and mechanical properties of the nugget zone.



2. For a given travel speed, there was an optimum rotational speed that resulted in the highest nugget hardness. At a given rotational speed, increasing the travel speed resulted in higher average nugget zone hardness.
3. An obvious correlation did not exist between the nugget zone grain size and hardness; further detailed investigation of the second phase particles within the nugget zone and the manner in which their population is affected by the FSP thermal cycles is underway to explain the observed results.
4. The variation in tensile properties with FSP parameters was in general agreement with the trends observed for variations in microhardness.

### Acknowledgements

The authors gratefully acknowledge the financial support of this work by the CSIRO OCE Postdoctoral Fellowship Grant and the Light Metals Flagship. They thank Professors Rajiv Mishra and Yuri Estrin for insightful discussions and their colleagues Wendy Borbidge, David Ritchie, Stephanie Davy, Claude Urbani, Matthieu Larroque and Ian Thomas for assistance with various aspects of the experimental program.

### References

- [1] R.S. Mishra, in: *Friction Stir Welding and Processing*, edited by R.S. Mishra and M.W. Mahoney. ASM International, Metals Park, OH (2007), p. 314.
- [2] R.S. Mishra and Z.Y. Ma: *Mater. Sci. Eng. Vol R50* (2005), p.1.
- [3] Z.Y. Ma: *Met. Mater. Trans. Vol.39A* (2008), p. 642.
- [4] A.P. Reynolds, in *Friction Stir Welding and Processing*, edited by R.S. Mishra and M.W. Mahoney. ASM International, Metals Park, OH (2007), p. 51.
- [5] Kh.A.A. Hassan, P.B. Prangnell, A.F. Norman, D.A. Price and S.W. Williams: *Sci. Tech. Weld. Join. Vol. 8* (2003), p. 257.
- [6] C.G. Rhodes, M.W. Mahoney, W.H. Bingel, R.A. Spurling and C.C. Bampton: *Script. Mater. Vol 36* (1997), p. 69.
- [7] M.W. Mahoney, C.G. Rhodes, J.G. Flintoff, R.A. Spurling and W.H. Bingel: *Met. Mater. Trans A. Vol 29A* (1998), p. 1955.
- [8]. C.B. Fuller, M. W. Mahoney, M. Calabrese, L. Micono: *Mater. Sci. Eng. Vol. A527* (2010), p.2233.
- [9] A.H. Feng, D.L. Chen and Z.Y. Ma: *Met. Mater. Trans A Vol.41A* (2010), p. 957.

## MICROSTRUCTURE AND MECHANICAL PROPERTIES OF A FRICTION STIR PROCESSED Al-Zn-Mg-Cu ALLOY

Margarita Vargas<sup>1</sup> and Sri Lathabai<sup>2</sup>

Friction stir processing (FSP) was performed on AA 7075-T6, a heat treatable high strength Al-Zn-Mg-Cu alloy. The two main FSP parameters, the tool rotational ( $\omega$ , RPM) and travel speed ( $v$ , mm/min), were varied systematically in order to understand their influence on the microstructure and mechanical properties of the processed zone. It was demonstrated that the range of FSP parameters used did not significantly influence the nugget zone grain size. However, the mechanical properties of the processed zone varied significantly. For instance, at a given rotational speed, increasing the travel speed increased the microhardness of the nugget (stir) zone; for a given travel speed there appeared to be an optimum rotational speed which resulted in the highest microhardness. For all parameters tested, the processed zone displayed lower tensile strength, but significantly higher ductility when compared to the as-received alloy. The variation in tensile strength with changes in rotational and travel speed followed the same general trend as in the case of microhardness. In all cases, the ductility was within the range 20-25% elongation. The observed mechanical properties could not be explained as a direct consequence of grain refinement, by the Hall-Petch relationship. It is suggested that these are a result of the complex interactions between the FSP thermo-mechanical effects and the processes of dissolution, coarsening and re-precipitation of the strengthening precipitates in this alloy.

**Keywords:** friction stir processing, 7xxx aluminium alloy, mechanical properties.

1st TMS-ABM International Materials Congress, July 26-30, 2010 Rio de Janeiro, Brazil

<sup>1</sup> Postdoctoral Research Fellow, CSIRO Process Science and Engineering, Clayton, Victoria, Australia.

<sup>2</sup> Principal Research Scientist, CSIRO Process Science and Engineering, Clayton, Victoria, Australia.