Effect of Dynamically Recrystallized Grain Size on the Tensile Properties and Vibration Fracture Resistance of Friction Stirred 5052 Alloy

Kuo-Tsung Huang^{*1}, Tuan-Sheng Lui^{*2} and Li-Hui Chen^{*2}

Department of Materials Science and Engineering, National Cheng Kung University, Tainan, Taiwan 701, R. O. China

5052H34 Al-Mg plates were annealed and then friction stir processed at various rotation speeds ranging from 500 to 1500 rpm to investigate the tensile properties and vibration fracture resistance. The experimental results indicate that grain refinement could be observed at the stir zone with an average grain size varying from 5–16 μm . Based on the observed microstructure and tensile deformation resistance data, the k_y slope value of the Hall-Petch equation can be determined. A refined grain size in the stir zone is a common feature of the friction stirred specimens. Different rotation speeds have different corresponding grain sizes and this can be attributed to dynamic recrystallization during friction stir processing (FSP). The effect of grain size on vibration fracture resistance in the stir zone was also examined. Results show that the vibration fracture resistance of the stir zone decreases with increasing the rotation speed. An increase in grain size due to higher rotation speed is detrimental to the vibration propagation resistance, and a small variation in grain size can result in significant changes in the duration of stage I of the D-N curves. The inward crack propagation behavior was found to be the main controlling factor on vibration fracture resistance. This result agrees with the variation in crack propagation rate. [doi:10.2320/matertrans.47.2405]

(Received May 11, 2006; Accepted July 6, 2006; Published September 15, 2006)

Keywords: friction stir processing, Hall-Petch equation, aluminum-magnesium alloy, crack propagation behavior

1. Introduction

Friction Stir Processing (FSP) is a solid state modification process designed to refine the structure of the stir zone and improve the properties of aluminum alloys.¹⁾ It is achieved by the generation of frictional heat and plastic flow between a friction rod and a substrate. This produces dynamic recrystallization and refinement in the friction stir zone.^{2,3)} Al-Mg alloys, which are classified as non-heat treatable, have a great potential for microstructural evolution by deformation processing and thus possess better mechanical properties.^{4,5)} Our previous studies^{6,7)} have shown that frictional stirred specimens showed a considerable improvement in tensile deformation ductility, although the proof stress value increased only slightly.

In general metallic materials, variation of flow stress depends on grain size according to the Hall-Petch equation^{8,9)} $\sigma_y = \sigma_0 + k_y d^{-1/2}$ where σ_0 may be interpreted as the friction stress and k_y is a positive constant of yielding associated with the stress required.¹⁰⁾ This relationship serves to demonstrate that the flow stress tends to increase with decreasing grain size. In order to investigate whether the grain size in the stir zone is dependent on the Hall-Petch equation, four rotation speeds were used under an identical friction stirring process. The experimental results of this study will also be compared with previous studies which investigated similar Al-Mg base metals. Lloyd and Court¹¹⁾ indicated that the commercially direct chill cast Al-Mg alloy, AA5052, had a k_y slope of about 223–267 $\text{MPa}\mu\text{m}^{-1/2}$. On the other hand, Hayes *et al.*¹²⁾ concluded that the annealing treatment of the ECAP (equal channel angular pressing) process caused the removal of mobile dislocations in the grain structure, and the k_y slope was about 165–170 $\text{MPa}\mu\text{m}^{-1/2}$. However the stir zone experienced dynamic recrystallization during FSP. Some previous studies^{13–15)}

showed that the as-stirred specimens contained grains of high dislocation density. If the dislocation density of the grains in the interior of a material affects the slope value, the effect of dynamic recrystallization on the k_y value needs to be examined.

Al-Mg alloys are currently used in the automotive industry. The materials often experience an abnormal vibration which is one of the most common reasons for fracture failure in the transportation field. In particular when the applied vibration frequency meets the resonant frequency, the vibration can cause catastrophic failure.¹⁶⁾ In the past decade, a number of studies have been undertaken to characterize the microstructural evolution resulting from friction stir processing,^{2,17,18)} but there is still a lack of vibration fracture data due to this being a relatively new process. A previous study pointed out¹⁹⁾ that as-stirred Al-Mg alloy can effectively lengthen the fatigue life compared to the traditional joining method. In our previous investigation on the resonant vibration of Al-Mg alloy,^{7,20)} it was found that grain refinement is able to improve the crack propagation resistance. However, in the friction stirring process, the heat input during FSP in the stir zone increases with increasing the tool rotating speed causing the grain size to grow up.^{10,21)} In this study, the rpm-dependent microstructural feature was investigated, and the sensitive response of k_y value and stage I duration in the D-N curve of the as stirred specimen pertaining to the grain size was clarified.

2. Experimental Procedures

5052H34 Al-Mg plates were chosen to achieve a fully-annealed microstructure at 625 K for 2 hours, after which the plates were subjected to a single pass by FSP. The schematic illustration of FSP is shown in Fig. 1. Four tool rotation speeds were chosen as 500, 800, 1000 and 1500 min^{-1} , while the tool moving speed was fixed at 160 mm min^{-1} with 1.5° of tool angle and 19.6 MPa of the downward push force. The chemical compositions of the plates are listed in Table 1. The

^{*1}Graduate Student, National Cheng-Kung University

^{*2}Corresponding author, E-mail: z7408020@email.ncku.edu.tw

Fig. 1 Schematic illustration of the friction stir process (FSP).

Fig. 3 Schematic illustration of the tensile specimen (FSP).

Table 1 Chemical composition of the samples (mass%).

	Mg	Fe	Cr	Si	Mn	Al
5052	2.58	0.26	0.21	0.15	0.10	Bal.

Fig. 2 Hardness profile across the stir zone.

base metal specimen is designated as “BM” hereafter. The as-stirred specimens at 500, 800, 1000 and 1500 min⁻¹ are designated as “F5”, “F8”, “F10” and “F15”, respectively.

To explore the microhardness profile in the vicinity of the stir zone, a Vickers indenter with a 0.98 Nf load for 15 s was used. Microstructural inhomogeneity in all the as-stirred specimens was observed. The microhardness profiles along the cross section of the as-stirred specimens are shown in Fig. 2, however the microhardness of the stir zone decreases with increasing the tool rotation speed. Consequently, in the following tensile test, in order to eliminate the influence of advancing or retreating microstructure, the specimens were cut entirely from the stir zone. In order to examine the effects of the microstructural features and tensile properties, the strain rate was fixed at $8.3 \times 10^{-4} \text{ s}^{-1}$, and a tensile specimen with 15 mm gauge and 3 mm thickness was used, as shown in Fig. 3.

On the other hand, in order to investigate the effect of

dynamically recrystallized grain size on the crack propagation behavior of various as-stirred specimens, the V notches specimen was used, as shown in Fig. 4(a). Figure 4(c) schematically depicts the vibration set-up that was used. Each test specimen was clamped onto a vibration shaker with a deflection sensor attached to the end of the specimen to measure the deflection amplitude. In order to fix the initiating surface strain of each specimen, the initiating deflection was fixed at 6.5 mm. Prior to testing, the frequency of the vibration shaker was varied to determine a specimen’s resonant frequency. When the vibration frequency of the shaker meets the natural frequency of the specimen, *i.e.* at resonant frequency, the deflection reaches a maximum, as indicated in Fig. 4(b). For all specimens, the resonant frequency was in the range of $42 \pm 1 \text{ Hz}$.

The effect of microstructural differences in the four as-stirred specimens on their crack propagation paths was examined by an optical microscope equipped with an image analyzer. All specimens were surface finished by polishing with $0.3 \mu\text{m Al}_2\text{O}_3$ so that the crack propagation path could be observed directly on the specimen surface. Consequently, quantitative data of crack tortuosity could be determined using the method shown in Fig. 5. Then the crack tortuosity value could be defined as the length of the main crack divided by the projected length of this crack along its macroscopic propagation direction (shown by the dotted line in Fig. 5). In addition, to measure the crack propagation rate, the vibration test was temporarily stopped after each minute of vibration testing.

3. Results and Discussion

3.1 Effect of rotation speed on the average dynamically recrystallized grain size and tensile properties

Figure 6 shows the optical microstructures of the four as-stirred specimens with different rotation speeds. The stir zones are composed of fine equiaxed recrystallized grains, and the average grain size tended to increase as rotation speed increased. The average grain sizes of F5, F8, F10 and F15 were 5.5, 8.1, 11.3 and 15.6 μm , respectively. A typical microhardness profile can be recognized for the four as-stirred samples and the specimens with larger grain sizes had lower average microhardness as shown in Fig. 2.

Fig. 4 Shape and dimension of the resonant vibration test specimen for (a) vibration geometry and dimension, (b) resonant frequency, (c) the resonant vibration setup.

Fig. 5 Schematic drawing of the cracking features generated by resonant.

Some previous studies^{5,14,22} have suggested that dynamically recrystallized grains are nucleated at a specific site pertaining to the prior microstructure of the base metal during the friction stirring process. After the stirring tool has passed, dynamically recrystallized grains grow statically during the cooling of the thermal cycle.^{23,24} Grain growth occurs rapidly at the recrystallization temperature ($>0.5 T_m$, T_m being the absolute melting temperature) so that the grain size growth in the stir zone is promoted by an increase in the process temperature. Our previous study⁶ showed that the process temperature increases with increasing rotation speed. There are reasons why a higher rotation speed results in larger equiaxed grains in the stir zone.

Figure 7 shows the typical tensile strain-elongation curves of the as-stirred specimens. They all show a similar serrated yielding behavior. Differences in the serrated flow pattern can be recognized including the onset strain and intensity of the serrated flow, compared with Table 2. However, the smaller grains possessed a greater area fraction of grain boundaries along the line of the mobile dislocations and therefore the number of dislocation tangles was greater which

were strong enough to hold the mobile dislocations, and long enough to let Mg atoms form atmospheres around them.²⁵ This effect caused the smaller onset strains and the larger stress drops of serrations as shown in Fig. 7(b). Furthermore, Fig. 7(a) also indicates that the yield stress decreased slightly with increasing the rotation speed. In addition, the results can be further compared to the fully annealed base metal as shown in Table 2. A better uniform elongation is a consequence of the fine dynamically recrystallized grain of the as-stirred specimens.

The effect of grain size on yield stress of the as-stirred specimens can be further illustrated in Fig. 8, however the experimental results actually follow the Hall-Petch equation ($\sigma_y = 176d^{-1/2} + 64$) and are linearly proportional to the average grain size. As mentioned above,^{11,12} experimental results obtained from fully annealed Al-3Mg has a k_y slope of about $165\text{--}170 \text{ MPa } \mu\text{m}^{-1/2}$, while the commercially direct chill cast Al-Mg alloy, AA5052, possesses a larger k_y slope of about $232\text{--}267 \text{ MPa } \mu\text{m}^{-1/2}$. The k_y value obtained from friction stirred materials is close to the annealed Al-3Mg sample. It is to be noted that there are only small variations in k_y value between the as-stirred and fully annealed specimens, but there is a distinct microstructural change in the grains which is closely related to the dislocation density of dynamically recrystallized grains.

Ito *et al.*²⁶ have suggested that various deformation processes lead to a larger k_y slope value because the grains contain a high density of dislocations. Some other studies^{15,18} have shown that dynamic recrystallization which occurs during FSP often leaves some grains with a high density of dislocations in the stir zone. This result suggests

Fig. 6 Optical Microscopic of (a) F5(500rpm), (b) F8(800rpm), (c) F10(1000rpm), (d) F15(1500rpm).

Fig. 7 Relation of (a) nominal strain vs. elongation curves, (b) strain drop vs. elongation.

Table 2 Effect of rotation speed on tensile properties of as stirred samples.

specimens	U.T.S (MPa)	Y.S. (MPa)	U.E. (%)	T.E. (%)	tortuosity	mean grain size (μm)
F5	232	141	14	20	1.18	6
F8	236	124	18	28	1.17	8
F10	233	116	20	28	1.16	11
F15	232	108	20	30	1.13	16
BM	207	101	15	29	1.21	46

that the dislocation density of the friction stirred materials is slightly higher than that of annealed materials.²⁰⁾ Our TEM observations, as shown in Fig. 9, agree with this finding.

3.2 Effect of rotation speed on the vibration fracture resistance

Figure 10(a) shows the D-N curves (deflection amplitude vs. vibration cycle) of the specimens under the resonant frequency. As illustrated in Fig. 10(a), all specimens exhibit a common feature that the deflection amplitude increases for a critical duration in the initial stage and subsequently decreases in the later-stage, and thus the D-N curve can be generalized into two stages. Stage I: the deflection amplitude increases as vibration cycles increase until a maximum is reached. Stage II: the deflection decreases from the peak point as the vibration cycles increase further.

It should be noted that the F5(500 min⁻¹) curve possesses

Fig. 8 Hall-Petch relationship curves.

the longest duration of stage I resulting from the finest dynamically recrystallized grain size. Figure 10(a) shows the locus of deflection amplitude of the four stirred samples. The duration of stage I tends to decrease as the grain size

increases. In addition, Fig. 10(a) also shows that the slope of stage I of the D-N curves tends to raise up as the grain size increases. According to our previous studies^{7,16)} an increase in work hardening rate can raise the effective elastic modulus, which can increase the deflection by a reduction in the damping capacity. Results can be further compared with n value as shown in Fig. 11, and the strain hardening rate ($F5 < F8 < F10 < F15$) can be clearly elucidated. Based on D-N curves as shown in Fig. 10(a) the work hardening stage is designated as stage I, and the vibration fracture resistance is strongly dependent on the duration of stage I. When the vibration cycles are prolonged, there is a drastic decrease in deflection amplitude, after a maximum point that can be designated as stage II, which results from the deviation of the actual vibration frequency from the resonant frequency and the inward propagation of the major crack.^{16,27)}

Our previous study⁶⁾ confirmed that Al-2.5Mg specimens showed a deviation of the actual vibration frequency from the resonant frequency when the deflection amplitude was decreased from the maximum value that resulted from the effect of reducing the effective cross-sectional area of the test specimen. Therefore, the vibration life in this study can be defined as the vibration cycle number when the deflection

Fig. 9 TEM image of (a) BM, (b) F5, (c) F10, (d) F15.

Fig. 10 Deflection amplitude vs. number of vibration cycles curve (a) D-N curve, (b) vibration life.

Fig. 11 n value vs. true strain curve by tensile test.

Fig. 12 Crack propagation rate under constant initial-deflection conditions.

amplitude reached its maximum value (*i.e.* the above-mentioned duration of Stage I). Figure 10(b) reveals that the vibration life decreases with increasing the average dynamically recrystallized grain size. Our results also confirm that these specimens had a lower deflection slope which reduced the driving force of crack propagation. This is another reason why a longer vibration life was acquired. However the lower n value (see Fig. 11) in the specimens with finer grain size can reduce the deflection slope. The abovementioned factors and the experimental data as shown in Fig. 10(b) demonstrate that both rotation speed and grain size greatly affect the vibration fracture resistance of friction stirred specimens, and the results can be further compared to the base metal (fully annealed sample). The refining of dynamically recrystallized grain size plays an important role in increasing the vibration life.

3.3 Examination of the crack propagation features

Figure 12 shows significant differences in the crack propagation resistance of the friction stirred specimens with different rotation speeds. The crack propagation rate in-

creases with increasing the rotation speed, which is related to the coarser dynamically recrystallized grain size. This result can be further compared to the observed crack propagation behavior, as shown in Fig. 13. For the F15 specimen, SEM evidence indicates that slip bands are more significant in the vicinity of the main crack.

As illustrated in Figs. 6 and 13, the effect of average dynamically recrystallized grain size on vibration fracture resistance is probably correlated with the evolution of microstructural features during cyclic deformation process. Figure 6 confirms that a smaller grain size can increase vibration fracture resistance resulting from plastic deformation in the vicinity of the main crack as shown in Fig. 13(a). Moreover, as shown in Fig. 14, the vibration fracture pattern revealed that both specimens demonstrated a flat feature, which is closely related to slip deformation behavior. The patterns can also be easily proven by comparing the crack propagation paths shown in Figs. 14(c) and 14(d). Figure 14(d) shows that a specimen with larger grain size

Fig. 13 Optical micrographs and SEM micrographs showing the macroscopic crack propagation feature (a)(c) F5, (b)(d) F15.

Fig. 14 SEM micrographics and Optical microstructure of (a)(c) F5, (b)(d) F15. The arrows indicate the macroscopic crack propagation direction.

results in a flatter surface. Consequently, Fig. 10(b) shows that the as-stirred specimens with smaller grain size possessed longer vibration life. According to above-mentioned observations, it's reasonable to suggest that the grain size plays an important role in vibration propagation behavior.

These results agree with the variation in crack tortuosity which is indicated in Table 1. However, when the range of dynamically recrystallized grain size is larger, the crack propagation rate is more significant pertaining to the vibration propagation behavior.

4. Conclusions

According to the experimental results discussed in the previous sections, the following conclusions can be drawn:

- (1) Significant microstructural refinement caused by the phenomenon of dynamic recrystallization was obtained in the friction stirred specimens. Significant microstructural differences in the stir zone were recognized when the rotation speed of the spindle was changed.
- (2) For the friction stirred specimens, the grain size increased with increasing rotation speed. The yield strength deteriorated owing to the increased grain size, which can be quantitatively correlated with the Hall-Petch equation. The angle of the k_y slope was slightly larger than the fully annealed base metal samples. This was related to the dislocation density in the grain interior of the material. This feature can be easily proven by comparing the results of serrations caused by dynamic strain aging.
- (3) The vibration fracture resistance of friction stirred specimens is closely related to the duration of stage I which decreases with increasing the grain size. The grain size increase induces the slope of stage I of the D-N curves to rise and is consequently detrimental to the crack propagation resistance.
- (4) After the main crack has grown to a critical length, the effective cross-section area of the specimens reduced. All stirred specimens deviated from the actual vibration frequency resulting in the deflection amplitude decreasing from its maximum value.

Acknowledgements

Financial support from the National Science Council of Taiwan is gratefully acknowledged (contract No. NSC 95-2221-E-006-118).

REFERENCES

- 1) Y. J. Kwon, I. Shigematsu and N. Saito: *Mater. Trans., JIM* **45** (2004) 2304–2311.
- 2) J. Q. Su, T. W. Nelson, R. Mishra and M. Mahoney: *Acta Mater.* **51** (2003) 713–729.
- 3) C. G. Rhodes, M. W. Mahoney, W. H. Bingel, R. A. Spurling and C. C. Bampton: *Scr. Mater.* **36** (1997) 69–75.
- 4) Y. S. Sato, Y. Sugiura, Y. Shoji, Seung Hwan C. Park, H. Kokawa and K. Ikeda: *Mater. Sci. and Eng. A* **369** (2004) 138–143.
- 5) Y. S. Sato, H. Kokawa, K. Ikeda, M. Enomoto, S. Jogan and T. Hashimoto: *Metall. Mater. Trans., A* **32** (2001) 941–948.
- 6) K. T. Huang, T. S. Lui and L. H. Chen: *Mater. Trans., JIM* **45** (2004) 3216–3222.
- 7) K. T. Huang, T. S. Lui and L. H. Chen: *Mater. Trans., JIM* **46** (2005) 2268–2275.
- 8) E. O. Hall: *Proc. Phys. Soc. B* **64** (1951) 747.
- 9) N. J. Petch: *J. Iron Steel Inst.* **174** (1953) 25.
- 10) Y. S. Sato, M. Urata, H. Kokawa and K. Ikeda: *Mater. Sci. and Eng. A* **354** (2003) 298–305.
- 11) D. J. Lloyd and S. A. Court: *Mater. Sci. and Technol.* **19** (2003) 1349–1354.
- 12) J. S. Hayes, R. Keyte and P. B. Pragnell: *Mater. Sci. and Technol.* **16** (2000) 1259–1263.
- 13) K. V. Jata, K. K. Sankaran and J. J. Ruschau: *Metall. Mater. Trans. A* **31** (2000) 2181.
- 14) K. V. Jata and S. L. Semiatin: *Scripta Mater.* **43** (2000) 743–749.
- 15) Y. S. Sato and H. Kokawa: *Metall. Mater. Trans. A* **32** (2001) 3023.
- 16) D.-S. Jiang, L. H. Chen and T. S. Lui: *Mater. Trans., JIM* **41** (2000) 499–506.
- 17) G. Liu, L. E. Murr, C.-S. Niou, J. C. McClure and F. R. Vega: *Scripta Mater.* **37** (1997) 355–361.
- 18) K. V. Jata, K. K. Sankaran and J. J. Ruschau: *Metall. Mater. Trans. A* **31** (2000) 2181.
- 19) M. Czechowski: *J. Mater. Processing Tech.* **164–165** (2005) 1001–1006.
- 20) K. T. Huang, T. S. Lui and L. H. Chen: *Mater. Trans., JIM* **46** (2005) 3051–3058.
- 21) Y. J. Kwon, I. Shigematsu and N. Saito: *Mater. Trans., JIM* **45** (2004) 2304–2311.
- 22) D. P. Field, T. W. Nelson, Y. Hovanski and K. V. Jata: *Metall. Mater. Trans. A* **32** (2001) 2869–2877.
- 23) Y. S. Sato, M. Urata and H. Kokawa: *Metall. Mater. Trans. A* **33** (2002) 625–635.
- 24) Y. Li, L. E. Murr and J. C. McClure: *Mater. Sci. Eng. A* **271** (1999) 213–223.
- 25) X.-M. Cheng and J. G. Morris: *Scripta Mater.* **43** (2000) 651–658.
- 26) Y. Ito, N. Tsuji, Y. Saito, H. Utsunomiya and T. Sakai: *J. Jpn. Inst. Met.* **64** (2000) 429.
- 27) D. S. Jiang, T. S. Lui and L. H. Chen: *Scripta Mater.* **36** (1997) 15–20.