Magnesium Technology 2011
Edited by: Wim H. Sillekens, Sean R. Agnew, Neale R. Neelameggham, and Suveen N. Mathaudhu
TMS (The Minerals, Metals & Materials Society), 2011

MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Mg-1.7Y-1.2Zn SHEET PROCESSED BY HOT ROLLING AND FRICTION STIR PROCESSING

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Keywords: Friction Stir Processing, Magnesium-Rare Earth, Texture, Hot Rolling

Abstract

The use of lightweight structural materials is an integral part of mass reduction strategy in transportation applications. Magnesium based sheet products have gained significant interest in the automobile industry. Newer magnesium alloys such as Mg-Y-Zn have potential to develop sheet products with superior mechanical properties owing to improved precipitation hardening response. In the present work, rolled sheet of an Mg-1.7Y-1.2Zn alloy containing small amounts of Al and Ce was investigated. Microstructure and mechanical properties were examined in as-rolled, rolled+aged, friction stir processed (FSP) and FSP+aged conditions. Mechanical properties (YS, UTS and %El) of the sheet showed certain anisotropy in rolling and transverse directions, which was marginally reduced upon ageing. However, FSP led to a significant range of mechanical properties depending on the test direction. Ageing of the FSP sheet showed improvement in mechanical properties and reduced anisotropy in the two directions. The static recrystallization due to ageing caused reduced anisotropy in FSP treated sheet. The overall strength-ductility variation is discussed in terms of relative contributions of grain boundary strengthening, texture and precipitation strengthening.

Introduction

Magnesium alloys have gained significant interest among automobile manufacturers due to low density and high specific strength that translate to improved fuel economy and reduced gaseous emissions [1-3]. Despite significant potential of magnesium sheet for hot forming of complex automobile panels [4], its role in automotive applications is still limited due to poor ductility and strength. For example, AZ31 sheets produced by the conventional direct-chill (DC) process or even a more advanced twin roll casting (TRC) technique find limited applications primarily due to their low ductility, although specific strength of Mg alloys is high [5]. The highest yield strength achievable with the current commercial wrought Mg alloys is about 240 MPa and there is a requirement to develop improved wrought alloys and products having yield strength more than 300 MPa [6].

Grain refinement and precipitation hardening are two primary mechanisms for improving room temperature strength in Mg alloys, whereas, ductility could be improved by grain refinement as well as texture modification. Ductility improvement in fine grained Mg alloys is due to activation of prismatic and pyramidal slip systems at grain boundaries and limited tendency for twinning [7]. Extensive ductility in coarse grained Mg alloys is achievable by the addition of alloying elements, such as, Ca, Th, Mn or certain rare earth (RE) elements (*e.g.*, Ce), which was realized in 1940 [8]. Barnett *et al.* [9] have demonstrated cold rolling reduction of ~30, >90 and ~15% in pure Mg, Mg-0.2Ce and AZ31 alloys, respectively. Mishra *et al.* [10] have demonstrated extensive ductility in hot extruded Mg-0.2Ce alloy due to weakening of texture in the presence of cerium. Extrusions conducted at other than the optimized process parameters delivered low ductility in the alloy due to incomplete recrystallization [11]. Hantzsche *et al.* [12] have recently reported reduced planer and in-plane anisotropy in the mechanical behavior of ZEK100 Mg alloy (containing an RE-element) sheet compared to ZM21 alloy (having no RE-element), that were hot rolled at the temperature of 450°C up to a thickness of ~1.35 mm. The decreased anisotropy in the mechanical behavior of ZEK100 alloy was due to weakening of basal texture that was governed by different recrystallization kinetics compared to the ZM21 alloy. Sikand *et al.* [13] observed grain refinement and improved mechanical properties (ductility as well as strength) in the porthole extrusion of ~3 to 3.5 mm thick walled AM30 circular tubes compared to conical die extrusions. Porthole extruded 4 mm thick walled circular tubes of Mg alloys with <0.5% Ce addition showed improved ductility; but, found ineffective in Mg-3Al alloys due to higher affinity of Ce with Al [14]. The weakening of texture due to Ce addition in Mg is therefore offset by the presence of high Al content and requires attention during the design of newer alloy systems.

Severe plastic deformation (SPD) processes, such as, Equal Channel Angular Pressing (ECAP), High Pressure Torsion (HPT), Accumulative Roll Bonding (ARB), etc have opened up new opportunities for the development of ultra fine grained (UFG) lightweight materials [15]. Friction stir processing (FSP) is a relatively new process for developing UFG alloys with enhanced mechanical properties. A schematic diagram of the FSP is shown in Fig. 1. Important aspects of this process, such as, process parameters, process modeling, microstructure and resultant mechanical properties, etc, have been comprehensively addressed by Mishra and Ma [16]. The process uses a rotating tool comprising of a threaded pin and tool shoulder to apply severe plastic deformation and frictional heating that produces significantly refined grain size.

Grain boundary strengthening employing SPD has limited potential and newer Mg alloys having precipitation hardening response are required. Mg-Y-Zn is a promising alloy system that could deliver exceptional mechanical properties at ambient as well as elevated temperatures due to the presence of icosahedral (I) ternary phase [17]. Three different types of ternary phases in the Mg-Y-Zn alloy systems have been reported; which are, icosahedral quasicrystal Mg_3YZn_6 (I-phase), cubic $Mg_3Y_2Zn_3$ (W-phase) and 18-R long-period ordered (LPO) $Mg_{12}YZn$ (Z-phase) [18-19]. The formation of ternary phases depends on the Zn/Y ratio of the alloy composition. Lee *et al.* [19] have identified Zn/Y ratio as 5~7 for the presence of I-phase in as cast Mg-Y-Zn alloy systems. Room temperature strengthening in Mg-Y-Zn systems greatly depends on the fraction of quasicrystalline I-phase, since, the I-phase precipitates with a definite orientation relationship (OR) with the matrix and forms strong interface with it. The I-phase is resistant to coarsen up to 440°C and has low interfacial energy, thus impedes grain boundary sliding and hinders dislocation movement at elevated temperatures.

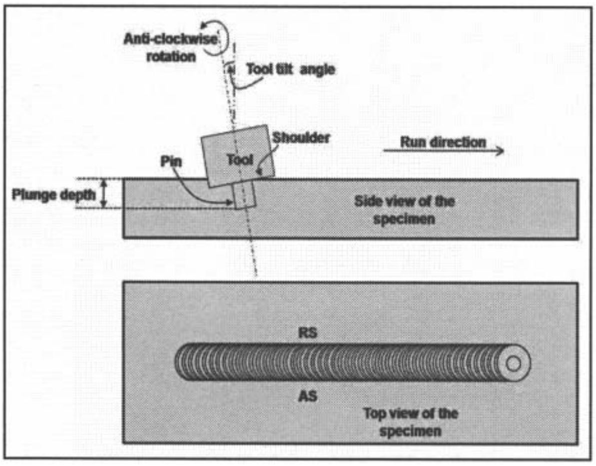


Fig. 1 Schematic diagram of the Friction Stir Processing (FSP). AS: Advancing Side; RS: Retreating Side

The present study was aimed to determine the effect of FSP on the room temperature mechanical properties of hot rolled sheets of an Mg-1.7Y-1.2Zn alloy. FSP was used as the technique to obtain refined microstructure. The observed mechanical properties have been discussed in terms of correlation with the texture anisotropy in the rolled and friction stir processed sheets, which were later aged under the chosen parameters.

Experimental Procedure

The composition of Mg-Y-Zn alloy was Y-1.7%, Zn-1.2%, Al-0.53%, Mn-0.27%, Ce-0.013%, Mg-balance. FactSage thermodynamic calculations predicted four equilibrium phases coexisting at 450°C, *i.e.*, $MgYZn_3$, Mg_2Y, Mg and β-Mn. It was predicted that at room temperature, two additional phases, *i.e.*, $Mg_{43}Y_4Zn_3$ and $Ce(Mg,Al)_{12}$ could precipitate. The alloy was cast as rectangular plate in size of 14×30×1.3 cm^3. The cast plates were hot rolled at 450°C to a final thickness of 1.9 mm in nine passes with a total reduction of ~85%. Reheating of the rolled material was carried out at 450°C after each pass. The rolled sheets after the final pass were annealed at 450°C for 10 minutes. FSP of rolled sheets was carried out with a simple cylindrical threaded tool. The tool had a pin of 4.5 mm dia and 1.5 mm height. Diameter of the tool shoulder was 11.5 mm. A single pass FSP was carried out employing tool rotation rate of 500 rpm and translation speed of 101.6 mm per minute. The tool tilt angle was kept as 2.5°. The depth of processing/plunge depth was chosen as ~1.5 mm and the direction of processing was kept same as rolling direction of the sheets. Ageing of as-rolled and FSP sheets was conducted at the temperature of 160°C for 96 hrs. The ageing parameters were selected based on optimized conditions observed for similar kinds of Mg alloys [6].

Dog bone tensile specimens of 1.5 mm gage length, 1.2 mm gage width and ~0.8 mm thickness were machined from the as-rolled, rolled-aged, FSP treated and FSP-aged sheets. Table I shows details of different types of samples used. In the case of FSP sheet, tensile samples were machined from centre line of the nugget region removing ~200 µm top layer by polishing. Tensile testing was carried on a computer controlled mini tensile testing machine employing a strain rate of 1×10^{-3} s^{-1}. Optical, stereo and scanning electron microscopy of the sheet material processed under different conditions were carried out to study the microstructure and fracture response of tensile specimens. For the microstructural investigations, samples were polished up to 0.5 µm diamond paste followed by etching in a solution containing 4.5 g picric acid, 10 ml acetic acid, 10 ml distilled water and 70 ml ethanol.

Table I. Sample description for mini tensile tests of Mg-1.7Y-1.2Zn sheet.

Sample ID	Material Condition	Test Direction
As-rolled_RD	As-rolled (450°C)	Rolling
As-rolled_TD	As-rolled (450°C)	Transverse
Aged_RD	Rolled and aged (160°C, 96h)	Rolling
Aged_TD	Rolled and aged (160°C, 96h)	Transverse
FSP_RD	Rolled and FSP*	Rolling
FSP_TD	Rolled and FSP*	Transverse
FSP_aged_RD	Rolled, FSP* and Aged	Rolling
FSP_aged_TD	Rolled, FSP* and Aged	Transverse

* FSP was conducted in the rolling direction

Results & Discussion

Microstructure

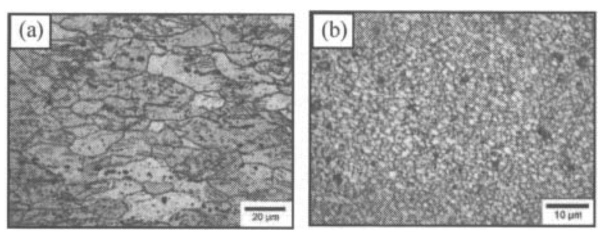


Fig. 2. Optical micrographs (longitudinal section) of Mg-1.7Y-1.2Zn alloy sheet, a) as rolled and b) after FSP. FSP processed micrograph was taken from nugget region showing substantial grain refinement

The average grain size of as-cast Mg-1.7Y-1.2Zn alloy was measured as ~730 µm. Figure 2 shows optical micrographs of the alloy sheet in the as-rolled and FSP conditions. Micrographs were taken in longitudinal section of the sheet. The average grain size of as-rolled sheet was determined as ~20 µm (Fig. 2a), which was refined close to 1 µm after FSP as observed in Fig. 2b. The microstructure of the as-rolled sheet showed a mix of coarse and fine grains with numerous coarse grains having twinned structure, suggesting that recrystallization was not fully complete after rolling. A few elongated bands of coarse grains were observed in the FSP sheet. Frictional heating and extensive plastic deformation during the FSP resulted in significant grain refinement due to dynamic recrystallization, which again appeared to have completed partially. No significant change in the grain size after aging was observed for both the as-rolled and FSP sheets; however, in the case of aged FSP sheet, bands of elongated coarse grains were slightly broken up due to the progress of static recrystallization during ageing.

Mechanical properties and textural effects upon FSP

Fig. 3 shows engineering stress-strain curves of the as-rolled and FSP treated sheet in rolling and transverse directions. The tensile properties, such as, yield strength (YS), ultimate tensile strength (UTS) and total elongation (%) determined from these curves are shown in Table II. For the as-rolled sheet, yield stress in rolling direction is 247 MPa, which is higher compared to 179 MPa in the transverse direction. The difference in UTS in the two directions is low and total elongation is similar (~25%). Several twins in the as-rolled microstructure were observed suggesting partial completion of dynamic recrystallization and significant deformation texture is expected due to the low Ce content (~0.013%) in the alloy. An inversion in the mechanical properties and considerable anisotropy is observed in the FSP treated sheet. Yield strength of 178 MPa in the rolling direction was measured, which is substantially lower than 306 MPa observed in the transverse direction. Correspondingly, UTS in the processing direction is very low, but, interestingly, even the elongation in this direction is poor (2.9%) compared to 24.5% in the transverse direction. FSP treated sheet therefore delivered high YS, high UTS and high elongation in transverse direction compared to the processing direction.

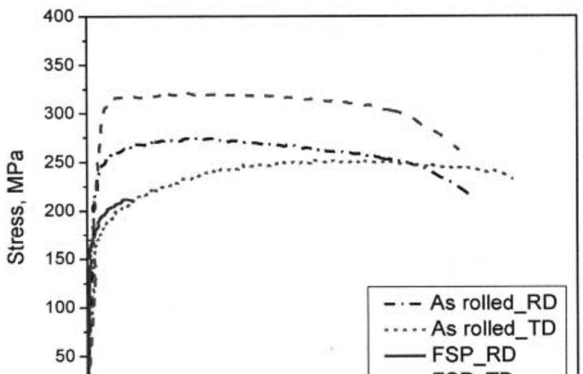


Fig. 3. Effect of FSP and test direction on the stress-strain curves of the rolled Mg-1.7Y-1.2Zn sheet

The observed mechanical behavior is due to strong textural effects produced during FSP. Earlier investigations have suggested that shear plastic flow during FSP resulted in a texture in which {0002} basal planes roughly surrounding the rotating pin surface within the nugget region leading to an "onion ring" structure [20,21]. The basal slip was presumed to be the dominant slip system within the nugget region for the shear plastic flow due to large difference in the critical resolved shear stress (CRSS) of basal and nonbasal slip that produced strong textural effects after FSP. A wide variation in texture was observed in regions within and away from the stir zone, *i.e.*, transition and base material regions, which correlated well with significant anisotropy in the mechanical behavior in different directions. Schuddin *et al.* [22] have reported alignment of {0002} basal planes with the tool shoulder surface in the upper stir zone. A similar texture response is anticipated in the present study. The low yield stress of 178 MPa observed in the processing direction is due to easier basal activity as a result of preferential alignment of basal planes. In transverse direction, a high yield stress of 306 MPa suggests predominant non-basal activity, since CRSS for non basal slip systems is about 2 to 6 times higher for polycrystalline Mg alloys [23]. Woo *et al.* [21] have reported early fracture in transverse direction for FSP AZ31B rolled plate due to plastic incompatibility created by the orientation difference between the stir zone and the transition zone. However, the observation of high yield strength coupled with high ductility in transverse direction in the present study was a result of sample extraction within the nugget region or stir zone. The large variation in yield stress in different directions in spite of similar grain size (~1 μm) reveals that Hall-Patch relation is significantly influenced by strong textural effects produced during FSP.

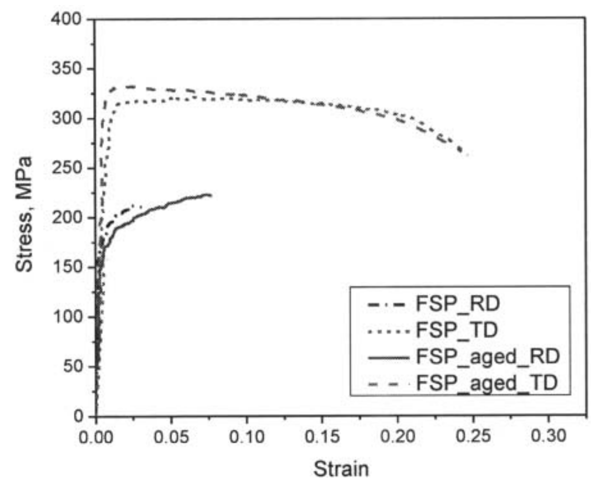


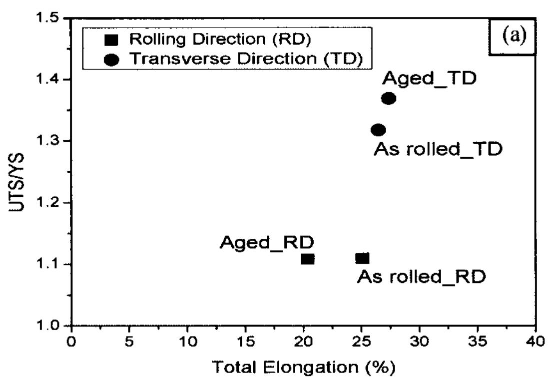
Fig. 4. Effect of ageing and test direction on the stress-strain curves of the FSP Mg-1.7Y-1.2Zn sheet

Table II. Mechanical properties of as-rolled, FSP and aged sheet.

Sample ID	Tensile Properties		
	Yield Strength (MPa)	UTS (MPa)	Total Elongation (%)
As-rolled_RD	247	274	25.1
As-rolled_TD	179	245	27.4
Aged_RD	240	266	20.4
Aged_TD	189	249	26.5
FSP_RD	178	212	2.9
FSP_TD	306	320	24.5
FSP_aged_RD	172	222	7.2
FSP_aged_TD	324	333	24.1

Effect of ageing on the mechanical behavior

Fig. 4 shows effect of ageing and test direction on the stress-strain behavior of FSP sheet. The compiled mechanical properties are shown in Table II, which suggest that ageing has marginal influence on the strength properties of as-rolled sheet; however, FSP treated and subsequently aged sheet showed improvement. Yield strength of 306 MPa in the transverse direction is increased to 324 MPa upon ageing of the FSP sheet; however, ductility is not changed (~25%). On the other hand, in the processing direction, yield strength is nearly unaffected, but, ductility is improved from 2.9% to 7.2% upon ageing. Fig. 5 is a graphical comparison showing effect of ageing and test direction on the UTS/YS ratio for the as-rolled and FSP sheets. In spite of high yield strength in the rolling direction (Table II), the UTS/YS ratio is low (~1.1) compared to that in the transverse direction (~1.35), both for as-rolled as well as aged sheets (Fig. 5a). Therefore, the as-rolled sheet possesses high strain hardening capacity in the transverse direction, which is nearly unaffected upon ageing. The total elongation for all four samples (*i.e.*, As-rolled_RD, As-rolled_TD, Aged_RD and Aged_TD) are comparable as observed from the figure. Mechanical properties after FSP and subsequent ageing treatment show large variation in the two test directions as shown in Fig. 5b. The UTS/YS ratio is little higher in the processing direction (~1.2) as compared to the transverse direction (~1.05) after FSP. Upon ageing, this ratio is nearly unchanged in the transverse direction, but, improved to ~1.3 in the processing direction). The total elongation values have a large variation in the two directions both for the FSP as well as FSP-aged sheet.



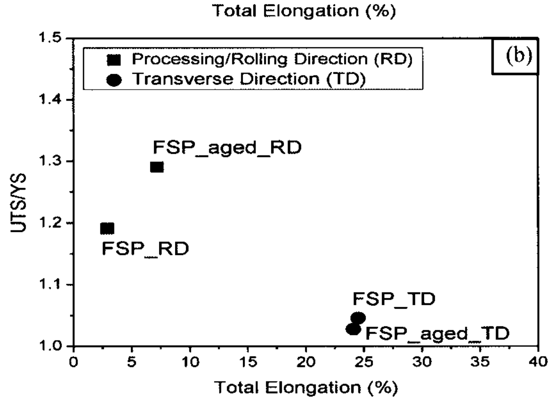


Fig. 5. Effect of ageing and test direction on UTS/YS ratio v/s total elongation for a) as rolled sheet and b) FSP sheet

FSP is carried out at strain rates of the order of 10^0 to 10^2 s^{-1} and up to strain of 40 depending on the process parameters. The process results in substantial grain refinement due to dynamic recrystallization, however, the final microstructure has deformation induced texture. FSP causes frictional heating and extensive mixing of material within the stir zone, hence, dissolution of existing phases takes place in the matrix. FSP leads to variation of texture in the stirred zone of Mg alloys but generally the grain size is relatively uniform. Ageing imposes two effects on microstructure in the stir zone, i) static recrystallization takes place that could lead to further refinement in the microstructure, at least in the elongated grained regions, since the low angle boundaries within these grains may change to high angle grain boundaries under the driving force of stored energy due to high dislocation density, and ii) precipitation of intermetallics phases occurs. Both of these effects favor improved yield strength in the aged material, whereas, grain refinement causes improvement in ductility. Extensive ductility observed in this study in the transverse direction for FSP and FSP-aged sheet is due to combination of microstructural refinement and texture that favors nonbasal slip. However, the reason for poor ductility in the processing direction is not clear. Detailed textural investigations are required to understand the anisotropic mechanical behavior observed in this work.

Stereo micrographs and correlation with the mechanical behavior

Fig. 6 shows stereo macrographs of fractured mini tensile specimens of as-rolled and FSP treated sheet tested in the rolling/processing and transverse directions. The right side-bottom portion of each tensile specimen (e.g., part D in Fig. 6a) was tilted 90° upright to observe the fracture response from the side plane. Thickness and width of the specimens were measured at necked region and compared with original dimensions so as to determine overall geometrical changes at fracture, such as, % reduction in width, thickness and area. The plane of initiation of fracture was identified in each case. The angle between the fracture plane and the tensile loading axis was also determined. The results are shown in Table III for different test specimens. Figs. 6a and b show that shear fracture in as-rolled specimens initiated from the top or bottom plane. The as-rolled specimen tested in rolling direction shows uniform reductions of ~16% in thickness as well as width accompanied with ~30% reduction in area. The shear fracture angle was found as 57°. Similar fracture response is obtained in transverse direction with slightly lower reductions in width, thickness as well as area. Shear fracture occurred at an angle of 52°. The lower values of geometrical changes in the transverse direction are due to higher strain hardening capability and lower post uniform elongation as observed in Fig. 3. For an isotropic ultrafine grained copper, Zhao *et al.* [24] reported nearly uniform reduction in thickness and width for tensile specimen geometry having width and thickness ratio (*W/T*) close to unity. Such geometry causes shear deformation both at the gage top or bottom plane as well as side planes resulting in homogenous development of shear bands. In the present study, *W/T* ratio was close to 1.5, which produced similar geometrical changes in width and thickness for the as-rolled tensile specimens, although, slightly lower values were observed in transverse direction compared to rolling direction, which was due to the presence of deformation texture after hot rolling and complete recrystallization could not occur.

Table III. Geometrical parameters of fractured tensile specimens of as rolled, FSP treated and aged sheet

Sample ID	**Geometrical changes in fractured tensile specimen at necked region**			**Fracture initiation plane**	**Angle between loading axis and fracture plane (°)**
	Reduction in width (%)	**Reduction in thickness (%)**	**Area reduction (%)**		
As rolled_RD	16.7	15.6	29.7	Top plane	57
As rolled_TD	13.6	10.5	22.3	Top Plane	52
Aged_RD	14.2	15.7	27.6	Top Plane	55
Aged_TD	13.4	14.2	29.1	Top Plane	53
FSP_RD	0.9	5.4	6.3	Top Plane	31
FSP_TD	25.9	11.1	34.1	Side Plane	58
FSP_aged_RD	3.3	7.3	10.4	Top Plane	30
FSP_aged_TD	30.2	8.3	35.9	Side Plane	60

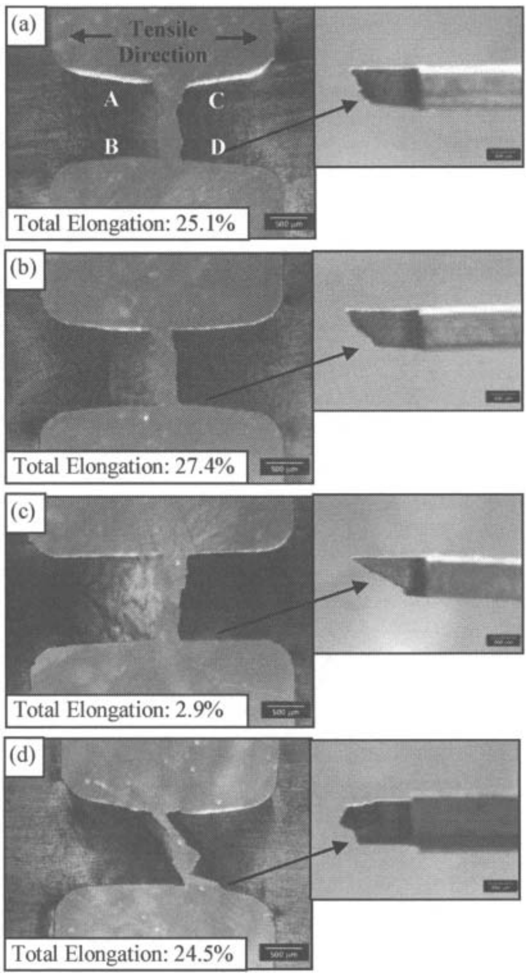


Fig. 6 Stereo macrographs of fractured tensile specimens as per sample ID shown in Table I, a) As rolled_RD, b) As rolled_TD, c) FSP_RD and d) FSP_TD. Macrographs on the right side show side plane of the fractures samples, which were taken by tilting right side bottom portion (e.g., part D shown in Fig. a) upright 90°. Fig. a, b and c show that fracture initiated from top plane, whereas, Fig. d shows that fracture initiated from side plane.

Figs. 6 c and d show that FSP treatment of rolled sheet greatly affected fracture response of tensile specimens tested in the processing and the transverse directions. In the processing direction, the FSP treated sheet showed fracture initiation from the top or bottom plane of tensile specimen with an acute shear failure angle of 31° (shown in Table III), whereas, in the transverse direction, failure is initiated from the side plane at a shear angle of 58°. It is interesting to observe that in the processing direction, reduction in width is mere less than 1%, whereas, thickness reduced by 5.4% accompanied with an area reduction of 6.3%. In the transverse direction, the geometrical changes in width and thickness are very high, but, reversed compared to the processing direction. The reduction in width and thickness is 25.9% and 11.1%, respectively, whereas, area reduction is 34.1%, which is substantially high. Textural anisotropy has played a significant role in the observed mechanical behavior. The results correlate well with the % elongation results shown in Table II. In the processing direction, the poor elongation of 2.9% suggests that shear bands could not develop well in the gage section, in spite of easy basal slip that caused yielding at a lower stress 178 MPa. In the transverse direction, the yield stress as well as total elongation both were very high and confirm with intense shear bands developed within the gage section, which is visible in Fig. 6d. The fracture localization occurred at side plane instead of top plane (see inset in Fig. 6d showing location of fracture initiation) due to higher reduction in the width, which led to the shear failure at an angle of 58°.

The effect of ageing of FSP treated sheet on the geometrical changes during tensile tests is shown in Table III. In the transverse direction, there is no appreciable change in the geometrical parameters of FSP-aged specimen compared to FSP alone and total reduction in area is similar in both cases, *i.e.*, ~35%, which is consistent with total elongation value of ~25% with or without ageing of the FSP sheet. In the processing direction, however, a noticeable change is observed upon ageing. Reduction in width and thickness of FSP_aged_RD specimen was measured as 3.3% and 7.3%, respectively, which is an improvement over the values of 0.9% and 5.4%, observed for the FSP_RD specimen. The total elongation of 7.2% (Table II) and area reduction of 10.4% (Table III), both are higher for the FSP_aged_RD condition compared to FSP_RD specimen that delivered respective values of 2.9% and 6.3%.

Conclusions

Hot rolled sheet of an Mg-1.7Y-1.2Zn alloy was processed by FSP and aged in as-rolled and FSP conditions. As-rolled sheet showed some anisotropy in the mechanical behavior in rolling and transverse directions that was marginally reduced after ageing. FSP resulted in substantial refinement in grain size, however, large difference in mechanical properties was observed in the two directions. The substantial anisotropic behavior was due to strong textural effects. The mechanical properties were found improved and anisotropy was reduced upon ageing. Small sized tool was employed for FSP in this study. Advances in FSP, such as, feasibility of using large dimension tools, multi-pass FSP and robotic controlled processing have potential scope in implementing FSP for the large-sized automotive body panels. Further optimization studies on the FSP and ageing parameters are necessary to achieve the said goal.

Acknowledgements

The first author, V. Jain, gratefully acknowledges J. William Fulbright Foreign Scholarship Board (FSB), the US Department of State's Bureau of Educational & Cultural Affairs (ECA), the Government of India, the Institute of International Education (IIE) and United States-India Educational Foundation (USIEF) for the award of *"Fulbright Nehru Doctoral & Professional Research Fellowship"* under the grant No. 1369/DPR/2010-2011 that enabled him to take up the present work at CFSP, Missouri S&T. Mr. Jain is a Graduate student at Deptt of Materials & Metallurgical Engg, IITK-Kanpur (India) and he is thankful to his PhD supervisors, Dr. Gouthama (Assoc. Professor, IITK-Kanpur) and Dr. Anil K. Gupta (Director, AMPRI, CSIR, Bhopal & Ex-Head Division of Engg. Materials, NPL, CSIR, New Delhi) for their constant encouragement and valuable suggestions during this work.

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