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Short communication

Combining surface mechanical attrition treatment with friction stir processing to optimize the mechanical properties of a magnesium alloy

Zhen Zhang[a,b], Yaozu Li[a,b], Jinhua Peng[a,c], Peng Guo[a], Ji'an Huang[a], Pengju Yang[a,b], Shan Wang[a,c], Chang Chen[a,b,*], Wei Zhou[c,d], Yucheng Wu[a,b]

[a] School of Materials Science and Engineering, Hefei University of Technology, Hefei, 230009, China
[b] National–Local Joint Research Center of Non-ferrous Metal Materials and Processing Technology, Hefei, 230009, China
[c] Institute of Industry and Equipment Technology, Hefei University of Technology, Hefei, 230009, China
[d] School of Mechanical and Aerospace Engineering, Nanyang Technological University, 50 Nanyang Avenue, Singapore

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ABSTRACT

Friction stir processing (FSP) and surface mechanical attrition treatment (SMAT) were performed on an AZ31 magnesium alloy to improve the mechanical properties. The microstructure, macro-texture, as well as mechanical properties were investigated by optical microscopy, transmission electron microscopy, X-ray diffractometer and uniaxial tensile tests respectively. FSP significantly refined the initial grain structure and modified the texture of the experimental material; and the elongation got increased with a loss in yield and flow stresses. SMAT introduced a severely localized deformation layer on sample surfaces, which greatly strengthened the local micro-hardness and yield stress of parental matrix. By combining SMAT with FSP, the loss in yield and flow stresses caused by FSP could be well compensated by the SMAT routing. This optimized the mechanical properties of the experimental material.

1. Introduction

Magnesium alloys show superior properties such as low density, high specific strength and good electromagnetic shielding, etc. They have received increasing attention recently under global requirement for carbon emissions reduction. However, due to the less symmetrical hexagonal crystal structure, the ambient formability of magnesium is quite limited [1,2]. In recent years, many efforts have been made to improve the ambient formability of magnesium using approaches such as texture modification [3–5], alloying additions [6–8], grain refinement [9–11], etc. Compared with other metals, grain refinement is believed to be more effective for magnesium than other metals in improving the comprehensive mechanical properties. This is because, other than strengthening effect, reducing grain size also suppresses contraction twinning in magnesium which always plays as a most preferential nucleation site for micro-crack. This would improve the ductility of magnesium as a bonus effect [12–14].

Severe plastic deformation (SPD) method has been considered as an industrial reliable, controllable, relatively inexpensive routing to realize grain refinement compared to other methods, e.g. rapid solidification [15]. As a severe plastic deformation (SPD) method, friction stir processing (FSP) technique attracted more attention from people in magnesium field in recent decades [16–19], because it could significantly improve the ductility of magnesium without loss in ultimate strength. Moreover, it could be conducted locally on a target region of structural component without changing the geometry of the workpieces. Although FSP shows superiority over other conventional processing strategies, the dark side is obvious. When FSP was imparted, a rather large plasticity could be obtained. However, due to the evitable modification of texture introduced by FSP, a quick drop in yield stress, as well as in flow stress, was always concomitant. This could be considered as the dark side of the application of FSP technique on magnesium material.

On the other hand, surface mechanical attrition treatment (SMAT) is another severe plastic deformation method that could produce submicron sized grains on sample surfaces by introducing extremely high strain locally on the processed surfaces [20–23]. This would significantly strengthen the micro-hardness and yield stress of deformed layer on the surface. Compared with other surface treatment methods, SMAT would introduce a much thicker, finer-grained and more uniform deformation layer on base material [24]. This means that by using SMAT the modification in mechanical properties was more obvious for bulk material. Although the material could be strengthened to great extent when grain size was reduced to submicron scale, the plasticity

* Corresponding author. School of Materials Science and Engineering, Hefei University of Technology, Hefei, 230009, China.
E-mail address: chench@hfut.edu.cn (C. Chen).

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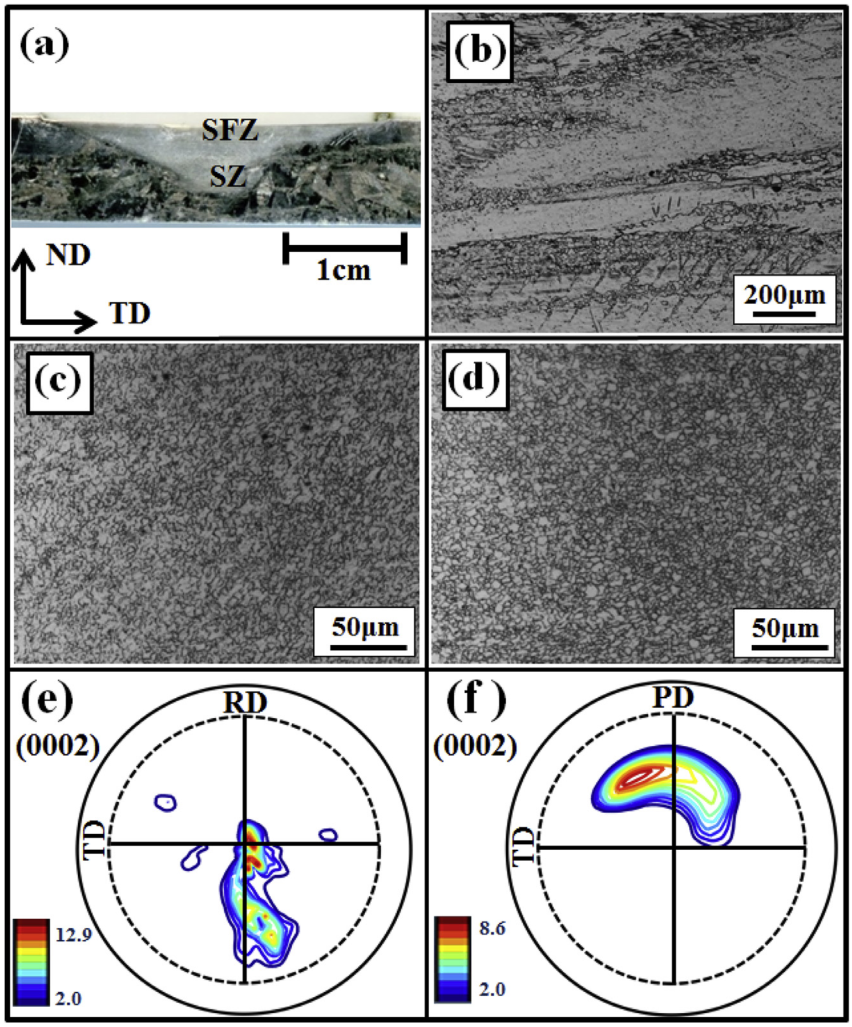

Fig. 1. (a) Macro-morphology viewed on the ND (normal direction)-TD (transverse direction) plane along PD (processing direction); typical metallurgical microstructure from (b) base material, (c) shoulder affected zone and (d) nugget zone; (0002) pole figures (e) before and (f) after FSP.

usually suffers an evident loss as a consequence. It is then interesting to know whether it is possible to take advantage of the beneficial effects from FSP and SMAT simultaneously and to make them complement each other's disadvantages, with the aim to obtain an optimized comprehensive mechanical property in magnesium.

In the present work, friction stir processing (FSP) was first conducted on AZ31 magnesium plates, and then the FSPed samples were exposed to the surface mechanical attrition treatment (SMAT) on both sides. The microstructure and texture were characterized by the optical microscope and X-ray diffraction. The micro-hardness and tensile properties were measured to understand the relationship between the microstructure, texture and mechanical properties for the experimental material.

2. Experimental procedure

The experimental material used in the present work was commercial AZ31 plates/sheets with a nominal composition of 3 wt% Al, 1 wt% Zn and the balance Mg. Twin-roll casting plates with a thickness of 6 mm were used as the base material for FSP. Such relatively large thickness was used to keep sufficient rigidity during FSP. A computerized numerical control (CNC) machine was modified to conduct the processing, with stir needles as machining tools. The geometry of the processing tool is provided in the Supplementary Fig. S1. In the present work, FSP was conducted along the roll-casting direction (RD) of the plates with a tool rotation speed of 1000 r/min and a proceeding speed of 90 mm/min. After FSP, ~1.5 mm thick sheets were cut from the processed surface of the work-pieces. This thickness was chosen to obtain uniformly fine-grained FSPed regions and the largest areas for further SMAT processing on both sides. Round samples with a diameter of 49 mm were cut from the ~1.5 mm sheets with the FSPed regions located in the center of the samples. The surface of Φ49 mm sheets was then ground, polished and sent for SMAT processing for both sides. In the present work, a SPEX SamplePrep 8000 M Mixer/Mill was used for SMAT processing. After putting steel grinding balls into the sample pot, the Φ49 mm sheet samples were then attached to the cover with the treated surface facing the milling balls inside the sample pot. The pot was then installed onto the milling machine for SMAT processing. During processing, the pot vibrated at a frequency of ~50 Hz and followed a path of three-dimensional 8-font with a rotation speed of 875 rad/min. In this way, the milling balls would impact multi-directionally and repetitively with a rather high energy onto the surface of the samples attached to the cover of the sample pot, which would effectively introduce a localized SPD layers on sample surfaces. The adjustable parameters for such SMAT were the condition of milling balls (material, quantity, size) and the processing time. In the present work, we used thirty Φ5 mm steel milling balls for processing with varying processing time of 15, 30, 60, 90 and 120 min for both sides. It needs to be stressed that such method gave milling balls much higher impact energy than conventional planet-type grinding mills; this was why it was expected to produce more uniform and thicker deformed layers on samples surfaces. The FSP + SMAT routing was schematically shown in Supplementary Fig. S2.

Metallographic observation was conducted on the plane perpendicular to the proceeding direction (ND-TD plane, ND presents the normal direction of the processed plate, TD presents the transverse direction). The samples were prepared following conventional mechanical polishing procedure and etched with a solution of picric–acetic. JEOL 2100 F TEM was used to examine the deformed layer introduced by SMAT processing. The specimens were also sectioned perpendicular to the PD, ground to less than 100 μm. The cross-section of SMATed layer was further thinned by focused ion beam (FIB) to be electron transparent for TEM observation. The macro-texture was measured by X-ray with a beam size of 6 mm × 6 mm on a Panalytical diffractometer in Schulz reflection geometry; the used X-ray tube was copper with a wavelength of 0.154 nm. The sample preparation followed standard mechanical polishing procedures and the samples were finally immersed in a solution of 5% nitric acid +95% ethanol for ~10 s to remove any residual stress introduced by sample preparation. The microhardness was measured on ND-TD plane for all samples with 300 g for 10 s. Tensile samples were machined along the processing direction (PD) with a gauge length of 27 mm and a gauge width of 4 mm.

3. Results and discussion

3.1. FSP induced microstructure, texture and mechanical tensile behavior

The initial material used for FSP was commercial twin-roll casting plate with a thickness of 6 mm. As shown in Fig. 1, the starting material shows an inhomogeneous microstructure with a basal texture. After FSP, the stirred zone showed a typical conical shape from ND-TD view. The microstructure from different regions was also further examined. It could be clearly seen that the original grains were significantly refined and became rather homogeneous in the stirred zone. The average grain size was measured to be around 5–10 μm. Moreover, the macro-texture was also modified, with most basal poles tilted towards the proceeding direction (PD) on (0002) pole figure after FSP, as shown in Fig. 1e–f.

In order to reveal the influence of microstructure and texture, induced by FSP, on the mechanical properties of the experimental material, firstly we measured the micro-hardness distribution along ND across the stirred zone on the ND-TD plane, as shown in Fig. 2a. It could be clearly seen that the micro-hardness increased from ~60 H V for base material to more than 80 H V for the region close to the processed surface. It was reasonable to attribute such micro-hardness increment to the grain refinement effect caused by FSP. To further reveal the plastic behavior of the material processed by FSP, uniaxial tensile tests were conducted on the samples cut from the top surface of the FSPed workpieces. The strain-stress curves were shown in Fig. 2b. The as-received experimental material showed a typical tensile curve with very limited elongation (6%). After FSP, however, the elongation was enormously raised to be higher than 40%, which indicated an evident improvement in ductility. Our previous work showed that, apart from the FSP induced grain refinement effect, such good ductility originated more from the modified texture caused by FSP [25]. It needs to be stressed here that the yield stress dropped sharply as soon as FSP was applied. It was reasonable to believe that such low strength was expected considering the tilted basal texture which facilitated the most common basal slip in the FSPed samples. In a word, FSP produced homogeneous and fine grain structure as well as a modified basal texture for the experimental magnesium alloy; and this caused an evident improvement in ductility. At the same time, the yield stress was sharply reduced, which was considered as the dark side of FSP method when imposed on magnesium alloys.

3.2. SMAT induced microstructure, texture and mechanical tensile behavior

In order to reveal the influence of SMAT method on the microstructure and texture evolution, firstly we used ~1 mm hot-rolled sheets for SMAT processing. The relatively thin sheets were used as the base material to fully manifest the effect of SMAT on the entire mechanical performance of the bulk material. Moreover, the initial hot-rolled sheets showed a well-developed basal texture and equa-axial grains with an average size of ~10 μm, which allowed us to detect any deviation from initial microstructure and texture originating from the thin SMAT induced layers. The thickness of SMAT induced layers was measured to be ~11, 46, 143, 171 μm, for varying processing time of 30, 60, 90 and 120 min. This could be clearly observed on the lateral section of SMAT sample, as shown in Fig. 3. This present work showed that a distinct deformation layer was already formed after 90 min SMAT processing; and the layer thickness didn't change much with further processing.

Since it was difficult to reveal the microstructure within the severely deformed layers at higher resolution by optical microscopy, we cut a specimen from the lateral section for TEM observation. The results showed that new grains with submicron or even nano-scale size were produced on sample surface by SMAT. The macro-texture of the SMATed surface was also tested. An inclined (0002) pole was seen after SMAT, which indicated the weakening of basal texture. This was more clearly revealed by employing an accurate scanning for (0002) peaks, as shown in Fig. 3f. It could be seen that the peak intensity gradually decreased with processing time; and the peak value dropped to less than ~1/3 of base material after 120 min processing. In addition, the micro-hardness was also measured on lateral section from one surface to the other, for samples with different processing time (Fig. 4a). In general, the micro-hardness was found increase greatly from the central part (base material) to SMATed surface; and the SMAT induced hardness increment also increased with processing time. The influence of SMAT on mechanical behavior was also tested in uniaxial tensile tests, as

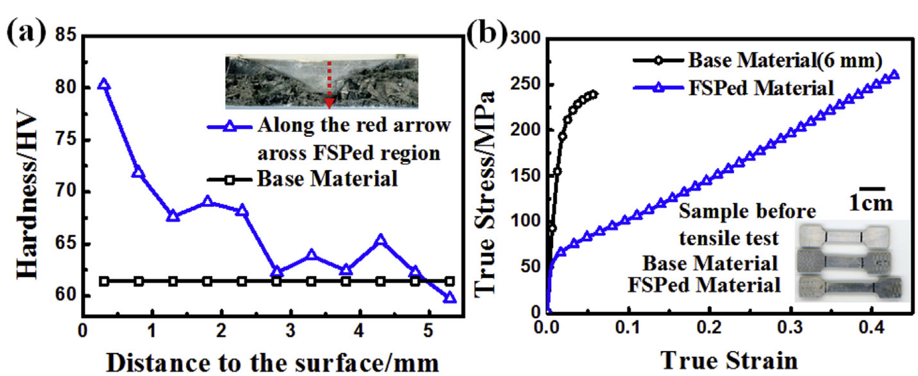

Fig. 2. (a) Micro-hardness tested on the ND-TD plane along ND (indicated by the red arrow in the inset) across the center of processed region (b) Stress-strain curves of the base and FSPed material. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

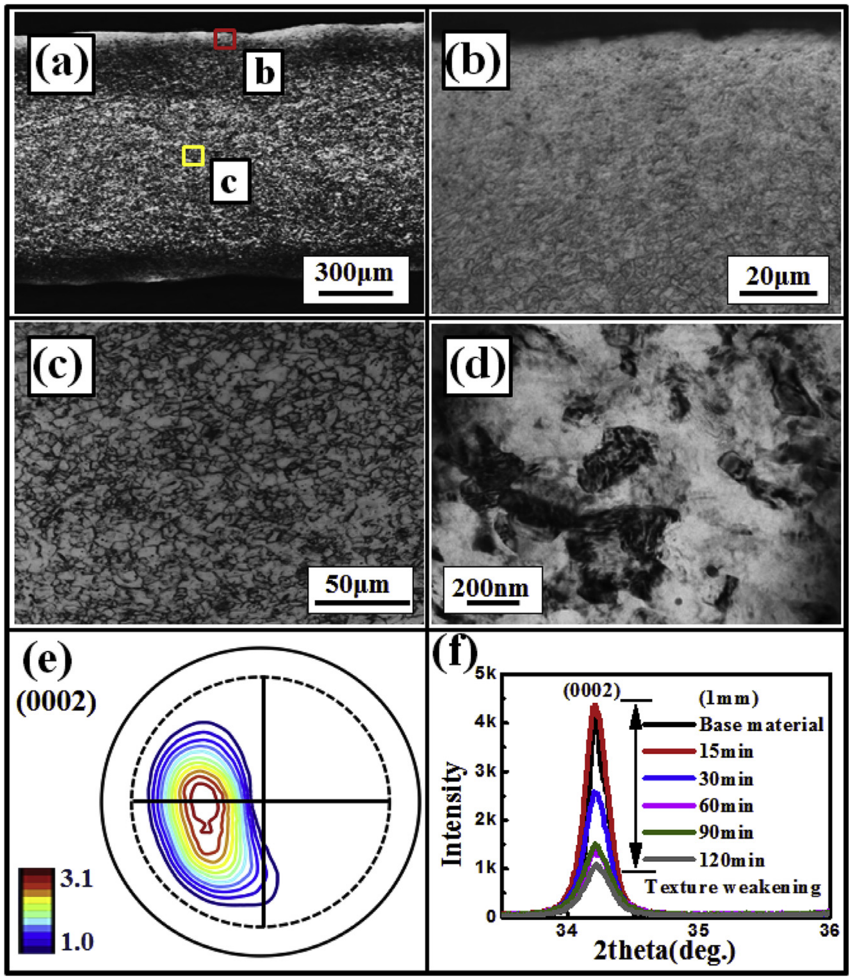

Fig. 3. (a) Macro-morphology on the cross-section of a sample processed by surface mechanical attrition treatment (SMAT); the metallurgical microstructure of area (b) and area (c); (d) TEM morphology acquired at a depth of ~60 μm below the processed surface; (e) (0002) pole figure measured by X-ray on the SMATed surface, (f) the profile of (0002) peaks which indicates a gradual weakening of basal texture with processing time.

shown in Fig. 4b. It was interesting to see that although it only produced a rather thin deformation layer (< 180 μm) on the processed surfaces the yield stress underwent an evident raise after SMAT. This implies a good potential of SMAT in strengthening the yield stress for magnesium alloys. However, the elongation underwent an evident drop from 20% to 12% after 90 min SMAT processing. It needs to be stated again that the base material here (used for pure SMAT processing) was different from that used for FSP. It was hot-rolled sheets with ~10 μm equa-axial grains and well-developed basal texture, thus shows a higher elongation and strength than the twin-roll cast 6 mm plates used for FSP.

3.3. FSP + SMAT processing to acquire optimized mechanical properties

It is reasonable to think that if similar SMAT induced strengthening effect works on FSPed samples, it would compensate the loss in yield stress after FSP. Such would light up the dark side of the application of FSP on magnesium. In the present work, ~1.5 mm sheets were cut from the processed surface of FSPed samples and then sent to SMAT processing for 90 min on both sides. Sheet samples after combined

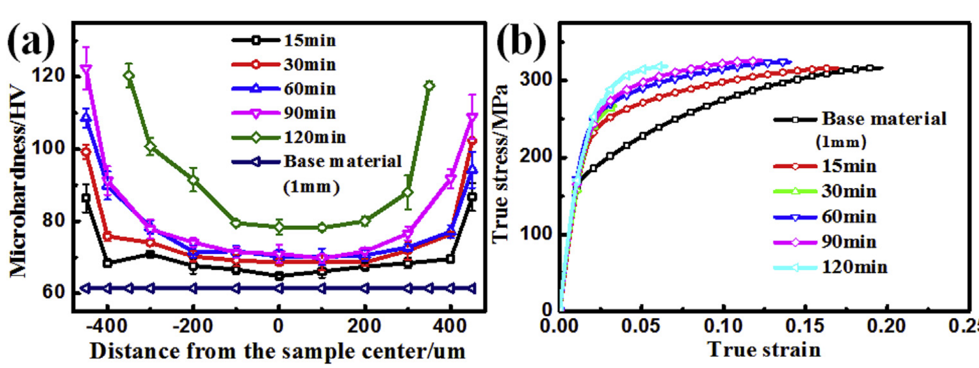

Fig. 4. (a) Micro-hardness tested on lateral section from top to bottom surface of SMATed specimens with different processing time; (b)Stress-Strain curves of samples for base and SMATed material.

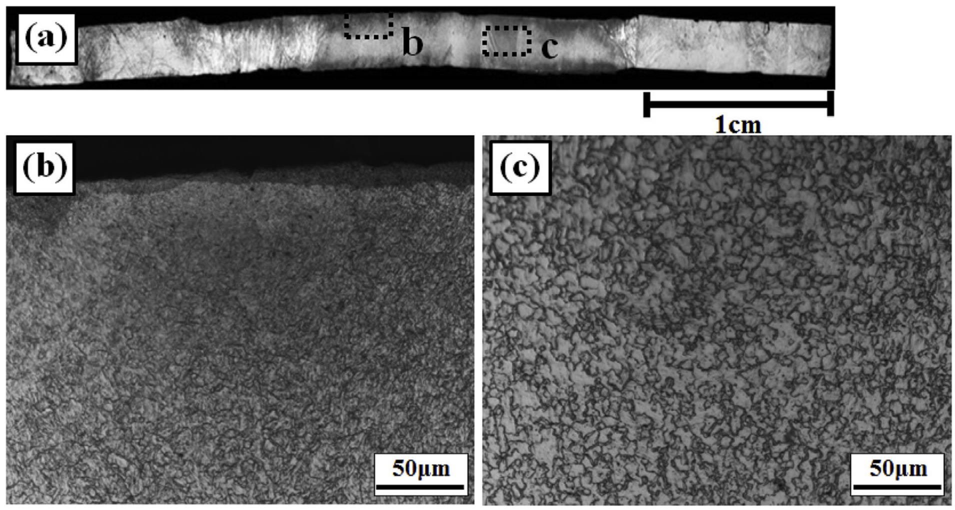

Fig. 5. (a) Macro-morphology of a SMAT + FSP treated specimen; (b) Morphology of severely deformed layer on the surface; (c) Microstructure of FSPed region.

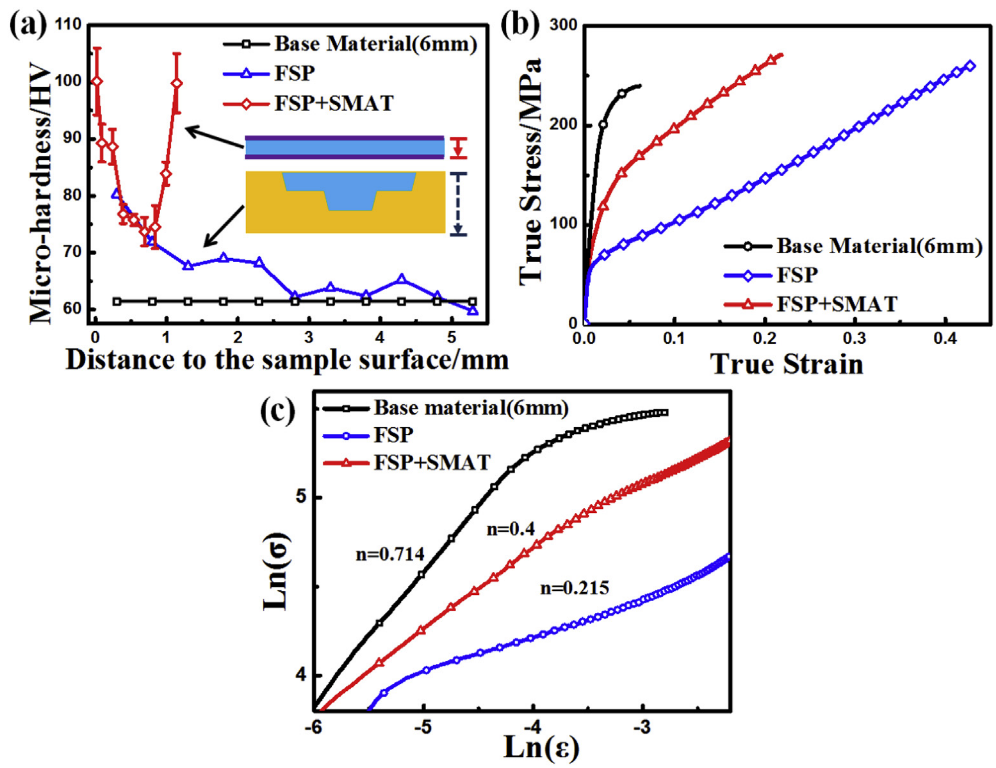

Fig. 6. (a) Micro-hardness along the ND right in the center of processed region (b) Stress-strain curves of samples treated in different ways (c) Strain hardening index of different treatments.

treatment of FSP + SMAT were then obtained with the processed region seated across the center. A typical morphology of the plate on ND-TD plane after combined treatment was shown in Fig. 5a–c. It could be seen that the microstructure got significantly refined near the processed surface, which was similar to the metallographic structure of base material after pure SMAT processing. On the other hand, in the center part of the plate after combined treatment of FSP + SMAT, the microstructure was similar to the base material after pure FSP which indicated that SMAT only effectively modified the microstructure near the surface within confined thickness (< 180 μm). Micro-hardness was also measured on the ND-TD plane along ND right in the center of the processed region. The micro-hardness distribution profiles of these three different processing routings were drawn on a single figure for comparison, as shown in Fig. 6a. It clearly shows that FSP caused an increment in micro-hardness and a further raise on the surface was obtained after SMAT. The tensile tests results also indicated a raise in yield stress from ~52 Mp to ~70 Mpa after SMAT (34.6% increment); and an obvious strengthening in flow stress, as well as in strain hardening index, could be more clearly seen in Fig. 6b and c. Although the SMAT routing reduced the elongation of the FSPed samples, which showed an extraordinary high value of ~40%, the remaining value (20%) was still higher than the initial twin-roll casting plate (6%).

4. Conclusion

In summary, friction stir processing (FSP) significantly refined the grain structure of the initial AZ31 Mg alloy plates. Homogeneous equiaxed-grain microstructure with tilted basal texture was produced in stirred region after FSP. The elongation of the experimental material increased from 6.1% to 40% after FSP, accompanied by an obvious loss in yield stress. On the other hand, surface mechanical attrition treatment (SMAT) introduced severely localized deformation layers on sample surfaces, which greatly strengthened the local micro-hardness and yield stress of parental matrix. By combining SMAT with FSP, the loss in yield and flow stress caused by FSP could be well compensated by SMAT routings which optimized the mechanical properties of the experimental material.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.msea.2019.04.051.

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