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# Changes in Grain Size, Texture, and Mechanical Properties of AZ31/($TiO_2$)p Nanocomposites Processed by Isothermal Multidirectional Forging

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**Abstract**

In the current study, magnesium-matrix AZ31/ 1.5 vol.% ($TiO_2$)p nanocomposites manufactured using stir casting underwent extrusion process. The as-cast ingots were extruded, then processed by multidirectional forging (MDF) up to 8 passes at constant temperature of 320°C. Investigating microstructures showed that after the second pass, the size of matrix grains underwent a significant decrease. However, this decrease didn't continue in subsequent passes and grain size increased at the fourth pass. In the sixth pass, grain size decreased again, resulting in the smallest microstructure in all the samples. However, in the last two passes, grain size increased similar to the case of the fourth pass. The results of shear punch and Vickers' microhardness tests showed that changes in shear yield strength, ultimate shear strength, and hardness followed a similar trend. Furthermore, the results of these tests showed that the best mechanical properties are observed in the first two passes after which no further improvement is observed in shear strength and hardness of the samples while fourth, sixth, and eighth passes resulted in better mechanical properties compared to the extruded sample.

## 1. Introduction

Magnesium-matrix nanocomposites are among advanced materials which are used to overcome the weaknesses of magnesium alloys including low strength and elastic modules. Due to addition of strengthening particles, these materials have better mechanical properties compared to magnesium alloys while retaining their low density. Magnesium-matrix composites are often used to reduce fuel consumption in various industries including aerospace and automobile industries as well as in sport equipment and other industrial applications [1-3]. SiC whiskers/ particles, graphite particles/fibers, $Al_2O_3$ particles/fibers, carbon nanotubes (CNTs), $TiO_2$ particles, Cu particles and other materials are often used as strengthening particles in magnesium-matrix composites [4, 5]. Among heterogeneous strengthening phases, ceramic particles offer advantages including isotropic properties, great mechanical characteristics, chemical inactivity, high thermal stability and controllable expansion coefficient, which makes them ideal for using in metal-matrix composites [6].

The processing method selected for manufacturing of composites is important in achieving uniform distribution of strengthening particles and suitable mechanical properties [7]. Composites strengthened with nanoparticles are manufactured using various methods including powder metallurgy, impact casting, stir casting, mechanical alloying, and several other methods among which stir casting is the cheapest and simplest method [8-10]. In the study by Poddar et al., magnesium-matrix composites strengthened using SiC particles were manufactured using stir casting method

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which resulted in uniform distribution of particles [9].

Although casting methods are simple and cost-efficient, presence of casting defects (such as pores) in casted composites results in decrease in mechanical properties and limits their applications. Furthermore, lack of proper control over process parameters during casting can result in nonuniform distribution of particles in the matrix.

Using secondary processes including extrusion can eliminate porosity and improve mechanical properties of the composites [11, 12]. Wang et al. investigated the effects of extrusion process on mechanical properties and microstructures of AZ91/SiCp composites and reported that extrusion process eliminates segregation between particles in casted composites and significantly improves particle distribution [13]. Using Severe Plastic Deformation (SPD) methods is one of the most effective techniques for improving particle distribution and mechanical properties of composites. SPD processes are metal forming processes which are used to produce ultra-fine grain metals. The main aim of SPD processes is to manufacture light-weight parts with high strength [14].

Multi-Directional Forging (MDF) is one of the most important SPD processes where each pass creates a large plastic stress in samples while the shape shows no significant changes at the end of the process. In common deformation methods including forging, extrusion, and rolling, several passes are required to produce the necessary stress which can result in thin products which are unsuitable for industrial applications. However, SPD methods are designed to overcome this limit [7, 14].

One of the most important studies regarding SPD processes and their effects on microstructure and properties of magnesium-matrix composites is the study by Qiao et al. which investigated the changes in microstructure and mechanical properties of AZ91/Nano-SiCp composite processed using ECAP method [4].

In another study, Nie et al. investigate the effects of MDF process on microstructures and tensile properties of magnesium-matrix composites strengthened with SiC particles and reported that grain size in MDF composites decreases compared to casted samples. Furthermore, the result of tensile tests showed that increase in the number of MDF passes improves the tensile yield strength and ultimate tensile strength of the samples [7]. In the current study, magnesium-matrix AZ31/$TiO_2$ nanocomposites manufactured using stir casting underwent extrusion process. Then, shear punch and Vickers' hardness tests were used to investigate the mechanical properties of these samples at room temperature and changes in microstructures were investigated using Scanning Electron Microscopy.

## 2. Materials and Methods

The magnesium alloy AZ31 (Mg–3wt.% Al–1wt.% Zn–0.2wt.% Mn) was used as the matrix and $TiO_2$ particles with density of 4.23g/cm$^3$ and melting point of 1843°C were used as the strengthening phase. In this study, 1.5vol.% of strengthening powder with average particle size of 38nm was used in sample manufacturing.

The AZ31/($TiO_2$)p nanocomposite was manufactured using stir casting method. In this process, AZ31 alloy parts were melted in a graphite crucible using an electrical resistance furnace. After reaching the melting point of 720°C, the strengthening particle powder was injected into the molten alloy using argon gas and this process continued for 10 minutes. In order to create sufficient turbulence and achieving uniform particle distribution in the matrix, during the powder injection process, a stirrer made from Refractory steel 41-48 was used to stir the molten mixture and create a composite slurry which was then transferred to a steel mold. After the casting process, samples underwent extrusion process. Extrusion process was carried out at temperature of 400°C and extrusion ratio of 3.78 in order to improve mechanical properties and achieving better microstructures in casted samples.

After the extrusion process, the cylindrical extruded bars were cut into cubic shapes through machining and then again cut into tickets with dimensions of 13×13×20mm (Fig. 1).

**Fig. 1.** Samples after machining process.

In the next step, tickets were processed using 2, 4, 6, or 8 passes of MDF process at 320°C. The loading was applied using an MTS equipment with compression speed of 1mm/min. The lubricant used in the process was a Teflon tape which was used to fully cover the samples before each pass. The 1×1×1.54mm dimension ratio of the samples was retained during the MDF process and the loading direction changed by 90 degrees for each pass (using Z→Y→X →Z sequence). Fig. 2 shows the schematic of MDF process.

In order to investigate the changes in microstructures during MDF process, Scanning Electron Microscope (SEM) was used. To this end, samples with thickness of 3mm were cut from tickets after extrusion and different passes of MDF process using a wire cut equipment. These samples were then mounted, polished, and etched (using a solution of 10mL acetic acid, 4.2g picric acid, 10mL distilled water and 70mL

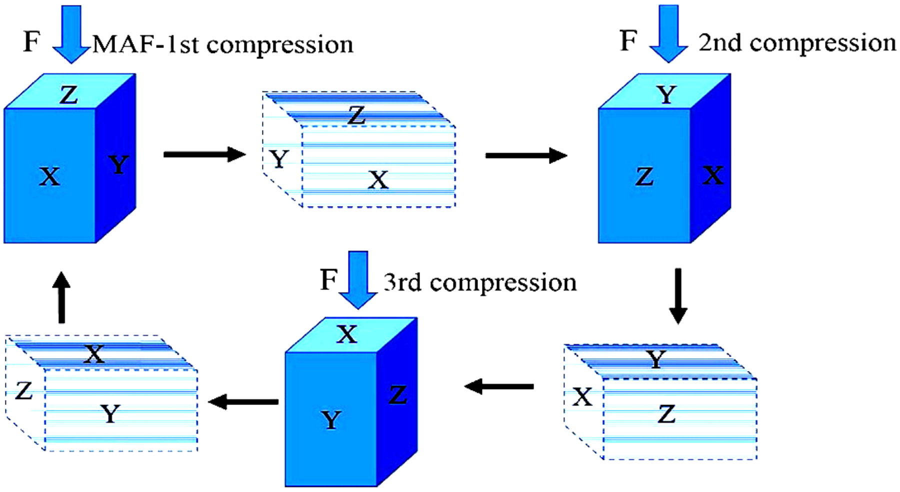


**Fig. 2.** Schematic of MDF process [15].

ethanol). The prepared samples were then investigated using SEM imaging. Texture studies were also carried out by measuring the intensity distributions of the {0002} pole figures on the planes perpendicular to the extrusion and MDF directions.

Investigating the mechanical properties of the extruded and MDF samples was carried out using Shear punch test (SPT) and Vickers' microhardness distribution test. The shear punch test was used to determine the yield shear strength and ultimate shear strength of the samples [16]. To this end, pieces with thickness of 1mm were cut from different samples and polished to remove the effects of cutting until reaching the thickness of 0.7mm. The test was carried out at room temperature using SANTAM equipment. Fig. 3 shows the schematics of SPT.

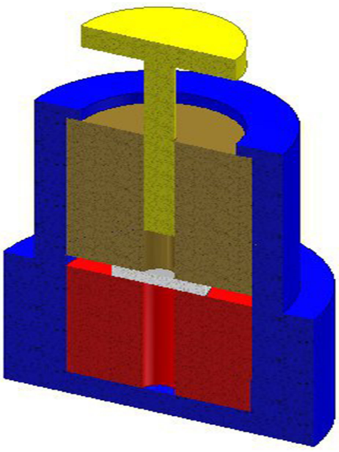

**Fig. 3.** Schematic of SPT.

Vickers' microhardness test was used to determine the hardness of materials using samples with thickness of 3mm. These samples were mounted and polished before being tested using Bareiss VTE 6046 hardness test equipment. This test was repeated at 100 different points for each sample and the average of all hardness values was reported at average surface hardness of that sample. Furthermore, a colored contour of hardness distribution was created for each sample using Surfer software.

## 3. Results and Discussion

### *3.1. Microstructure*

SEM images showing microstructures of $AZ31/TiO_2$ nanocomposites after extrusion and different passes of MDF process are presented in Fig. 4 while Fig. 5 shows the changes in the average grain size ($D_{avg}$) based on the number of MDF passes. As can be seen, with the increase in the number of MDF passes, grain size decreases compared to the extruded samples, resulting in lower $D_{avg}$. According to Fig. 4, it is clear that the changes in average grain size includes periodic increase and decrease at different number of passes. Fig. 4b shows that the $D_{avg}$ value in the second pass decreases to 14.30$\mu$m. This decrease in grain size can be due to better distribution of strengthening particles in the matrix and dynamic recrystallization which result in smaller grain size. However, the $D_{avg}$ value increases slightly in the fourth pass and in the sixth pass this value reaches 10.59$\mu$m which is the lowest value in all samples. Finally, at the end of the eighth pass, grain size increases, similar to the fourth pass.

Uniform distribution of particles in the matrix can be a requirement for achieving homogenous structures and reliable mechanical properties. The introduction of the nanoparticles in metal matrix can create nucleation sites during solidification which can result in refined microstructure [17]. Furthermore, when the as-cast materials are processed using thermo-mechanical processes including hot extrusion, nanoparticles act as hard barriers and prevent the movement of grain boundaries and stop grain growth. As a result, agglomeration of particles can increase grain size in parts with less concentration of strengthening particles.

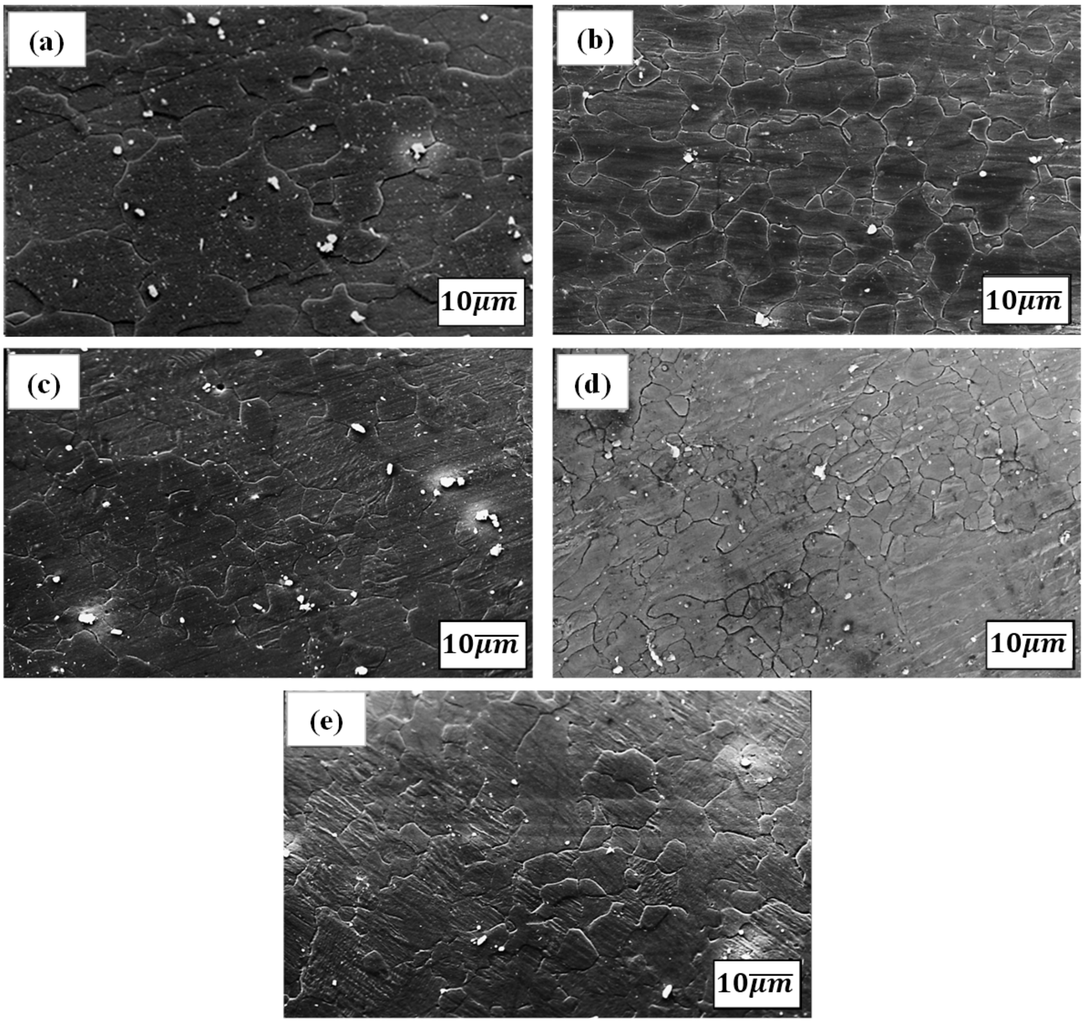


**Fig. 4.** Changes in microstructures of AZ31/$TiO_2$p nanocomposite samples in different conditions including a) Extruded samples, b) After 2 MDF passes, c) After 4 MDF passes, d) After 6 MDF passes, and e) After 8 MDF passes.

Although using SPD processes including MDF process can improve particle distribution, some studies have shown that constant changes in flow direction of particles with increase in the number of MDF passes can result in nonuniform distribution of particles in some of the passes [8]. Therefore, one of the reasons for the increase in grain size in fourth and eighth passes can be the nonuniform distribution of particles due to 90-degree change in loading direction between passes.

Furthermore, the processing temperature of the composite is another important factor in determining its microstructure because higher temperatures result in higher heat transfer to the samples at higher MDF passes which can result in dynamic recrystallization and increase in grain size.

In the study by Liao et al., AZ91-SiC nanocomposites were processed by Cyclic Closed-Die Forging (CCDF), which was carried out at 300°C and 400°C. Microstructurl investigation showed that grain size created at 300°C was 5.2$\mu$m while the grain size achieved at 400°C was 23$\mu$m. This indicated that lower processing temperature is more suitable for manufacturing of composites with smaller grain size [18].

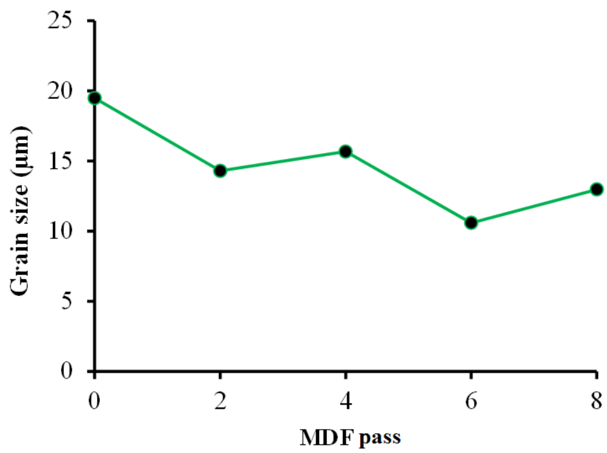


**Fig. 5.** Changes in average grain size based on the number of MDF passes.

### *3.2. Shear Behavior*

Fig. 6 shows the results of shear punch test of composite samples after 2, 4, 6, and 8 MDF passes and compares them with the result of the extruded sample. In these graphs, changes in shear stress is presented versus dimensionless displacement.

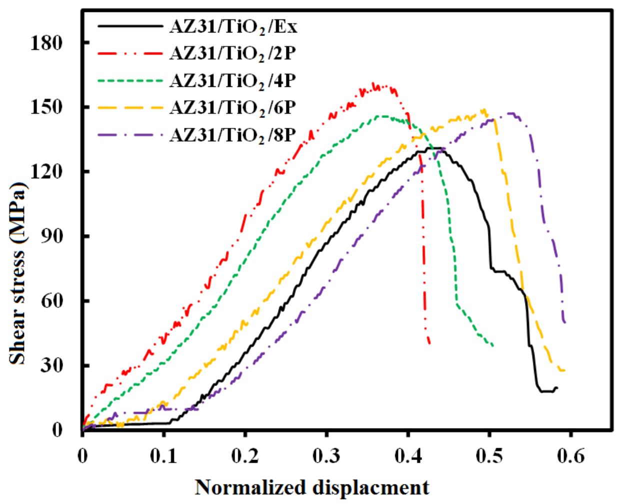


**Fig. 6.** The shear stress graphs based on dimensionless displacement for extruded samples and MDF samples with different number of passes.

In these graphs, the divergence point from elastic behavior is known as the Shear Yield Stress (SYS). Furthermore, the maximum amount of shear stress is known as the Ultimate Shear Stress (USS). Table 1 shows different values of shear yield stress and ultimate shear stress in samples processed under different conditions.

Based on Fig. 6 and Table 1, it can be said that at first, both shear yield stress and ultimate shear stress of the composite increase after MDF passes. The values for SYS and USS after the second MDF pass are 135.67MPa and 161.51MPa, respectively which show 30.97MPa (29.58%) and 30.63MPa (23.40%) improvement compared to values before MDF process. In the subsequent passes of MDF, the changes in mechanical properties follow a periodic increasing and decreasing trend. As can be seen in Table 1, in the fourth pass, SYS and USS decrease compared to the previous sample but increase again in the sixth pass. In the eighth pass, similar to the fourth pass, SYS and USS show another decrease. In general, comparison between mechanical properties of samples manufactured under different conditions shows that despite some decrease in mechanical properties after some MDF passes (For example, the fourth pass over the second pass), the samples processed using MDF show generally superior characteristics (in all passes) compared to the extruded sample (before MDF process). Furthermore, comparison between properties of MDF samples shows that the best results are obtained after the second pass while the samples processed with four MDF passes have the lowest mechanical properties in all MDF samples.

In general, it is an accepted fact that the properties of metals with Hexagonal closed pack (HCP) structure including magnesium are greatly dependent on their grain size due to having limited slippage systems [19]. In this regard, Hall-Petch equation shows that material's yield strength is inversely proportional to the square root of grain size ($d^{1/2}$). However, we have to remember that the strengthening of a composite is not simply dependent on its grain size and that along with Hall-Petch mechanism, several other mechanisms including Orowan, Load-bearing effect, and thermal expansion coefficient mismatch also affect the strengthening of composites [20].

It can be seen that changes in the shear strength after different MDF passes have a similar trend to the changes in grain size. However, changes in grain size alone cannot explain the changes in shear yield stress and ultimate shear stress of the extruded and MDF samples. This is due to the fact that average grain size in the second pass (14.30$\mu$m) is higher than average grain size in sixth and eighth passes (10.59$\mu$m and 12.97$\mu$m, respectively) but second pass of MDF results in the highest shear strength. One other factor affecting the strength of magnesium alloys is the matrix changes during SPD process. In fact, strength of magnesium alloys improves with decrease in grain size only if matrix remains unchanged [21].

For example, in the study by Akbaripanah et al., AM60 alloy was processed with 6 passes of ECAP process and the results showed that despite smaller microstructures after fourth and sixth passes, best mechanical properties (YS, UTS) were resulted from the second pass of the process which was due to the texture softening of matrix decreasing the alloy's strength. They also investigated the shear behavior of this alloy and concluded that tensile and shear behaviors of the alloy are similar during different ECAP passes [21].

**Table 1**
The values for shear yield stress and ultimate shear stress of extruded and MDF samples.

| | Number of passes | | | | |
|---|---|---|---|---|---|
| | Extruded | 2 | 4 | 6 | 8 |
| Shear yield strength (MPa) | 104.70 | 135.67 | 119.43 | 127.61 | 122.75 |
| Ultimate shear strength (MPa) | 130.88 | 161.51 | 145.61 | 148.88 | 146.97 |

In a study by Nei et al., AZ91/SiCp nanocomposites were subjected to MDF at 400°C for 6 passes. Investigating of microstructures and the results of tensile strength test showed that although increase in the number of MDF passes results in more homogeneous microstructure and SiC particle distribution, the yield strength after the first pass is higher than after the third pass. They believed the reason for this difference to be the texture softening due to rotation of base planes. These matrix changes disrupted the effects of strengthening mechanisms on composites' strength [7].

Finally, these results show that along with changes in microstructure, matrix changes can also explain some of the changes observed in the properties of samples during MDF process.

### *3.3. Microhardness Measurement*

In order to determine the homogeneity of hardness distribution on different locations on samples' surfaces, the hardness contour for each sample is presented in Fig. 7. Fig. 7a shows hardness distribution in nanocomposite samples before MDF process while contour maps presented in figures 7b, 7c, 7d and 7e are for MDF samples with 2, 4, 6, and 8 passes of the process. Furthermore, the graph presented in Fig. 7f shows the average microhardness values of each surface obtained using Vickers' microhardness test for different samples. The average hardness for extruded and MDF samples with 2, 4, 6, and 8 passes are equal to 61.8, 77.4, 67.6, 71.3, and 69.1 Vickers.

As can be seen in Fig. 7f, the highest average surface hardness is observed after the second MDF pass and with the increase in the number of MDF passes, average hardness value decreases before increasing again. The standard deviation shown on the contour maps indicates that similar to microstructural changes, at the end of the second pass, microhardness distribution is also significantly more uniform. Furthermore, it can be seen that the lowest homogeneity in hardness distribution is observed in the fourth MDF pass while the most homogeneous hardness distribution is observed in the sixth pass (when compared to other MDF samples).

Fig. 7f shows that changes in microhardness follow the same trend as the ultimate shear strength. The graph for SYS, USS and Hv values versus the number of MDF passes is presented in Fig. 8.

Finally, it is necessary to mention that the distribution of strengthening particles plays an important role in creating a homogeneous structure. In other words, a more uniform distribution of strengthening particles results in more homogenous microstructure and hardness distribution.

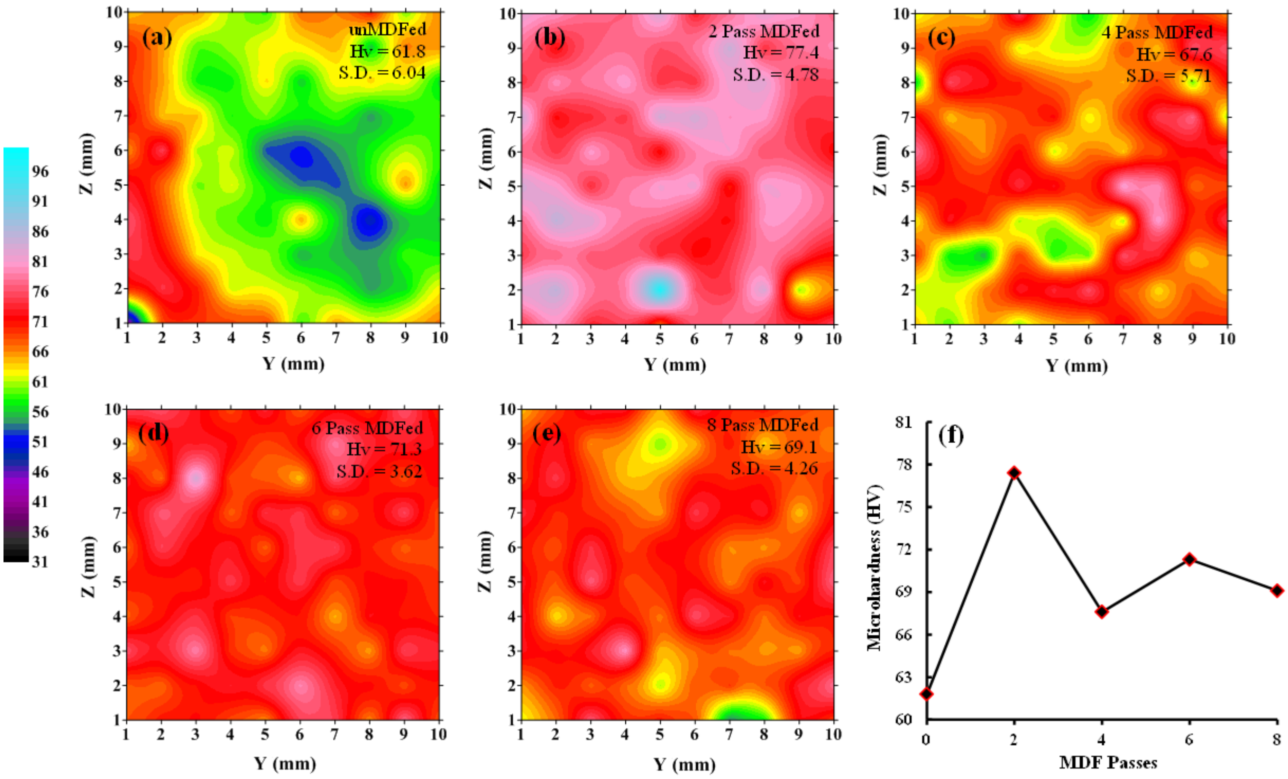


**Fig. 7.** The colored contour map of microhardness distribution on the surface of different AZ31/$TiO_2$p samples: a) Extruded sample, b) After 2 MDF passes, c) After 4 MDF passes, d) After 6 MDF passes, and e) After 8 MDF passes. The dependence of the microhardness of the MDFed sample on the number of passes is also shown in (f).

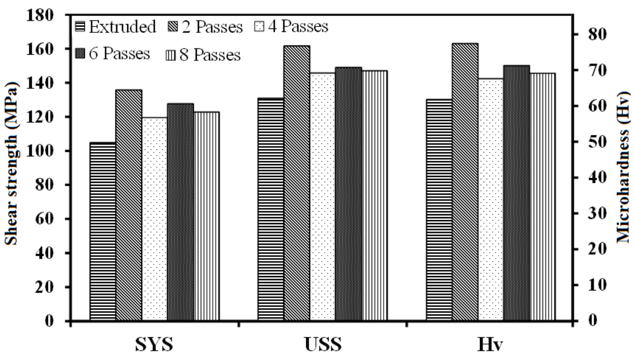


**Fig. 8.** Changes in mechanical properties of AZ3/$TiO_2$p nanocomposites based on processing conditions.

### *3.4. Textural Studies*

As can be seen in the Fig. 9, a typical fiber texture was produced due to extrusion in a way that the basal planes' normal is perpendicular to extrusion direction.By applying the MDF, the basal planes tend to be inclined from the periphery of the poles toward the center, meaning ND, due to forging and a split type poles are clear. The intensity of MDFed specimens are higher than extruded sample. During MDF, deformation resulted in increasing of the stored strain and DRX is able to reduce this strain. But, appearance of few DRXed grains at each pass confirms that strain accumulation has been happened and the texture intensity increased.

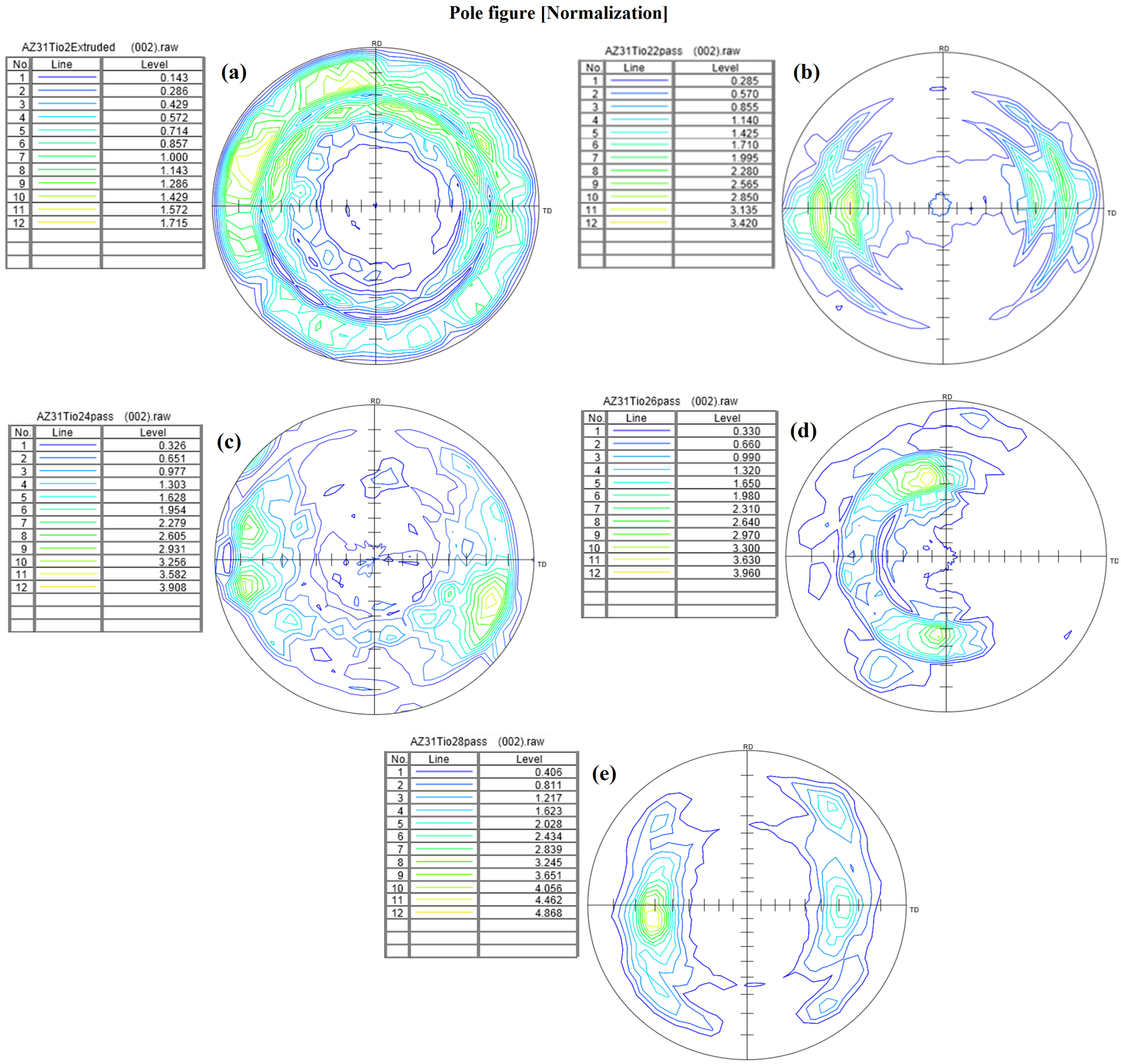


**Fig. 9.** (0002) pole figures of the different AZ31/$TiO_2$p samples: a) Extruded sample, b) After 2 MDF passes, c) After 4 MDF passes, d) After 6 MDF passes, and e) After 8 MDF passes.

As a similar result for all MDFed samples, it is clear that the maximum intensity is located at the angles about 50-80 degree in respect to ND. These type of rotation resulted in higher amount of Schmid factor in comparison to the extruded sample, which can be results in improvement of formability. On the other hand, the strengthening of processed samples is competitive procedure between grain size hardening, i.e. Hall-Petch criteria, and texture softening effect. But considering the Table 1 and grain size, it can be concluded that the strength of all samples is more dependent on grain size than texture evolution.

## 4. Conclusions

This study investigated the microstructures and mechanical properties of AZ31/$TiO_2$p nanocomposites processed using extrusion and multi-directional forging processes. The important conclusions based on the results are as follows:

- Investigating microstructures showed that MDF process results in decrease in grain size. The average grain size decreases after the second pass of MDF process but increases between second and fourth MDF passes. Grain size decreases again after the next two passes while the final two passes result in a slight increase in grain size. Nonuniform distribution of strengthening particles and static recrystallization can be the reasons for this increase in the grain size.

- The results of SPT test show that shear yield strength, ultimate shear strength, and surface hardness of samples have the highest values after the second pass of MDF process. With increase in the number of MDF passes, a periodic increase and decrease was observed in these properties. However, the values for various mechanical properties in MDF samples (regardless of the number of passes) remained higher than samples without MDF process. This means that multidirectional forging process results in improving hardness and shear behaviors of the samples but the results of SPT test and Vickers' microhardness show that after the second pass of MDF, subsequent passes do not result in significant improvement of mechanical properties, which makes further MDF passes unnecessary.

- Based on changes in microstructures of the samples, it can be concluded that changes in the grain size alone connot explain the increase or decrease in mechanical properties. The strength of MEFed samples is determined by the competition of grain size strengthening effect and texture softening effect, and the texture softening depends on the texture type and texture intensity.

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