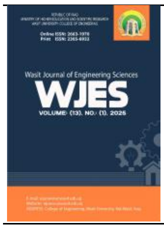

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Original Research

Influence of High-Pressure Torsion Method on the Mechanical Properties of Ti-6Al-4V.

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ABSTRACT

Titanium alloys, particularly Ti-6Al-4V, are among the most widely used groups of alloys and have been extensively used for biomedical and aerospace applications due to their mechanical properties. The mechanical properties of the sample were evaluated through microhardness measurements. The tests were performed using a Vickers Microhardness Test apparatus to obtain hardness (HV) values, which were used as an indicator of the material's behavior. However, improving its mechanical properties remains a critical research priority to ensure efficient behavior and long-term viability. Therefore, this article aims to explore the effect of severe plastic deformation, particularly the High-Pressure Torsion (HPT) method, on the microstructure and mechanical properties of Ti-6Al-4V under an applied pressure of 3 GPa at ambient temperature. Various numbers of HPT turns (1, 3, 5, 7, and 9) are employed and compared with the bulk sample. The findings showed that the microstructural development occurred in the edge area .Based on SEM and OM images, a change in structural composition was observed, and microhardness increased significantly by 32.4% when the number of HPT turns was increased to 7 compared with the bulk sample. The mechanical properties of the Ti-6Al-4V alloy showed improvement after turns 5 and 7.

Keywords: High-pressure torsion, severe plastic deformation, titanium alloy, microhardness.

1. Introduction

Due to its amazing combination of robust mechanical properties, lightweight composition, and outstanding corrosion resistance, Ti6Al4V emerged as the first titanium alloy used as a viable biomaterial. Several structural applications in aerospace, power, and sports equipment use the Ti-6Al-4V alloy due to its bimodal microstructure, low density, high strength, excellent toughness and corrosion resistance, as well as its outstanding high-temperature properties and formability. Medical and dental implants also make use of this metal due to its exceptional biocompatibility. Several researchers have recently assessed the HPT processing of the alloy to enhance its potential qualities and practical applications. Reviewing these publications reveals that, with one exception, all investigations were carried out using commercially available Ti-6Al-4V alloys that underwent HPT processing [1]. The microstructure and characteristics of the Ti-6Al-4V alloy changed during high-pressure torsion (HPT), with the specific changes depending on the initial microstructure. This study builds on that work by examining the effects of different starting bimodal microstructures on the Ti-6Al-4V alloy when processed at room temperature. In particular, the purpose of the study was to analyze how the grain size and microhardness of various microstructures changed during HPT processing 2], [3]. **The** microstructural refinement technique has been used to improve grain refinement, including severe plastic deformation (SPD). The SPD is a new metal-forming technique in which the material undergoes significant plastic deformation without altering its size or shape. Materials can have their grain size reduced for specific applications using SPD techniques. at the same time, enabling remarkable grain refinement [4]. It is well established that SPD processing yields ultra-fine grained (UFG) materials with distinctive mechanical and physical characteristics. Potential industrial uses of SPD-processed materials include precise devices and biomedical implants [5], [6]. When designing and implementing biomedical materials, it is crucial to keep their therapeutic goals in mind [7], [8],[9]. Finally, for orthopedic implants to be effective, they must possess exceptional antibacterial and

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osseointegration properties. This means the implant must reliably connect to the surrounding bone without causing infection or fibrous tissue formation. When there is no relative movement between the implant and the surrounding bone under functional stress, we say that osseointegration has occurred.[10], [11]. Based on the above, Ti-6Al-4V is a material of critical strategic importance due to its numerous vital applications, particularly in the biomedical and industrial fields, aerospace, power, and sports equipment. Therefore, enhancing its mechanical properties is a top priority to ensure efficient performance and long-term viability. Accordingly, this research aims to improve these properties in this alloy using High-Pressure Torsion (HPT), one of the severe plastic deformation techniques.

2. Experimental Procedure

2.1 Materials

2.1.1 Material and Initial Preparation:

A Ti6Al4V alloy rod was provided at a diameter of 14 mm and a length of 100 mm. The rod was first machined to a diameter of 9 mm. Subsequently, it was sliced into 1.5 mm-thick samples using electrical discharge machining (EDM). The samples were subsequently ground on both sides using conventional 400, 600, 800, 1200, and 2000 grit emery papers to achieve a final thickness of 0.8 mm.

2.1.2 HPT Processing:

The HPT process was conducted at ambient temperature using the apparatus depicted in Fig. 1. This facility included upper and lower circular anvil dies and an inhibited die made from nitrided high-strength tool steel, which ensured the required surface hardness while maintaining exceptional dimensional precision. The die contains a spherical depression at the center of the inner surface of each anvil, measuring 0.3 mm deep and 10 mm in diameter. Figure 1 illustrates that when the two anvils are brought into contact for the torsional straining process, the two depressions are accurately aligned. Each HPT test entailed positioning a sample in the recess on the upper surface of the lower anvil, compressing the anvils to exert the specified pressure P, and subsequently rotating the lower anvil at 1 rpm to induce torsional strain [12], [13]. This type of HPT processing is often referred to as constrained HPT, as the disk is effectively secured under back-pressure. The practical application of high pressure and simultaneous torsional straining results in a restricted outflow of sample material at the disk's periphery, as illustrated in Fig. 2, leading to a marginal reduction in the disk's thickness with each straining process. The Ti6Al4V alloy was processed in the aforementioned HPT facility at 3 GPa with 1, 3, 5, 7, and 9 revolutions.

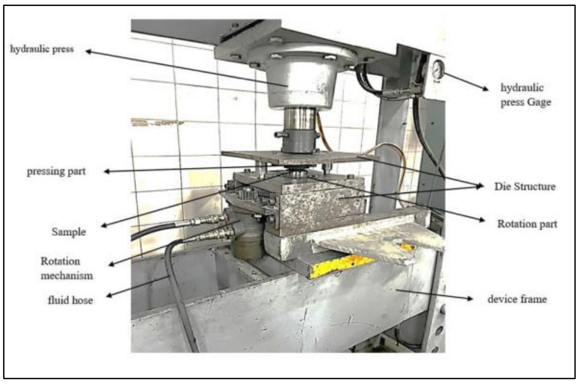

Figure1. High Pressure Torsion HPT rig that used in the current work.

2.1.3 Post-Processing and Characterization:

Grinding and polishing were performed consecutively on all processed samples with 400, 600, 800, 1200, 2000, and 3000-grit emery sheets.this is accomplished in stages on a grinding and polishing machine . Final polishing was conducted using alumina suspensions of particle sizes 6, 3, 1, and 0.5 µm to achieve a mirror-like finish with minimal residual surface. The surface roughness of the polished samples was assessed using stylus profilometry (Dektak, Veeco), with the average roughness (Ra) consistently measured at sub-10 nm.

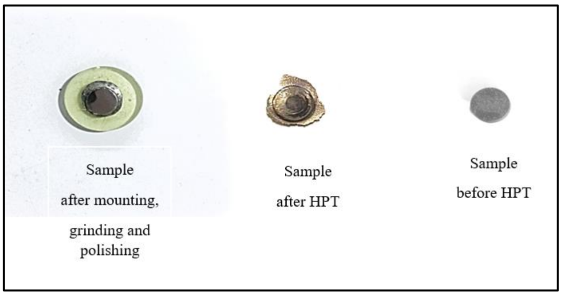

Figure 2. The samples after and before processing with HPT

2.2 Vickers Hardness Test

Vickers microhardness testing was performed to evaluate localized hardness variations in HPT-treated Ti6Al4V samples. The measurements were performed with a microhardness tester (HST, China) with a load of 1 kg and a dwell time of 10 seconds, as shown in Figure 3. Hardness measurements were obtained along a diagonal line through the samples at 1 mm intervals. At each specified location, the mean hardness was determined by averaging the individual Hv values obtained from three separate samples at that site. These measurements yielded extensive quantitative data on the specific variation of Hv along the diagonal of each sample under different testing settings. The standard error of the mean was computed for the three hardness measurements at each specified site. A Vickers microhardness test was performed in accordance with ASTM E92-72.

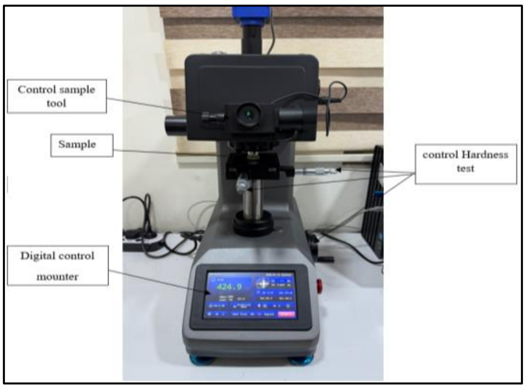

Figure 3. Vickers Microhardness testing machine

2.3 Microstructure observations

Microstructural analyses were performed on polished and as-received samples. The samples' clean, flat surfaces were etched with a solution of mixed nitric acid and hydrofluoric acid (10 mL HF, 5 mL HNO3, and 85 mL deionized water), and then examined using a scanning electron microscope (SEM; Thermo Scientific). The Ti–6Al–4V alloy's inherent corrosion resistance made it challenging to analyze the structure after several rotations. This technique was used to evaluate the alloy's characteristics before and after high-pressure torsion (HPT) processing, namely at 1, 3, 5, 7, and 9 turns.

3. Results and Discussion

3.1 Vickers Microhardness

Figure 4 shows the average microhardness values as a function of distance from the disk center after HPT processing. Before HPT processing, the alloy we examined had a microhardness (HV) of approximately 320. After one cycle of HPT, the average microhardness increased to 385 HV; the radial microhardness distribution was nonuniform, ranging from 380 HV at the edge to 370 HV in the core. After three rotations, the microhardness increased to 390 HV at the edge, although it remained relatively low at around 352 HV in the core. As the number of rotations increased from 1 to 5, the microhardness improved a lot. After five revolutions, the disk's edge values approach saturation, while the hardness values in the center increase significantly. After seven rotations, the microhardness on the outer edge stays roughly the same. In contrast, the hardness in the center continues to increase. After 9 rotations, the average microhardness is around 415 HV, and it is virtually the same throughout, except for a tiny area in the middle. As the number of rotations during high-pressure torsion (HPT) increases, the central area with lower hardness becomes smaller. The microhardness distribution remains mostly consistent, except for a central area about 1 mm wide. Hardness measurements were conducted at the disc's periphery, since this area endures maximum shear strain and hence demonstrates the most pronounced impact of the HPT procedure, as shown in Figure 5. The Vickers microhardness data obtained using the High-Pressure Torsion (HPT) technique exhibit considerable variation with increasing rotation. Without HPT, the bulk sample exhibited the lowest hardness due to its coarse-grained microstructure and reduced dislocation density [15]. Microhardness increased significantly after one HPT turn relative to the bulk sample. This rise might have occurred because HPT induces significant plastic deformation. This initiates the purification process and causes dislocations to occur much more frequently. These alterations to the microstructure strengthen the material by preventing dislocations from moving. The hardness increased significantly after being changed three more times. The major causes are that high-angle grain boundaries are growing larger, and grains are becoming larger. The Hall-Petch relationship states that as grain size decreases, a material becomes harder and stronger. This means that the material becomes stronger as the grains get smaller. The hardness value was almost the same after five turns as it was after three turns. Additionally, making it more deformed doesn't make it much harder. The hardness value was highest after seven turns. This might be because the structure with very tiny grains is more uniform, with denser grain boundaries, which inhibit dislocation motion and make the material stronger. The toughness decreased a little after nine rotations instead of seven; increased hardness (HV) is usually associated with decreased toughness due to decreased ductility and increased restriction of dislocation movement. This decline might be due to dynamic recovery processes, or it could result from grains becoming slightly coarser when the material is deformed for a long time and accumulates significant strain energy [16]. These steps could reduce the number of dislocations and relieve internal tension [16], potentially making the material slightly less hard. The findings show that the microhardness increases significantly following HPT processing as compared to the bulk state. After seven turns, it reaches its maximum position, then drops a bit as the degree of distortion increases. The percentage increase in hardness depended on the number of turns, as shown in Table 1.

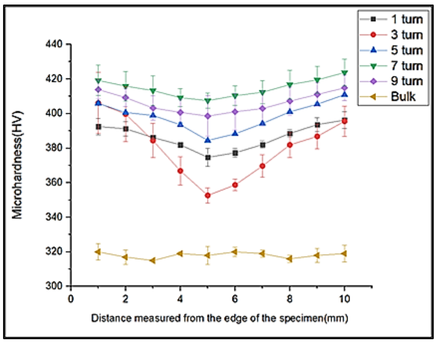

Figure 4. Values of the Vickers hardness for all samples

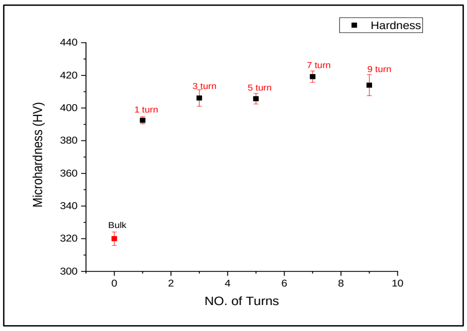

Figure 5. Evolution of Vickers microhardness in Ti-6Al-4V alloy as a function of HPT turns (1 to 9) processed at 3 GPa.

Table 1.The percentage of improvement in hardness.

NO. Turn	Percentage of Hardness
1	23.8%
3	23.6%
5	28%
7	32.4%
9	29.6%

3.2 Microstructure observations and measurements

The shear strain in the edge areas of disks treated by HPT is known to be much greater than in the core region. Thus, the microstructural development at the edge area often progresses at a faster rate until, in the end, the whole disk becomes quite homogenous in many materials. This is why, after varying the number of rotations, the experiment focused on microstructural changes near the disk edges [18]. Images of the disk edges of the Ti-6Al-4V alloy were taken using a scanning electron microscope (SEM), and Optical micrographs of the microstructures of the Ti-6Al-4V alloy before and after 1, 5, and 9 turns of HPT. It is evident from comparing Figures 6 and 7 that the HPT process has significantly distorted the grains[18], [19].

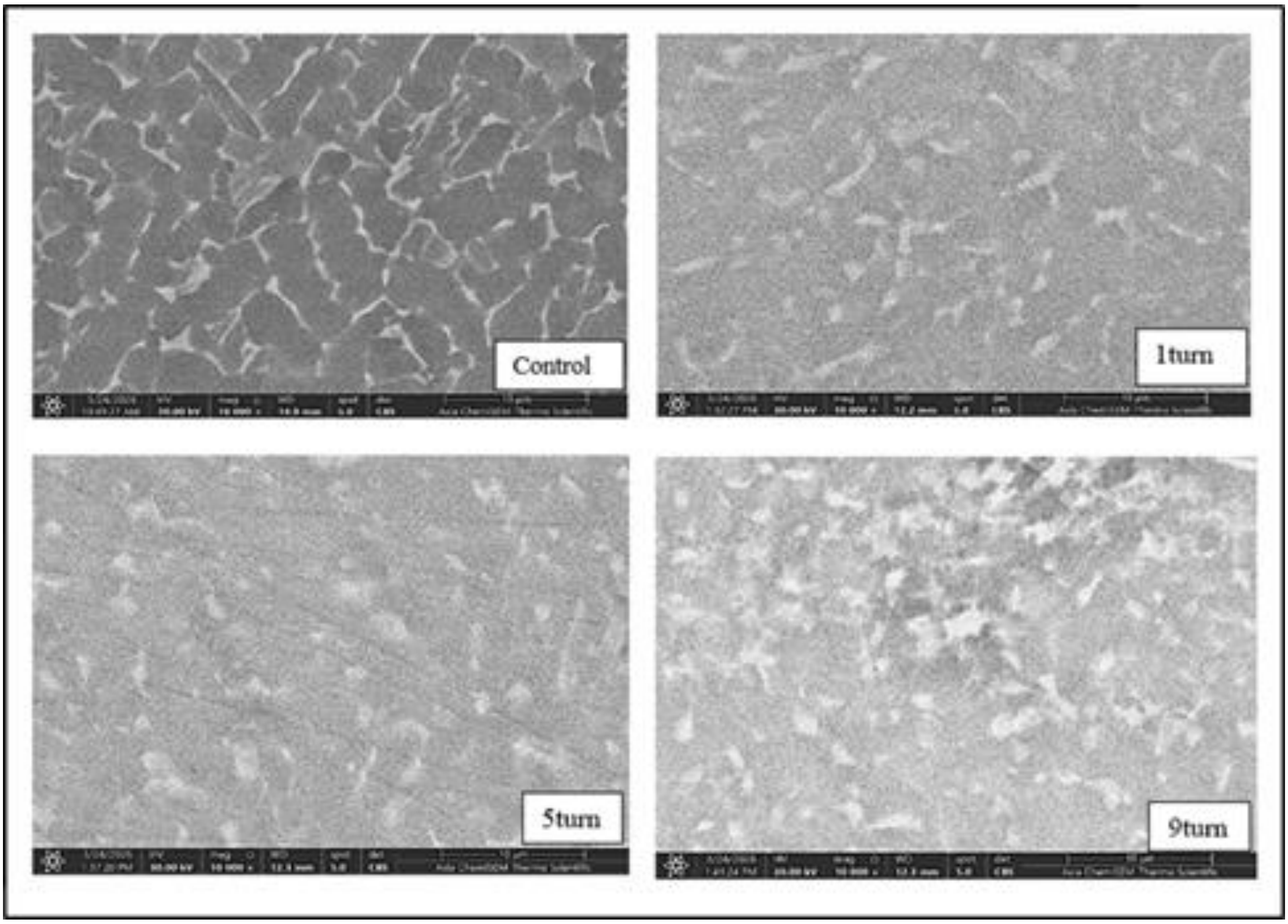

Figure 6. SEM micrographs showing the microstructural refinement of Ti-6Al-4V alloy after (a) 1 turn, (b) 5 turns, and (c) 9 turns of HPT processing.

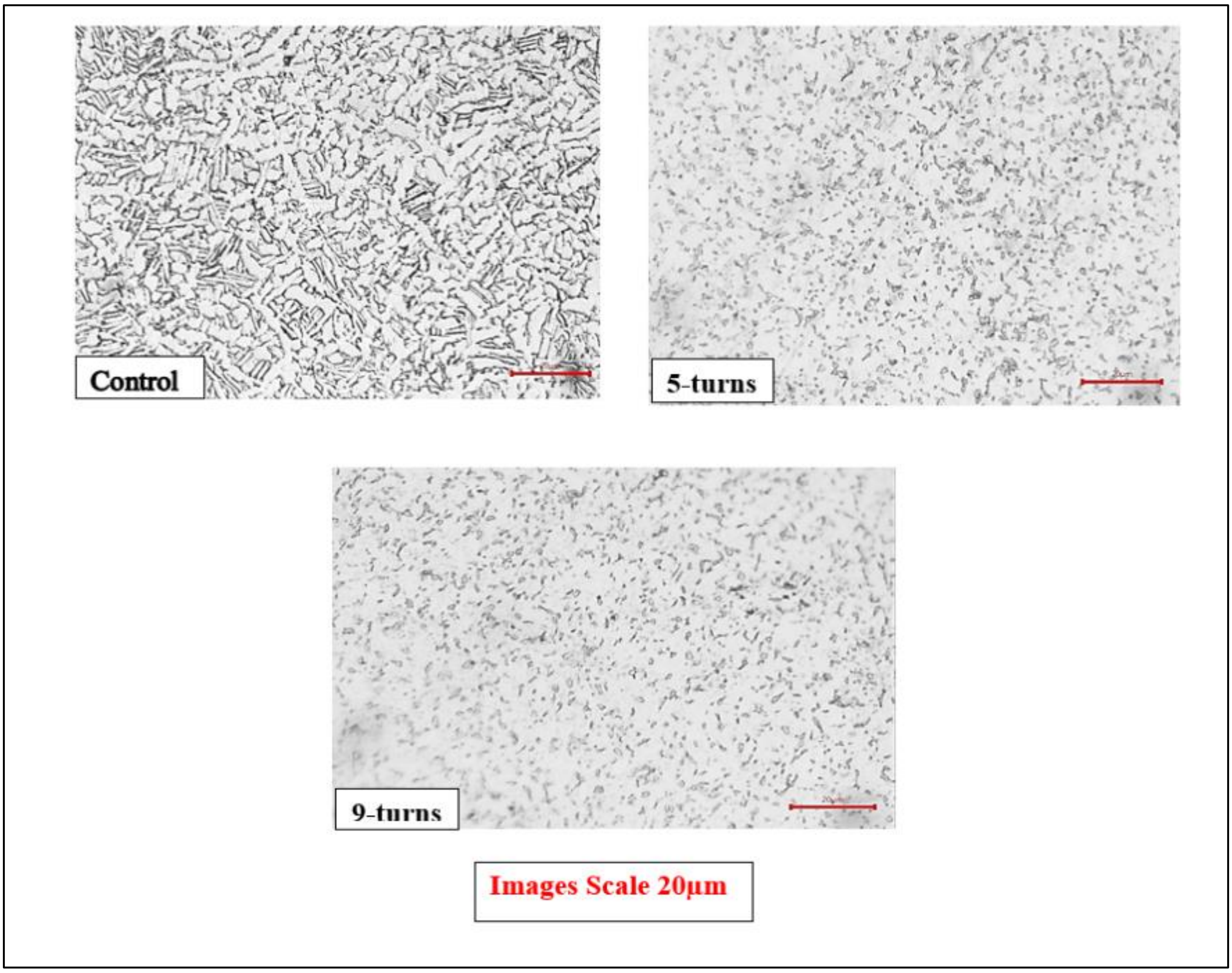

Figure 7. Optical micrographs of microstructures with scale 20μm

The Mises strain exerted on the disk during torsional straining may be approximated by the relationship established by HPT in-processing, as shown in equation (1)[21].

The Von Mises Strain values obtained in this work are representative of the plastic deformation experienced by the material after the High Pressure Torsion process. These values do not reflect a constant intrinsic property of the metal; instead, they depend on processing parameters such as N, the number of turns, r, the radius, and h, the sample thickness. Consequently, these values serve as an indication of the deformation intensity produced throughout the process rather than as an inherent physical attribute. In the HPT processing experiments, it was proposed that differences in hardness across HPT disks may be readily correlated throughout the disks by plotting the microhardness data against the corresponding strain, as shown in Table 2. Using this procedure, the Vickers hardness values were plotted as a function of distance from the disk center. At one turn, the structure seems heterogeneous, with relatively large grains exhibiting irregular boundaries. Distinct slide lines inside the grains signal that dislocation accumulation is beginning[21]. There may also be areas toward the center of the sample with less distortion. After five rotations, the original grains break down into smaller, more even grains. This shows that the structure has been much improved. The loss of most key grain features and the formation of a dense network of grain boundaries indicate that plastic deformation has increased and that the grains have transformed into a super-fine structure. The structure also becomes more homogeneous across the sample area. The pictures indicate that the nanostructure in the 9-turn section is almost uniform throughout.

And using the Tabor relation (equation (2))[22] to find the Equivalent Stress as shown in Table 3

$$\boldsymbol{\varepsilon} = \frac{2\pi N r}{h\sqrt{3}} \qquad (1)$$

$$\sigma = \frac{\text{Hardness MPa}}{3} = \text{MPa} \qquad (2)$$

Table 2. Calculated equivalent Von Mises strain values at the edge and center of the Ti-6Al-4V disks for different HPT turns.

NO. Turn	Microhardness HV	Equivalent Strain
Control	320	0
1	396.23	17.898
3	395.56	53.69
5	410.95	89.49
7	423.76	125.28
9	414.9	161.08

Table 3. Calculated Equivalent stress for different HPT turns.

NO. Turn	Microhardness HV	Equivalent Stress MPa
Control	320	1046
1	396.23	1295
3	395.56	1293
5	410.95	1343
7	423.76	1385
9	414.9	1356

Figure 8 illustrates the findings for the Ti64 alloy, showing that a theoretical model can be used to derive equivalent stress values from real hardness measurements obtained with the Vickers hardness equipment for the forming operations of high-performance alloys and metals, such as the Ti-6Al-4V alloy. All data points for each disk fit a plausible curve, indicating an initial increase in microhardness at the lowest equivalent stresses, followed by saturation of the microhardness readings at equivalent strains above 30. Moreover, the findings indicate that the markedly elevated microhardness values at 7 turns during saturation occur at an equivalent strain nearly equal to the saturation microhardness reading.

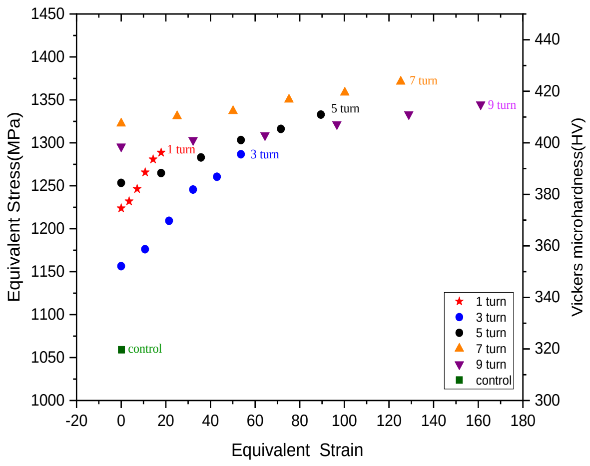

Figure 8. mechanical properties with different turn

4. Conclusion

This article demonstrates the importance of severe plastic deformation, particularly high-pressure torsion (HPT), in improving the mechanical properties of the Ti-6Al-4V alloy. This method was applied under pressure of 3 GPa at room temperature. Regarding the findings, the improvement in microhardness was notably 32.4%, achieved by increasing the number of HPT turns to 7 in steady state (saturation). This highlights the effectiveness of HPT in refining the microstructure and improving the mechanical properties of Ti-6Al-4V alloy. This suggests that microstructural refinement can enhance the mechanical properties essential for engineering applications, as shown by our findings, thereby facilitating future research on the biological dimension. These findings demonstrate that HPT is a powerful process for simultaneously enhancing microhardness and affecting mechanical properties, providing valuable insights into Ti alloys for engineering applications, and reinforcing the importance of continued investigation into severe plastic deformation methods.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationship that could have appeared to influence the work reported in this paper.

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Author Contributions

Hayder Masood- Writing – original draft, Methodology, Formal analysis, Data curation, Conceptualization, Anwer J. Al-Obaidi, Righdan Namus-Writing – review & editing, Validation, Supervision-Abd El-Nabi Z. El-Sayed-Resources,Conceptualization.

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