



Research article

Effect of ultrasonic nanocrystalline surface modification on hardness and elastic modulus of Ti-6Al-4V alloy

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Abstract: This study examined the effect of ultrasonic nanocrystalline surface modification (UNSM) on the mechanical properties of Ti-6Al-4V titanium alloy. Samples with dimensions of 80 × 10 × 5 mm were treated using varying amplitudes (20–40 μm), static loads (20–60 N), and processing temperatures up to 400 °C. The primary aim of this research was to identify the optimal processing parameters of UNSM to achieve superior mechanical properties and enhanced performance of the Ti-6Al-4V alloy. Systematic experiments were conducted by varying key parameters, such as ultrasonic amplitude, processing temperature, and applied static loads. The results revealed that the optimal UNSM parameters—30 μm amplitude, 400 °C processing temperature, and 40–60 N static load—significantly improved mechanical properties. Hardness increased from 394 (untreated) to 475 HV, while the elastic modulus reached 156 GPa, demonstrating substantial enhancements. Microstructural analysis confirmed that UNSM treatment promotes grain refinement, resulting in improved mechanical characteristics in the surface layer of the alloy. These findings highlight the potential of UNSM technology for applications requiring enhanced surface durability, strength, and wear resistance. This research provides valuable insights for industrial sectors, including aerospace, biomedical, and automotive industries, where high-performance materials are critical.

Keywords: Ti-6Al-4V alloy; hardness; ultrasonic nanocrystalline surface modification (UNSM); modulus of elasticity; microstructure

1. Introduction

Titanium alloys such as Ti-6Al-4V are widely used in various industries, including aerospace, biomedical, and automotive, due to their excellent mechanical properties, high corrosion resistance, and excellent strength-to-weight ratio. In recent years, intensive research has been conducted aimed at improving the mechanical and tribological characteristics of titanium alloys by modifying the surface or applying protective coatings. For example, Yang et al. [1] demonstrated that nitrogen-doped CoCrFeNiMn high-entropy alloy coatings significantly enhance the wear resistance and durability of Ti-6Al-4V alloy, highlighting the potential of advanced coating techniques in demanding applications. These findings emphasize the importance of surface engineering technologies for improving the performance of titanium alloys in industrial applications. A promising method aimed at achieving these goals is ultrasonic nanocrystalline surface modification (UNSM) [2]. UNSM is an innovative processing method based on the application of high-frequency ultrasonic waves to create a nanocrystalline structure on the surface of a material. This process involves exposure to ultrasonic waves with a frequency of 20–40 kHz, which causes intense plastic deformation of the metal surface. As a result, grains are reduced to nano sizes, and the dislocation density increases, which significantly improves the mechanical properties of the material. UNSM significantly changes the microstructure of the surface of titanium alloys. According to research, ultrasonic vibration leads to the formation of a nanocrystalline structure with a grain size of less than 100 nm. This is confirmed by the works of Gao et al. [3] and Kishore et al. [4], which demonstrated that reducing the grain size to the nanoscale improves mechanical properties by increasing grain boundaries, thereby restricting the movement of dislocations and enhancing the strength of the material. While the modulus of elasticity is a key parameter determining the stiffness of a material, it is primarily influenced by atomic bonding and microstructural changes, rather than dislocation motion. UNSM contributes to a significant increase in the modulus of elasticity of titanium alloys. For example, the study by Ye et al. [5] showed that after UNSM treatment, the modulus of elasticity of Ti-6Al-4V increased by 10%–15%. This is due to an increase in dislocation density and the formation of a nanocrystalline structure, which enhances resistance to plastic deformation. The increased dislocation density and the nanocrystalline structure formed after UNSM treatment significantly improved the wear resistance of titanium alloys. Studies by Amanov et al. [6] and Zha et al. [7] demonstrated that surface hardness increases, which enhances resistance to abrasive wear and reduces the likelihood of contact fatigue. This makes titanium alloys more durable and resistant to damage under operating conditions.

The fatigue strength of titanium alloys also increases significantly after UNSM treatment. Studies by Cao et al. [8] and Ye et al. [5] showed that after UNSM treatment, the fatigue strength of Ti-6Al-4V increased by 20%–30%, which is particularly important for the aerospace industry. The nanocrystalline structure contributes to a more uniform distribution of residual stress, reducing the likelihood of microcrack formation and propagation. The combination of a nanocrystalline structure, increased dislocation density, and a uniform distribution of residual stresses leads to an increase in the modulus of elasticity, improved wear resistance, and enhanced fatigue strength of the material. To achieve the best results from UNSM treatment, it is essential to optimize parameters such as the

amplitude and frequency of the ultrasonic waves, the tool feed rate, and the processing time. Recent advancements in machine learning have demonstrated significant potential in predicting and optimizing the mechanical properties of titanium alloys. For example, Han et al. [9] utilized machine learning algorithms to predict and optimize the mechanical properties of Ti-6Al-4V alloy processed through laser powder bed fusion, achieving improved precision and efficiency in parameter selection. These findings highlight the relevance of advanced computational techniques for enhancing material performance and guiding the development of new processing methods. However, experimental studies, such as UNSM, remain essential to validate and complement computational predictions, providing insights into the underlying mechanisms driving material improvements. Additionally, it is crucial to consider the interaction between these parameters to maximize the improvement of material properties. Developing optimal UNSM treatment parameters will allow for the full potential of titanium alloys to be realized in high-tech industries [8,10]. Given the above, the purpose of this study is to investigate the effect of UNSM at different temperatures on the mechanical properties of Ti-6Al-4V titanium alloy. This study is grounded in experimental evidence collected through systematic variations of UNSM treatment parameters. The findings are corroborated by microstructural analysis, hardness measurements, and elastic modulus evaluations, which provide direct support for the conclusions drawn. This work explores the mechanisms underlying the changes in the mechanical properties of Ti-6Al-4V under the influence of UNSM. Recent advancements in hybrid processing methods highlight the potential for combining multiple surface treatment techniques to enhance the mechanical properties of titanium alloys. For instance, the study by Li et al. [11] demonstrated the effect of an electro pulsing treatment combined with ultrasonic impact peening on the microstructure and mechanical properties of Ti-6Al-4V alloy, processed using a directed energy deposition method. This highlights the importance of ultrasonic-based methods in improving material performance. However, the effect of specific UNSM processing parameters on the mechanical properties of Ti-6Al-4V remains insufficiently studied, leaving a gap in understanding how to optimize these parameters for maximum performance. Although UNSM has been widely studied, the influence of key parameters such as amplitude, processing temperature, and static load on the mechanical properties of Ti-6Al-4V titanium alloy remains insufficiently explored. Additionally, there is limited understanding of the microstructural mechanisms underlying the observed improvements in mechanical properties. This research addresses these gaps by systematically analyzing how UNSM parameters affect the hardness and elastic modulus of the alloy, as well as its surface microstructure. The novelty of this work lies in identifying the optimal combination of UNSM parameters—amplitude, static load, and processing temperature—that lead to significant enhancements in material performance. Furthermore, this study provides valuable insights into the correlation between surface microstructure changes and improved mechanical properties.

The structure of the paper is as follows: the materials and methods used in the experiments are described first, followed by the presentation of the results, including mechanical property measurements and microstructural analysis. The findings are then discussed in the context of their potential applications, with a focus on how this research can contribute to the industrial adoption of UNSM technology in sectors such as aerospace, biomedical, and automotive industries.

2. Materials and methods

The surface treatment of titanium alloy samples was conducted using UNSM equipment. Figure 1 shows the schematic diagram of the UNSM equipment and the UNSM experimental setup. The

operation of the UNSM system is based on generating ultrasonic frequencies using a piezoelectric transducer. In this study, the experiments were performed at a fixed frequency of 20 kHz. Ultrasonic waves are transmitted to a tool in contact with the surface of the material being processed. The tool oscillates at a high frequency with a low amplitude. Upon contact with the material's surface, the ultrasonic waves create high-frequency impacts and friction, causing intense plastic deformation of the surface layer. As a result of this plastic deformation, the surface layer's structure is significantly refined, forming an ultrafine-grained structure. The static load is controlled via weights, while the ultrasonic generator produces waves at a specific frequency. These waves are transmitted through a converter and auxiliary equipment to an indenter with a tungsten carbide ball tip. Consequently, the indenter is subjected to a cumulative load, combining both a static component from the weights and a dynamic component from the ultrasonic vibrational energy.

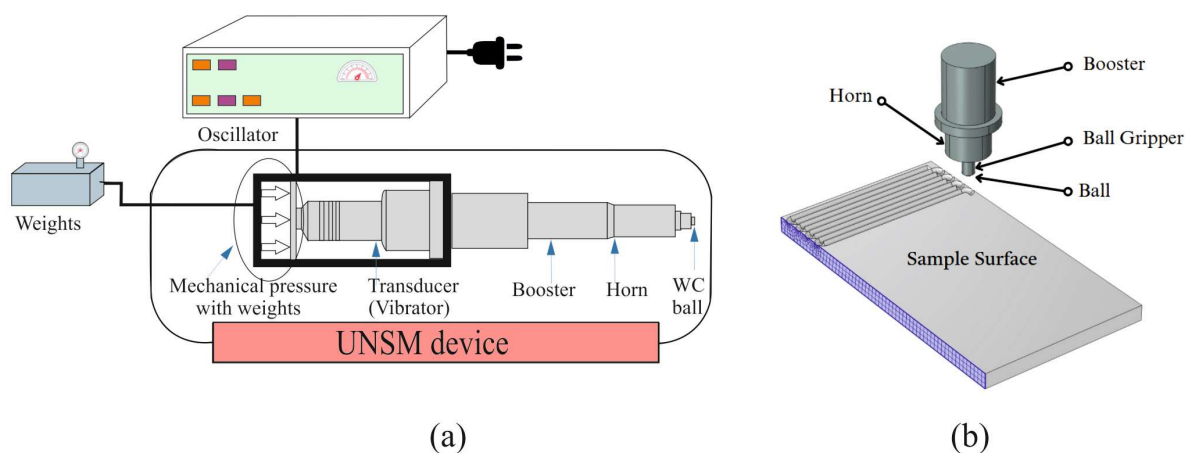


Figure 1. (a) Schematic diagram of the UNSM equipment and (b) the UNSM experimental setup.

Table 1 shows the UNSM treatment conditions and experimental parameters. The controlled variables were a 2.38 mm diameter tungsten carbide ball tip, a frequency of 20 kHz, a linear displacement pitch of 0.01 mm, and amplitudes of 20, 30, and 40 μm . These amplitudes were set and precisely controlled using the built-in software of the ultrasonic system. The amplitude was continuously monitored by integrated sensors within the ultrasonic generator to ensure stability and accuracy. Before each experiment, system parameters were calibrated to maintain the desired amplitudes during processing. The static load and scanning speed ranged from 20 to 60 N and 2000 mm/min or 33.3 mm/s, respectively.

Ti-6Al-4V titanium samples with dimensions of 80 mm in length, 10 mm in width, and 5 mm in height were used as the test material. UNSM was performed under the following temperature conditions: room temperature (RT), 350, and 400 $^{\circ}\text{C}$. Table 1 provides a summary of the experimental parameters used for the UNSM treatment of Ti-6Al-4V samples, including amplitude, static load, temperature, and processing speed. After the experiment, $1 \times 1 \text{ cm}^2$ samples were prepared for further research. The hardness of the samples was measured using a Fischerscope HM2000S (Fischerscope, Amsterdam, Netherlands), equipped with a Vickers indenter (a four-sided diamond pyramid with an angle of 136° between the faces). Measurements were conducted on the Vickers scale (HV), and each test was repeated five times to ensure accuracy. The phase composition was determined using an X'Pert Pro X-ray diffractometer (PANalytical, Amsterdam, Netherlands) with Cu-K α radiation, operating at a

voltage of 40 kV and a current of 30 mA. Scanning parameters were set at $35^\circ < 2\theta < 85^\circ$, with a step size of 0.02° and an exposure time of 5 s. For microstructure analysis, cross-sections of the samples were prepared. First, mechanical cutting was performed using silicon carbide cut-off wheels. Then, samples were ground and polished with diamond paste to obtain a smooth surface free from mechanical deformations. Chemical etching with Kroll's reagent, consisting of 100 mL of water, 1–3 mL of hydrofluoric acid, and 2–3 mL of nitric acid, was applied for 10 s. The microstructure was characterized using a TESCAN Vega scanning electron microscope (Tescan, Brno, Czech Republic).

Table 1. UNSM parameters used in this study.

No.	Amplitude (μm)	Static load (N)	Temperature ($^\circ\text{C}$)	UNSM speed (mm/s)
Ti 1	20	30	Room temperature	33.3
Ti 2	30	30		33.3
Ti 3	40	30		33.3
Ti 4	30	30	350	33.3
Ti 5	30	20	400	33.3
Ti 6	30	30	400	33.3
Ti 7	30	40	400	33.3
Ti 8	30	50	400	33.3
Ti 9	30	60	400	33.3
Ti 10	Untreated			

The key experimental parameters and the resulting responses are summarized in Table 2. The characterization methods included Vickers hardness testing, X-ray diffraction (XRD), and scanning electron microscopy (SEM).

Table 2. Input variables and output responses for UNSM treatment.

Input variables	Values/range	Output responses	Measured technique
Amplitude (μm)	20, 30, 40	Surface hardness (HV)	Vickers hardness test
Static load (N)	20, 30, 40, 50, 60	Elastic modulus (GPa)	Fischerscope HM2000S
Processing temperature ($^\circ\text{C}$)	RT, 350, 400	Grain refinement	SEM (TESCAN Vega)
Ultrasonic vibration (kHz)	20–40	Dislocation density	XRD
Tool material	Tungsten carbide	Phase composition (α and β phases)	XRD
Scanning speed (mm/s)	33.3	Microstructural changes	SEM (TESCAN Vega)
Sample dimensions (mm)	$80 \times 10 \times 5$	Wear resistance	Wear testing

3. Results and discussion

3.1. Hardness

One of the main parameters of UNSM is the amplitude. At low amplitudes, moderate plastic deformation of the surface occurs, leading to smoothing of the surface and a slight increase in hardness

due to the formation of submicron structures. Increasing the amplitude enhances plastic deformation, increases the depth of the modified layer, improves the surface structure, and significantly increases hardness by raising dislocation density and reducing grain size [12]. At high amplitudes, maximum surface hardness is achieved due to the formation of nanocrystalline structures as a result of intense plastic deformation of the surface layer. The results of this study demonstrate the significant impact of processing parameters on the mechanical properties of the Ti-6Al-4V alloy treated with UNSM. Processing parameters play a pivotal role in the effectiveness of UNSM in improving the mechanical properties of Ti-6Al-4V alloy. Parameters such as amplitude, static load, and processing temperature directly influence the material's microstructural changes and resulting hardness. Below, the influence of each parameter is discussed in detail, highlighting the optimal conditions for achieving maximum hardness without compromising the material's integrity.

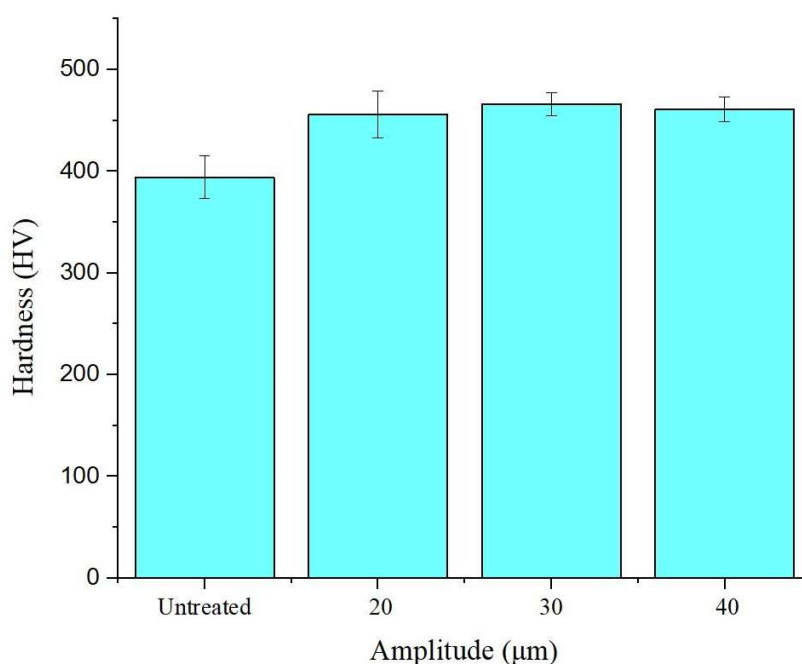


Figure 2. Hardness change of Ti-6Al-4V alloy samples at three different amplitudes (20, 30, 40 μm).

Figure 2 presents the hardness changes of Ti-6Al-4V alloy samples treated with UNSM under different amplitudes (20, 30, and 40 μm). The maximum hardness of the Ti-6Al-4V sample found at an amplitude of 30 μm is due to the fact that this amplitude provides the optimal balance between the depth of plastic deformation and the structural stability of the material. At 30 μm , conditions are created where significant grain refinement and an increase in dislocation density occur, leading to material strengthening. Lower amplitudes may not provide sufficient deformation for significant strengthening, while higher amplitudes, on the contrary, may cause excessive surface deformation, resulting in defects such as microcracks or overheating. This, in turn, reduces the material's hardness. Therefore, an amplitude of 30 μm is optimal, as it allows for maximum hardness without risking structural damage. Figure 3 shows the results of surface hardness measurements for samples treated at various temperatures (RT, 350, 400 $^{\circ}\text{C}$). The hardness measurements reveal an increase in hardness after processing at different temperatures, ranging from RT to 400 $^{\circ}\text{C}$. The surface hardness of the control sample was 394 ± 10 HV. UNSM treatment at RT resulted in an increase in hardness

to 465.96 ± 11 HV. However, when the temperature increased to $350\text{ }^{\circ}\text{C}$, the hardness decreased slightly to 462.58 ± 10 HV compared to the RT treatment. The reduction in hardness of the titanium alloy at $350\text{ }^{\circ}\text{C}$ may be due to the onset of recrystallization and softening of the material. At this temperature, partial restoration of deformations accumulated during previous processing is likely, which reduces crystal lattice defects and consequently lowers hardness. The maximum hardness value of 475.08 ± 10 HV was achieved after treatment at $400\text{ }^{\circ}\text{C}$.

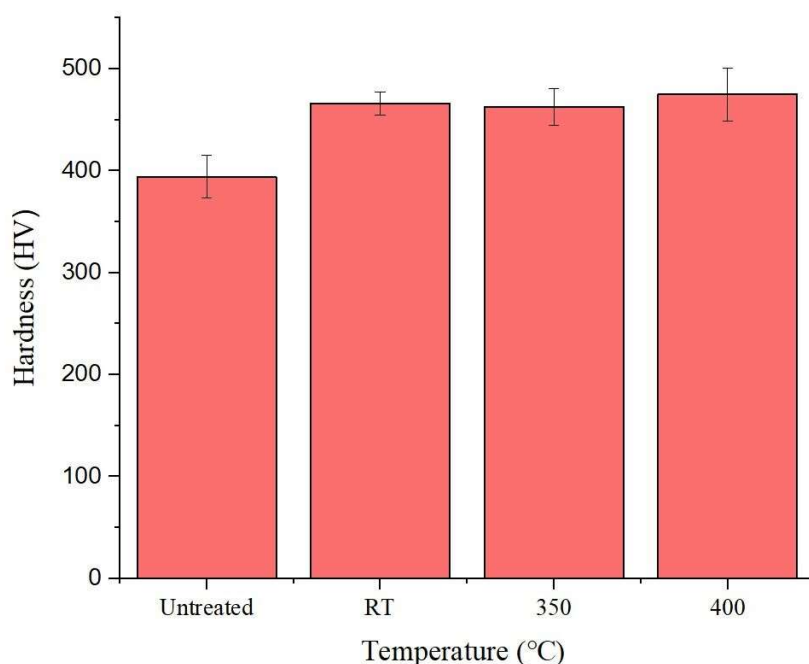


Figure 3. Hardness change of Ti-6Al-4V alloy samples at three different temperatures (room temperature, 350, 400 $^{\circ}\text{C}$).

At $350\text{ }^{\circ}\text{C}$, partial recovery of the deformations induced during prior processing occurs, leading to fewer crystal lattice defects and a corresponding reduction in hardness. The variations in hardness between these temperatures are relatively minor, likely due to grain growth at $350\text{ }^{\circ}\text{C}$, which slightly reduces the material's strength. However, when the temperature rises to $400\text{ }^{\circ}\text{C}$, another process occurs that increases the hardness of the material.

The increase in hardness due to the UNSM treatment can be explained by several factors. One of them is grain refinement in the surface layer of the material, which contributes to an increase in hardness [13]. Additionally, during UNSM treatment, residual compressive stress is generated in the surface layer, further enhancing hardness. At elevated temperatures, such as $400\text{ }^{\circ}\text{C}$, dislocation mobility increases, making grain refinement more efficient [14], which leads to higher hardness values. Furthermore, at high temperatures and high deformations, titanium alloys can undergo dynamic recrystallization, where new grains form during deformation. This results in grain refinement and improved mechanical properties [15]. The improvement in surface hardness during UNSM treatment, especially at a temperature of $400\text{ }^{\circ}\text{C}$, indicates the high efficiency of this method in enhancing the wear resistance and mechanical properties of Ti-6Al-4V alloy components. The observed hardness increase after UNSM treatment is directly supported by data presented in Figures 2 and 3, where the treated samples show statistically significant improvements compared to the untreated ones. For example, at an amplitude of $30\text{ }\mu\text{m}$ and a load of 50 N , the hardness increases by approximately 30%

compared to the untreated sample. These changes are attributed to the grain refinement and increased dislocation density caused by the treatment. Another key parameter in the UNSM process that significantly affects surface hardness and other mechanical properties of materials is the load. Figure 4 shows the results of surface hardness measurements for samples treated with the UNSM method under various loads (10, 20, 30, 40, 50, and 60 N). Data indicate that as the load increases, the surface hardness of the samples also increases.

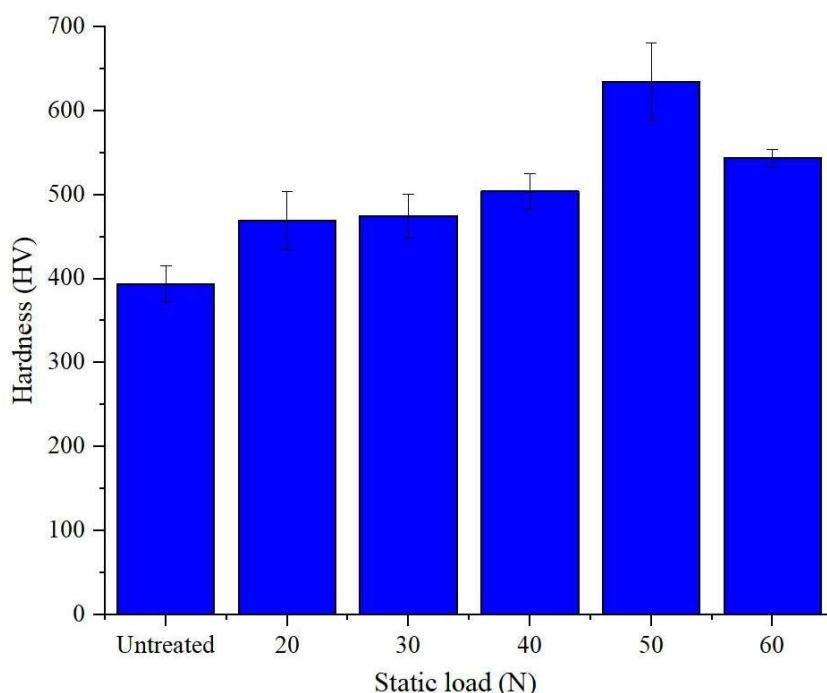


Figure 4. Hardness change of Ti6-Al-4V alloy samples under five different loads (20, 30, 40, 50, 60 N).

When the load increased to 20, 30, and 40 N, hardness increased to 469.42 ± 10 , 475.08 ± 10 , and 503.92 ± 11 HV, respectively. With a further increase in load to 50 N, there was a significant increase in hardness to 634.58 ± 11 HV, indicating a substantial improvement in the material's mechanical properties. However, at a load of 60 N, the hardness slightly decreased to 543.56 ± 11 HV compared to 50 N.

Increasing the load contributes to more intense plastic deformation and grain refinement on the surface of the material. This leads to the formation of an ultrafine granular structure, which increases the hardness [16]. However, excessive load can cause defects such as microcracks or excessive stresses, which reduce the material's mechanical properties. At high loads and temperatures, dynamic recrystallization may occur, promoting grain growth and consequently reducing hardness and strength. According to the Hall–Petch model, a decrease in grain size leads to an increase in the density of grain boundaries, which hinders the movement of dislocations, thus increasing the strength of the material [17]. As shown in Figure 5, as the grain size decreases, the yield strength increases, indicating an improvement in the hardness and strength of the material.

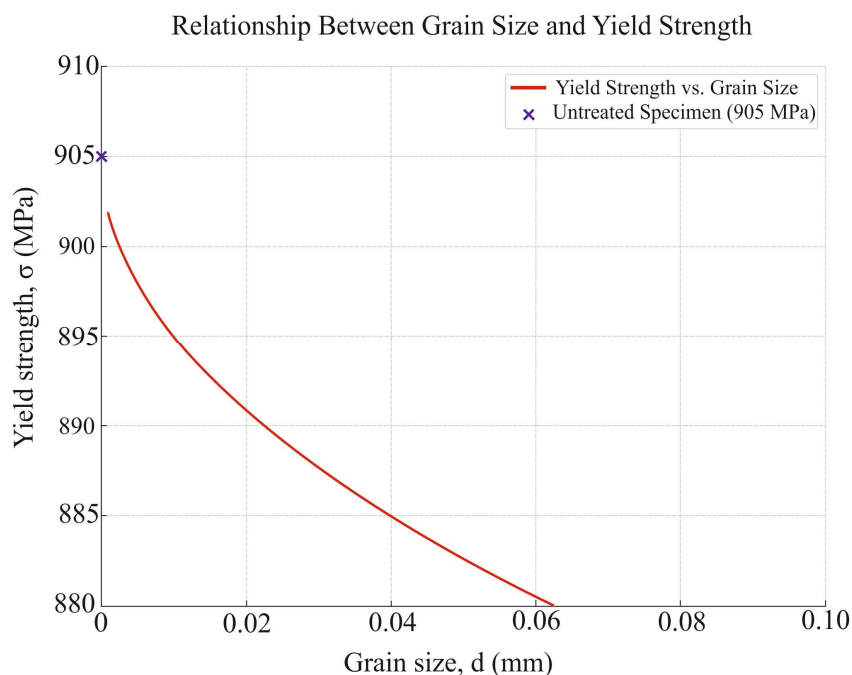


Figure 5. Dependence of the yield strength of Ti-6Al-4V alloy on the grain size according to the Hall–Petch model (Reproduced from Ref. [17] with permission).

Ti-6Al-4V alloy is characterized by high strength and hardness, which can be further enhanced by reducing the grain size. This effect is particularly important for applications where high wear resistance and strength are required, such as in the aerospace and medical industries. Optimizing the processing method, such as using UNSM, can focus on grain size reduction, thereby increasing the material's strength without altering its chemical composition. The results obtained demonstrate that increasing the load when applying UNSM technology positively affects the surface hardness of the samples.

3.2. Modulus of elasticity

The modulus of elasticity, or Young's modulus, plays a crucial role in evaluating the elastic properties of materials. It is a key indicator that helps predict the behavior of a material under mechanical loads. UNSM is an effective method for increasing the modulus of elasticity and other mechanical properties of titanium alloys such as Ti-6Al-4V. The elastic modulus was determined using a Fischerscope HM2000S instrument, which calculates the modulus based on the depth of penetration of the Vickers indenter and the applied load. The modulus of elasticity of titanium and its alloys depends on the specific composition of the alloy. For pure titanium (grade 2), the modulus of elasticity is typically around 105 GPa. In the case of the Ti-6Al-4V alloy (grade 5), the modulus of elasticity can range between 110 and 120 GPa [18,19]. In this study, the modulus of elasticity for Ti-6Al-4V alloy samples subjected to UNSM under various amplitude, static load, and temperature values was obtained. As shown in Figure 6, the modulus of elasticity varied depending on the processing parameters. The maximum modulus value was observed for the Ti9 sample (156 ± 2 GPa), treated at an amplitude of 30 μm , a static load of 50 N, and a temperature of 400 $^{\circ}\text{C}$. UNSM is a promising method for

modifying the surfaces of metallic materials, significantly improving their mechanical properties, including hardness and the modulus of elasticity. The presented data confirms the effectiveness of using this technology to control the mechanical properties of the Ti-6Al-4V alloy. Particular attention should be given to the fact that changes in processing temperature, from room temperature to 400 °C, combined with variations in amplitude and static load, have a significant effect on the modulus of elasticity. This may be due to alterations in the alloy's microstructure caused by temperature changes, as well as the characteristics of the nanocrystalline structure formed under various processing conditions.

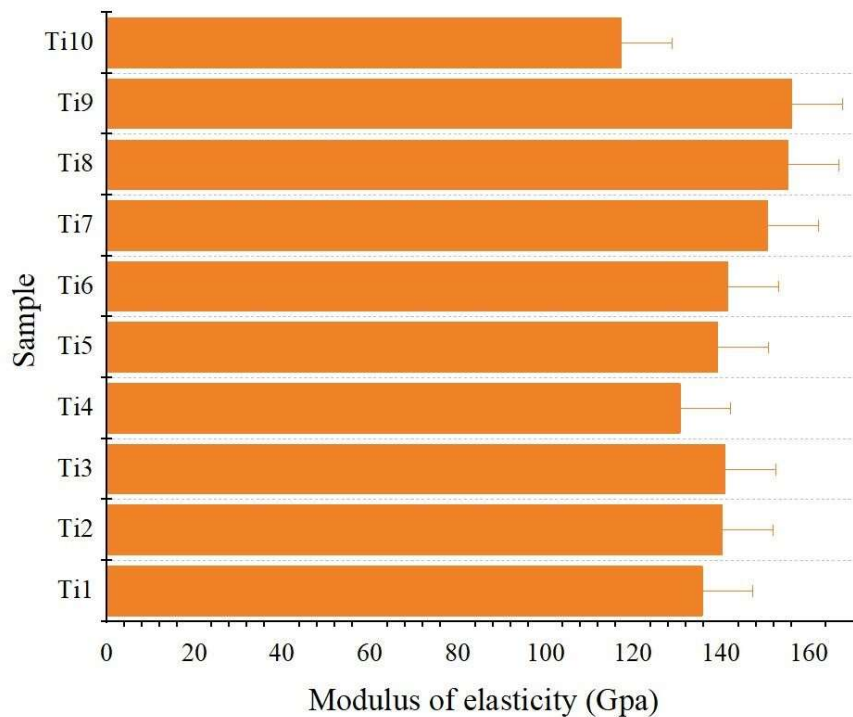


Figure 6. Modulus of elasticity of Ti-6Al-4V alloy samples under various processing parameters of UNSM.

UNSM can enhance the modulus of elasticity by modifying the microstructure, particularly through the formation of a nanocrystalline structure. While dislocation density primarily correlates with the strength of metals by impeding dislocation motion and increasing resistance to plastic deformation, the increase in the modulus of elasticity observed in this study is likely a result of the nanocrystalline structure and other microstructural changes induced by UNSM. These findings are consistent with studies by Amanov et al. [6] and Cao et al. [8], which highlight the role of grain refinement and structural modifications in improving mechanical properties.

Thus, the role of increased dislocation density is to create internal barriers to defect (dislocation) movement within the crystal lattice, preventing deformation and making the material more resistant to mechanical loads. As noted by Lütjering G et al. [19], in combination with a nanocrystalline structure, which also contributes to improved mechanical properties, this effect can significantly raise the modulus of elasticity of the treated material.

Optimization of UNSM treatment parameters allows for a substantial increase in the modulus of elasticity, which in turn enhances the durability and reliability of Ti-6Al-4V alloy products. The enhancement in the elastic modulus, as shown in Figure 6, can be attributed to the reduction in grain

size and the formation of a nanocrystalline structure in the treated surface layer. These microstructural changes primarily influence the modulus of elasticity, which determines the material's stiffness. While resistance to deformation at the elastic stage is governed by the modulus of elasticity, improvements in strength result from the restriction of dislocation motion caused by grain boundary refinement.

3.3. The effect of UNSM on the phase composition

XRD analysis was performed to identify phase composition changes and confirm microstructural modifications induced by the UNSM treatment. Due to equipment limitations, quantitative calculations such as grain size estimation were not performed. Figure 7 shows the X-ray diffraction patterns of the Ti-6Al-4V alloy at various UNSM treatment parameters. The spectra are compared for the initial sample and samples T2, T4, T5, T6, T7, T8, and T9. The X-ray diffractograms for each sample display peaks corresponding to the α -phase (hexagonal close-packed structure) and the β -phase (body-centered cubic structure). After heat treatment of the samples (under different modes and temperatures), the X-ray diffraction results reveal changes in the intensity of the α and β phase peaks. Heat treatment at lower temperatures promoted an increase in the proportion of the α -phase, as evidenced by the higher intensity of the peaks. This suggests the stability of the hexagonal phase under these specific conditions. This is confirmed by the increased intensity of the peaks, indicating the stability of the hexagonal phase under these conditions. After processing, samples showed a higher proportion of the α -phase, which correlates with increased strength, while a sufficient amount of the β -phase remains to ensure plasticity. The X-ray diffraction patterns of samples Ti2 and Ti7 show clear peaks corresponding to the α and β phases of titanium, as well as titanium oxide (TiO_2). For all treated samples, changes in the intensity and shape of the peaks were observed. For example, the alpha phase exhibited a peak with an interplanar spacing of $d = 1.726 \text{ \AA}$, while the beta phase had a peak with a spacing of $d = 1.641 \text{ \AA}$. These differences allow us to confidently state that the increase in peak intensity is related to changes in the content of the alpha and beta phases in the alloy after treatment. During processing by the UNSM method, plastic deformation of the material occurs, which leads to grain refinement and improved ordering of the crystal structure. This increases the intensity of X-ray diffraction peaks, as the fine-grained structure diffracts X-rays more effectively. Additionally, the number of defects such as dislocations and vacancies may decrease during processing, further contributing to an increase in peak intensity. Reducing defects improves the ordering of atoms, which increases the clarity and intensity of X-ray peaks. Thus, the increase in peak intensity is associated with the improvement in crystal structure ordering, the reduction of defects, and possible phase changes caused by UNSM machining. For the Ti6, Ti7, Ti8, and Ti9 samples, treated at higher parameters (elevated temperature and load), signs of changes in the intensity of titanium oxide peaks were observed. This may be due to the formation of nanocrystalline structures or changes in phase distribution. An increase in peak intensity in Ti6 and Ti7 samples indicates a possible improvement in crystal structure and an increase in the proportion of the alpha phase of titanium after treatment. This suggests a positive effect of UNSM on the mechanical properties of the alloy, such as hardness and strength. The most intense peak indicates maximum ordering in this direction. In alpha titanium, titanium atoms are located in the Wyckoff position 2c, indicating a specific symmetry in the structure [20]. This means that titanium atoms occupy specific points in the hexagonal cell, corresponding to space group P 63/mmc. Symmetry analysis shows that alpha titanium has a highly symmetrical hexagonal structure, typical of many metals, due to the dense packing of atoms in hexagonal crystals. X-ray diffraction of alpha titanium

confirms its hexagonal structure with highly symmetrical packing of titanium atoms. The X-ray diffraction data show high peak intensity, indicating good crystallinity in the sample. After UNSM treatment, X-ray analysis showed moderate peak broadening (an increase in FWHM), which indicates a reduction in grain size and an increase in dislocation density. A shift of peaks toward larger angles was also observed, which may be due to the compaction of the material and the accumulation of internal stresses caused by plastic deformation, as indicated in. The presented X-ray diffraction patterns demonstrate the effect of UNSM parameters on the microstructure of the Ti-6Al-4V alloy.

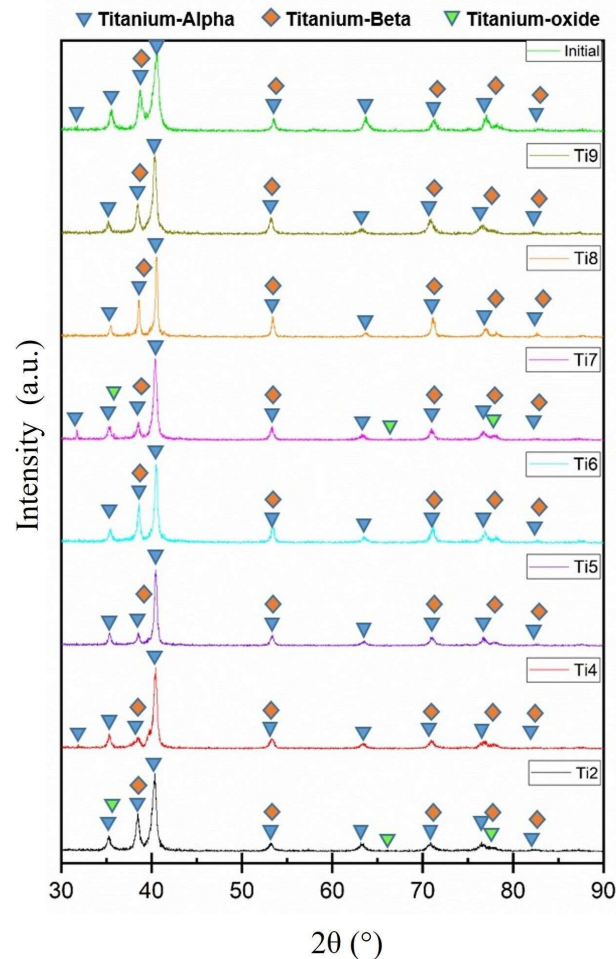


Figure 7. X-ray images of the Ti-6Al-4V alloy under various UNSM treatment parameters.

These changes, including peak shifts, intensity variations, and possible phase transformations, can be used to further optimize the machining process and enhance the mechanical properties of the alloy. Based on the X-ray diffraction results, it can be concluded that heat treatment and UNSM treatment achieved an optimal ratio of α and β phases, leading to an improvement in the mechanical properties of the material. The XRD results showed significant changes in peak intensity and slight shifts, indicating the presence of residual stress and alterations in the crystal structure. These findings complement SEM observations and mechanical property measurements, demonstrating the effectiveness of UNSM treatment.

Figure 8 presents the depth and width of the hardened zone for various UNSM processing parameters. It can be observed that increasing amplitude, static load, and processing temperature leads

to larger dimensions of the hardened zone. The maximum dimensions (depth: 450 μm , width: 300 μm) were achieved for sample Ti9 under 30 μm amplitude, 60 N load, and 400 $^{\circ}\text{C}$ processing temperature. These results confirm that intensive processing conditions promote deeper plastic deformation of the material.

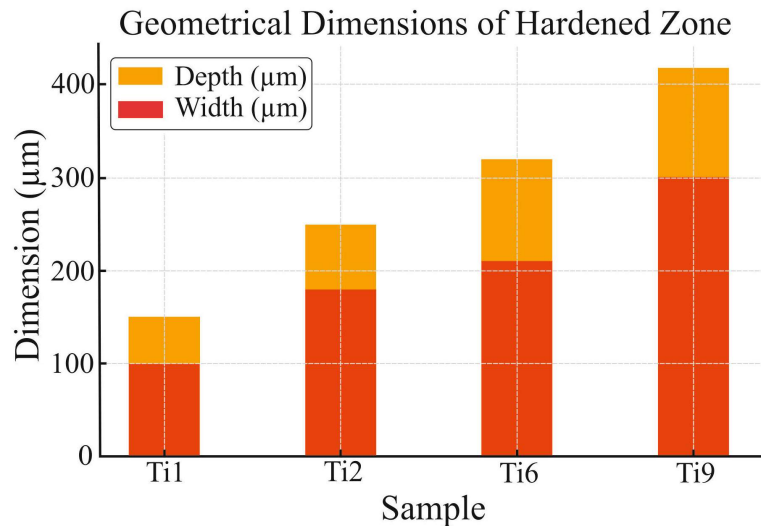


Figure 8. Variation in the depth and width of the hardened zone under different UNSM processing parameters.

3.4. The effect of UNSM on microstructure

Changes in the microstructure of the material caused by thermal and mechanical treatment play a key role in improving the mechanical properties of metals. In this context, microstructural analysis was performed to identify structural changes after processing. Depending on the processing parameters, such as amplitude, ultrasonic vibration, and applied load, significant differences in the surface structure could be observed. Figure 9 shows SEM images of the Ti-6Al-4V alloy surface obtained using various UNSM parameters: (a) initial structure without treatment, (b) treated at room temperature with an amplitude of 20 μm and a static load of 30 N, (c) treated at 400 $^{\circ}\text{C}$ with an amplitude of 30 μm and a static load of 40 N, and (d) treated at 400 $^{\circ}\text{C}$ with an amplitude of 30 μm and a static load of 50 N. These images illustrate the progressive grain refinement and microstructural changes induced by the UNSM process under different treatment conditions. At room temperature with an amplitude of 20 μm and a load of 30 N (Figure 9b), significant grain refinement is observed compared to the initial structure (Figure 9a). This suggests that even with moderate processing parameters, UNSM induces substantial changes in the microstructure, increasing the density of grain boundaries, and potentially enhancing the hardness of the material. At an elevated temperature of 400 $^{\circ}\text{C}$, with an amplitude of 30 μm and a load of 40 N (Figure 9c), further grain refinement is evident. This behavior indicates that an increase in temperature activates recrystallization processes and accelerates plastic deformation, leading to more pronounced structural changes. These findings align with studies showing that at elevated temperatures, the grain refinement process is more intensive due to increased diffusion and recrystallization. At the same temperature, but with a higher load of 50 N (Figure 9d), the surface structure becomes even denser, indicating extreme microstructural changes. However, an excessive increase in load can result in the formation of microcracks and other

defects, which reduce the material's overall strength. This is corroborated by Cao et al. [8], who showed that excessive plastic deformation during ultrasonic processing can lead to the formation of defects, diminishing the material's overall mechanical properties despite a reduction in grain size. No cracks or surface defects were observed in the UNSM-treated samples under the investigated parameters. However, previous studies have shown that extreme processing conditions or specific material characteristics can lead to crack initiation and propagation [21,22]. The absence of cracks in this study confirms that the applied parameters were optimized to avoid such defects.

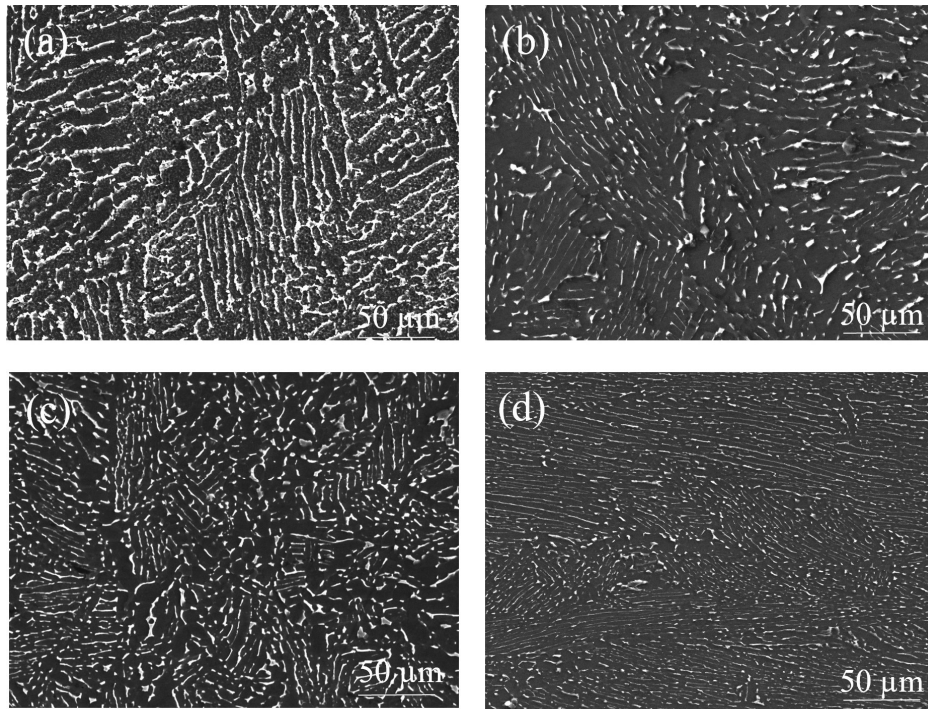


Figure 9. SEM images of the surface of the Ti-6Al-4V alloy under various UNSM treatment parameters.

4. Conclusions

The results of this study confirm the significant influence of UNSM parameters on the microstructure and mechanical properties of the titanium alloy Ti-6Al-4V. The optimal combination of ultrasonic amplitude, processing temperature, and static load leads to a notable increase in both hardness and modulus of elasticity in the alloy. This effect is achieved through grain refinement and an increase in dislocation density in the material's surface layer. Processing with the UNSM method results in a significant increase in the hardness of the Ti-6Al-4V alloy, achieving values greater than 466 HV under optimal parameters. The modulus of elasticity is significantly enhanced due to the formation of a nanocrystalline structure, contributing to improved stiffness and durability of the material. Microstructural analysis reveals that UNSM treatment promotes grain refinement, explaining the improvements in the alloy's mechanical properties. Microstructural analysis shows that the UNSM treatment leads to grain refinement, which explains the improvement in the mechanical properties of the alloy. Optimal processing parameters, including an amplitude of ultrasonic vibration of 30 μm , a

processing temperature of 400 °C, and a static load of 40–50 N, provide the best results. UNSM technology has shown its effectiveness in improving the mechanical properties of titanium alloys, especially in terms of hardness and modulus of elasticity. However, in order to achieve optimal results, it is necessary to carefully control the processing parameters, avoiding excessive loads that can lead to defects. These results open up new possibilities for the application of UNSM in aerospace, medical, and other industries where high mechanical properties of materials are important. These findings highlight the significance of the study and demonstrate the potential of UNSM to improve the performance of titanium alloys.

Use of AI tools declaration

The authors declare they have not used Artificial Intelligence (AI) tools in the creation of this article.

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Author contributions

Auezhan Amanov and Bauyrzhan Rakhadilov are involved in conceptualization, methodology and formal analysis. Zarina Aringozhina, Nurtoleu Magazov, Gulzhaz Uazyrkhanova are involved in review and editing and discussion. All authors have read and agreed to the published version of the manuscript.

Conflict of interest

The authors declare no conflict of interest.

References

1. Yang ML, Xu JL, Huang J, et al. (2024) Wear resistance of N-Doped CoCrFeNiMn high entropy alloy coating on the Ti-6Al-4V alloy. *J Therm Spray Tech* 33: 2408–2418. <https://doi.org/10.1007/s11666-024-01864-7>
2. Magazov N, Satbaeva Z, Rakhadilov B, et al. (2023) A study on surface hardening and wear resistance of AISI 52100 steel by ultrasonic nanocrystal surface modification and electrolytic plasma surface modification technologies. *Materials* 16: 6824. <https://doi.org/10.3390/ma16206824>
3. Gao JB, Ben DD, Yang HJ, et al. (2021) Effects of electropulsing on the microstructure and microhardness of a selective laser melted Ti6Al4V alloy. *J Alloys Compd* 875: 160044. <https://doi.org/10.1016/j.jallcom.2021.160044>
4. Kishore A, John M, Ralls AM, et al. (2022) Ultrasonic nanocrystal surface modification: Processes, characterization, properties, and applications. *Nanomaterials* 12: 1415. <https://doi.org/10.3390/nano12091415>

5. Ye Y, Wang H, Tang G, et al. (2018) Effect of electropulsing-assisted ultrasonic nanocrystalline surface modification on the surface mechanical properties and microstructure of Ti-6Al-4V alloy. *J Mater Eng Perform* 27: 2394–2403. <https://doi.org/10.1007/s11665-018-3248-3>
6. Amanov A, Urmanov B, Amanov T, et al. (2017) Strengthening of Ti-6Al-4V alloy by high temperature ultrasonic nanocrystal surface modification technique. *Mater Lett* 196: 198–201. <https://doi.org/10.1016/j.matlet.2017.03.059>
7. Zha X, Yuan Z, Qin H, et al. (2024) Investigating the dynamic mechanical properties and strengthening mechanisms of Ti-6Al-4V alloy by using the ultrasonic surface rolling process. *Materials* 17: 1382. <https://doi.org/10.3390/ma17061382>
8. Cao X, Xu L, Xu X, et al. (2018) Fatigue fracture characteristics of Ti6Al4V subjected to ultrasonic nanocrystal surface modification. *Metals* 8: 77. <https://doi.org/10.3390/met8010077>
9. Han C, Yan F, Yuan D, et al. (2024) Machine learning enabling prediction in mechanical performance of Ti6Al4V fabricated by large-scale laser powder bed fusion via a stacking model. *Front Mech Eng* 19: 25. <https://doi.org/10.1007/s11465-024-0796-0>
10. Ao N, Liu D, Xu X, et al. (2019) Gradient nanostructure evolution and phase transformation of α phase in Ti-6Al-4V alloy induced by ultrasonic surface rolling process. *Mater Sci Eng A* 742: 820–834. <https://doi.org/10.1016/j.msea.2018.10.098>
11. Li G, Xiao X, Zhang W, et al. (2023) Effect of electropulsing-assisted ultrasonic strengthening on fatigue properties of HIP Ti-6Al-4V alloy. *Metall Mater Trans A* 54: 3912–3927. <https://doi.org/10.1007/s11661-023-07142-5>
12. Yuan D, Shao S, Guo C, et al. (2021) Grain refining of Ti-6Al-4V alloy fabricated by laser and wire additive manufacturing assisted with ultrasonic vibration. *Ultrason Sonochem* 73: 105472. <https://doi.org/10.1016/j.ultsonch.2021.105472>
13. Ghamarian I, Samimi P, Telang A, et al. (2017) Characterization of the near-surface nanocrystalline microstructure of ultrasonically treated Ti-6Al-4V using ASTARTM/precession electron diffraction technique. *Mater Sci Eng A* 688: 524–531. <https://doi.org/10.1016/j.msea.2017.02.029>
14. Zhang H, Zhang S, Zhang S, et al. (2023) High temperature deformation behavior of near- β titanium alloy Ti-3Al-6Cr-5V-5Mo at $\alpha + \beta$ and β phase fields. *Crystals* 13: 371. <https://doi.org/10.3390/cryst13030371>
15. Luo Y, Heng Y, Wang Y, et al. (2014) Dynamic recrystallization behavior of TA15 titanium alloy under isothermal compression during hot deformation. *Adv Mater Sci Eng* 2014: 1–9. <https://doi.org/10.1155/2014/413143>
16. Rakhadilov B, Satbayeva Z, Ramankulov S, et al. (2021) Change of 0.34Cr-1Ni-Mo-Fe steel dislocation structure in plasma electrolyte hardening. *Materials* 14: 1928. <https://doi.org/10.3390/ma14081928>
17. Hansen N (2004) Hall–Petch relation and boundary strengthening. *Scr Mater* 51: 801–806. <https://doi.org/10.1016/j.scriptamat.2004.06.002>
18. Callister WD (2006) *Materials Science and Engineering: An Introduction*, New Delhi: Wiley India.
19. Lütjering G, Williams JC (2007) Titanium based intermetallics, In: Lütjering G, Williams JC, *Titanium. Engineering Materials, Processes*, 2 Eds., Heidelberg, Springer Berlin, 337–366. https://doi.org/10.1007/978-3-540-73036-1_8

20. Williams JC, Baggerly RG, Paton NE (2002) Deformation behavior of HCP Ti-Al alloy single crystals. *Metall Mater Trans A* 33: 837–850. <https://doi.org/10.1007/s11661-002-0153-y>
21. Hu Q, Liu W, Song Y, et al. (2024) Influence of solid solution treatment on fatigue crack propagation behavior in the thickness direction of 2519A aluminum alloy thick plates. *Eng Fract Mech* 302: 110069. <https://doi.org/10.1016/j.engfracmech.2024.110069>
22. Huang M, Wang L, Wang C, et al. (2024) Optimizing crack initiation energy in austenitic steel via controlled martensitic transformation. *J Mater Sci Technol* 198: 231–242. <https://doi.org/10.1016/j.jmst.2024.02.019>



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