ORIGINAL STUDY

STUDY OF THE EFFECT OF PRELIMINARY ULTRASONIC SURFACE MODIFICATION OF TI-6AL-4V ALLOY SUBSTRATE ON THE FORMATION OF A NITRIDED LAYER

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Abstract. *This study examined how ultrasonic nanocrystalline surface modification (UNSM) influences the formation of nitride layers in Ti-6Al-4V alloy during ion-plasma nitriding (IPN). We systematically varied UNSM parameters-specifically vibration amplitude, static load, and processing temperature-to assess their impact on the material's microstructure, hardness, elastic modulus, and tribological behavior. Our findings indicate that an optimized UNSM pre-treatment significantly boosts nitrogen diffusion, resulting in the creation of dense and uniform TiN/*Ti_2N *layers. Notably, samples subjected to UNSM under high-load and elevated-temperature conditions showed the most substantial improvements. These included a surface hardness increase of up to 25%, an elastic modulus rise of up to 18%, and enhanced wear resistance with a more stable and reduced friction coefficient (around 0.55). Further scanning electron microscopy (SEM) and X-ray diffraction (XRD) analyses corroborated these results by confirming microstructural densification, grain refinement, and a heightened intensity of nitride phases. These findings underscore the scientific value and practical applicability of UNSM as an effective surface activation method. Consequently, the hybrid UNSM + IPN approach emerges as a promising strategy for prolonging the service life of critical load-bearing components like biomedical implants and other engineering parts exposed to severe wear.*

Keywords: Ti-6Al-4V, elastic modulus, hardness, coefficient of friction, ion-plasma nitriding, UNSM.

1. Introduction

The push in contemporary materials science is towards innovative approaches for strengthening materials, particularly those vital for sectors like aerospace, medicine, and automotive [1]. Much effort is directed at surface modification techniques designed to boost critical material properties, including wear resistance, hardness, and corrosion resistance. While Ti-6Al-4V titanium alloy is a top choice for structural applications due to its remarkable specific strength, light weight, biocompatibility, and environmental resilience [2], its relatively soft surface remains a challenge, hindering its performance in high-friction and wear environments [3]. Overcoming these limitations often involves surface engineering. A popular approach, ion-plasma nitriding (IPN), improves surface characteristics by forming hard titanium nitrides (TiN, Ti_2N), which significantly boost hardness (up to 2000 HV), chemical stability, and resistance to wear and corrosion [4,5]. Despite these benefits, achieving deep, uniform nitrided layers remains challenging due to the inherent passive oxide film, low surface defect density, and slow nitrogen diffusion in titanium alloys [6]. These factors often lead to thin, brittle, or weakly adherent layers with compromised mechanical properties [7]. A promising strategy to enhance nitrogen uptake during nitriding is surface pre-activation. Among the most advanced methods is ultrasonic nanocrystalline surface modification (UNSM), which generates a nanostructured surface with high dislocation density and refined grains via high-frequency mechanical impacts [8,9]. This surface architecture facilitates nitrogen diffusion, leading to thicker, more uniform nitrided layers during subsequent ion-plasma nitriding (IPN) [10]. Additionally, UNSM improves layer adhesion and reduces the risk of in-service delamination or cracking. Despite recognizing the benefits of combining UNSM and IPN, existing

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literature lacks a systematic analysis of how UNSM parameters (vibration amplitude, static load, processing temperature) affect the nitrided layer's structural evolution, phase composition, and tribomechanical performance. Conventional surface treatments like shot peening, PVD coatings, and laser shock peening offer limited control over surface grain structure modification or activation of chemical diffusion [11–13]. Unlike these methods, UNSM allows for precise parameter control and produces a dense, defect-rich surface that works synergistically with nitrogen plasma during IPN, leading to enhanced final performance. Despite the recognized benefits of combining Ultrasonic Nanocrystalline Surface Modification (UNSM) and Ion-Plasma Nitriding (IPN), current literature lacks systematic studies on how specific UNSM parameters - such as vibration amplitude, static load, and processing temperature - affect the structural evolution, phase composition, and tribomechanical performance of the nitrided layer. Traditional surface treatment methods, including shot peening, PVD coatings, and laser shock peening, show limited capabilities in modifying the surface grain structure or activating chemical diffusion [11–13]. In contrast, UNSM provides precise parameter control and creates a dense, defect-rich surface that synergistically interacts with nitrogen plasma during IPN, leading to improved final performance.

2. Materials and methods

In this work, we aim to investigate how pre-treatment with ultrasonic nanocrystalline surface modification (UNSM) impacts the formation and characteristics of a nitrided layer on Ti-6Al-4V alloy during ion-plasma nitriding. Our primary focus will be a detailed analysis of the layer's microstructure and tribo-mechanical properties, including its hardness, elastic modulus, and wear resistance. During the UNSM process, we adjusted key parameters: amplitude, static load, and temperature (refer to Table 1 for specifics). These settings were chosen based on our earlier research [14], which showed they effectively refine the grain structure and boost both the microhardness and elastic modulus of the Ti-6Al-4V alloy. After UNSM, the specimens naturally cooled to room temperature. The UNSM-treated surfaces were then subjected to ion-plasma nitriding (IPN). The IPN process was carried out using a specialized laboratory setup (Model LDMC-20, Tianman Industrial Furnace). A schematic representation of the equipment is shown in Fig. 1. The treatment was performed at a temperature of 500 °C for 2 h. The pressure in the chamber was maintained at 400 Pa. A glow discharge was initiated between the anode (the vacuum chamber body) and the cathode (the metal specimen) upon application of voltage. This discharge ionizes nitrogen atoms by knocking out electrons, forming nitrogen ions. These ions bombard the metal surface and penetrate into it, leading to the formation of a nitride lattice (Fig. 2).

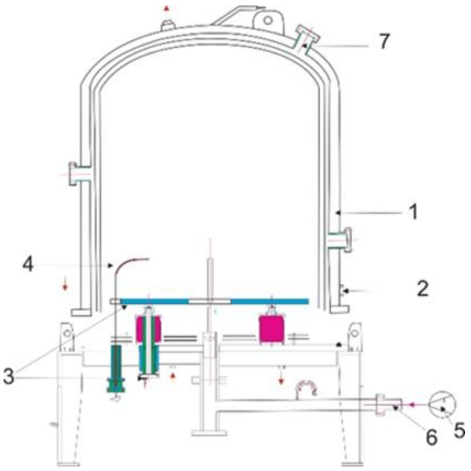

Fig. 1. Schematic diagram of the ion nitriding equipment: 1-vacuum chamber; 2-anode; 3-cathode (specimen holder); 4-vacuum chamber temperature sensor; 5-pumping system; 6-pressure relief plug; 7-vacuum viewing window.

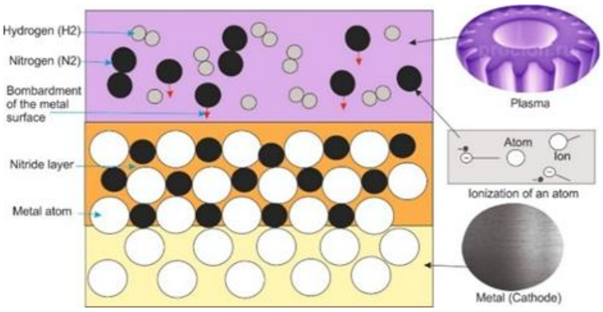

Fig. 2. The process of gas ion diffusion into the metal surface.

Table 1. Processing conditions for Ti-6Al-4V alloy specimens.

Samples	Sample Designation	Processing Conditions
S0	initial	initial
S1	UNSM-only	UNSM (20 μm,30 N, RT)
S2	UNSM-only	UNSM (30 μm,30 N, RT)
S3	UNSM-only	UNSM (30 μm,50 N, 400 °C)
S4	UNSM-only	UNSM (30 μm,60 N, 400 °C)
S0N	nitrided	nitrided
S1N	Combined	S1+ nitrided
S2N	Combined	S2+ nitrided
S3N	Combined	S3+ nitrided
S4N	Combined	S4+ nitrided

To evaluate the tribo-mechanical characteristics of the specimens, their microhardness, elastic modulus, and tribological properties were investigated. Hardness measurements were performed using a Vickers indenter on a FISCHERSCOPE HM2000S device (Sindelfingen, Germany). The indenter was a four-sided diamond pyramid with an angle of 136° between the opposite faces. Hardness was determined according to the Vickers scale (HV). The elastic modulus was calculated using the instrumented indentation method on the same device. All measurements were conducted according to the Vickers scale (HV), and each test was repeated five times to ensure accuracy. Tribological properties were evaluated in accordance with the ASTM G99 standard using a TRB3 tribometer (Anton Paar GmbH, Graz, Austria). A ball-on-disc configuration was used, where a 6.00 mm diameter 100Cr6 steel ball served as the counter body. The tests were conducted in ambient air at a laboratory temperature of 21.33 °C and relative humidity of 31.05%. The sliding radius was 2.00 mm, and the linear sliding speed was set to 10.0 cm/s. A constant normal load of 2.00 N was applied, and data were recorded at an acquisition rate of 10 Hz. The total sliding distance was 300.00 m. The test was conducted in a single-pass mode without pause, with initial homing enabled and no unloading at the end. The Ti-6Al-4V specimens were used as substrates without additional cleaning prior to testing. The phase composition was analyzed using an X'Pert Pro X-ray diffractometer (Panalytical, Amsterdam, Netherlands) with Cu-Kα radiation, operating at 40 kV and 30 mA. The scanning parameters were set to $35° < 2\theta < 85°$, with a step size of 0.02° and an exposure time of 5 s. The microstructure of the specimens was analyzed using a scanning electron microscope (TESCAN Vega, Tescan, Brno, Czech Republic). Prior to examination, cross-sectional samples were prepared through a series of standard metallographic procedures. First, the specimens were mechanically sectioned using silicon carbide cutting wheels. After sectioning, the surfaces were sequentially ground and polished using diamond paste to achieve a flat and deformation-free finish. To reveal the microstructural features, the polished samples were chemically etched for 10 s using Kroll's reagent, which consists of 100 mL distilled water, 1-3 mL hydrofluoric acid, and 2-3 mL nitric acid.

3. Results and discussion

3.1. Tribology Evaluation Results

Based on the analysis of the coefficient of friction (Fig. 3) and the wear surface morphology (Fig. 4), it was established that the parameters of UNSM significantly affect the tribological performance of the nitrided Ti-6Al-4V alloy. All samples exhibited two distinct stages: a running-in phase, during which the coefficient of friction (μ) increased rapidly, followed by a steady-state phase with stabilized friction behavior. The S4N sample (amplitude of 30 μm, load of 60 N, temperature of 400 °C) showed the lowest and most stable friction coefficient (~0.55) after a short running-in period (~40 m). SEM analysis revealed a uniform and dense wear track with minimal signs of plastic deformation, indicating high wear resistance and the presence of a protective nitride layer. The dominant wear mechanism is identified as mild abrasive wear. The S3N sample exhibited a slightly longer running-in phase and a steady-state of ~0.60-0.65. SEM images showed localized craters, micro-scratches, and indications of fatigue wear. Although the nitride layer was formed, its local heterogeneity led to selective degradation during prolonged contact. The S2N and S1N samples, treated at room temperature, displayed less stable friction behavior and a prolonged running-in period. Their coefficients of friction reached up to ~0.75 and exhibited fluctuations. SEM images revealed pronounced grooves, microcracks, and delamination zones. The prevailing wear mechanisms in these samples were abrasive and delamination wear, resulting from weak adhesion and insufficient depth of the nitrided layer. The S0N reference sample, without UNSM treatment, exhibited the most unstable friction behavior. After a short running-in phase, the coefficient of friction rapidly rose to ~0.80 and showed sharp fluctuations. SEM analysis indicated severe surface degradation, including plastic deformation and material detachment, consistent with intensive adhesive wear due to the lack of a reinforced surface layer. In summary, pre-treatment UNSM with optimized parameters enhances the formation of a dense and uniform nitrided layer, significantly improving wear resistance and stabilizing the frictional response. The S4N mode, in particular, proved to be the most effective, delivering superior tribological performance due to the combined effect of high amplitude, load, and temperature. In comparison to laser shock-peened and PVD-coated Ti-6Al-4V alloys, which typically exhibit coefficients of friction in the range of 0.65-0.80 under similar dry sliding conditions, our samples achieved more stable friction behavior and lower steady-state values (e.g., 0.55 for S4N), highlighting the tribological advantage of the combined UNSM and IPN approach [15].

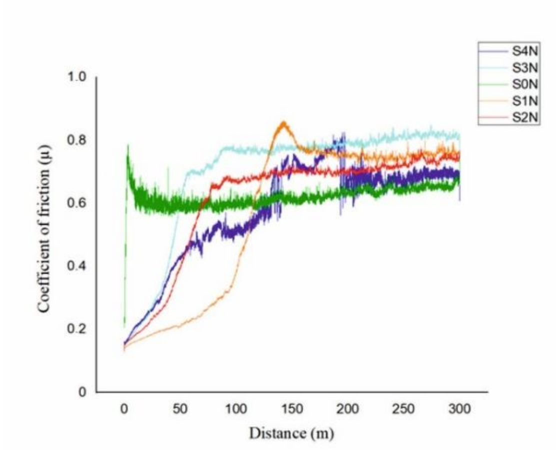

Fig. 3. Results of tribological tests under various UNSM treatment parameters after nitriding.

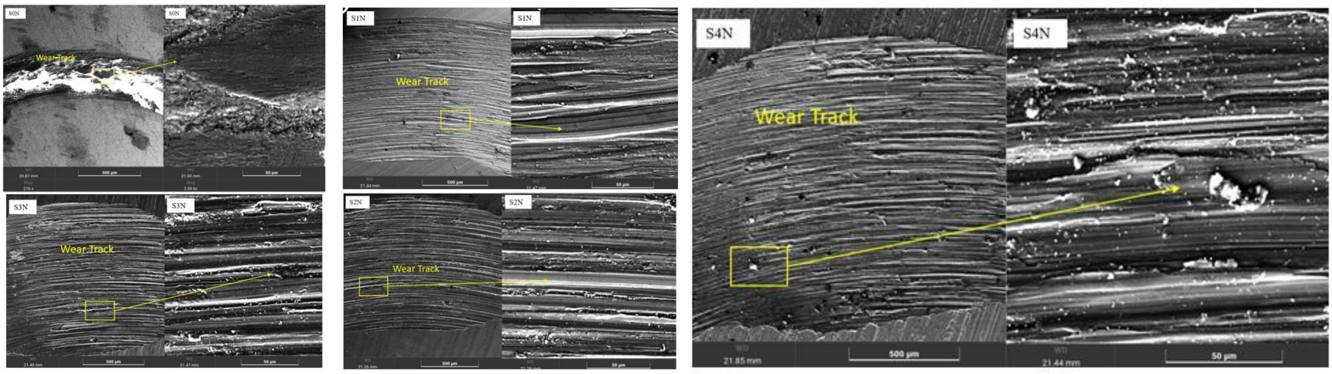

Fig. 4. SEM images of the wear surfaces of Ti-6Al-4V specimens after combined treatment by UNSM and nitriding.

3.2. X-Ray Phase Analysis

Fig. 5 presents the XRD patterns of Ti-6Al-4V specimens subjected to ion-plasma nitriding after UNSM treatment with varying parameters, as well as a reference sample S0N that was nitrided without prior ultrasonic treatment. In the S0N sample, prominent peaks of the α-Ti matrix dominate the pattern, accompanied by weak reflections corresponding to the $AlTi_3N$ and Ti_2N phases, indicating a relatively thin and less developed nitride layer. In contrast, the S1N sample (processed with minimum UNSM parameters) also shows dominant α-Ti peaks and only minor TiN signals, confirming limited nitrogen diffusion. The S2N pattern demonstrates a slight increase in the TiN peak intensity due to enhanced plastic deformation; however, the formation of the nitride layer remains insufficient without applying an elevated temperature. A significant increase in TiN peak intensity is observed in the S3N sample, alongside broader peaks that indicate a denser and more refined nitride layer. This result is attributed to the higher static load and processing temperature during UNSM. The S4N sample exhibits the highest intensity and sharpness of TiN and Ti_2N peaks, suggesting the formation of a thick, uniform, and nanocrystalline nitride layer. These findings confirm that pre-treatment by UNSM plays a crucial role in promoting phase transformations during nitriding. The combination of higher mechanical impact and thermal energy during UNSM, particularly in the S4N mode, provides optimal conditions for the TiN phase formation, as also supported by previous studies [9]. Thus, the XRD results confirm that preliminary ultrasonic surface modification plays a decisive role in phase transformations during ion-plasma nitriding, providing favorable conditions for the intensive formation of TiN phases. The most pronounced effect is achieved when high temperature and static load are combined during the UNSM process, as demonstrated in the S4N mode.

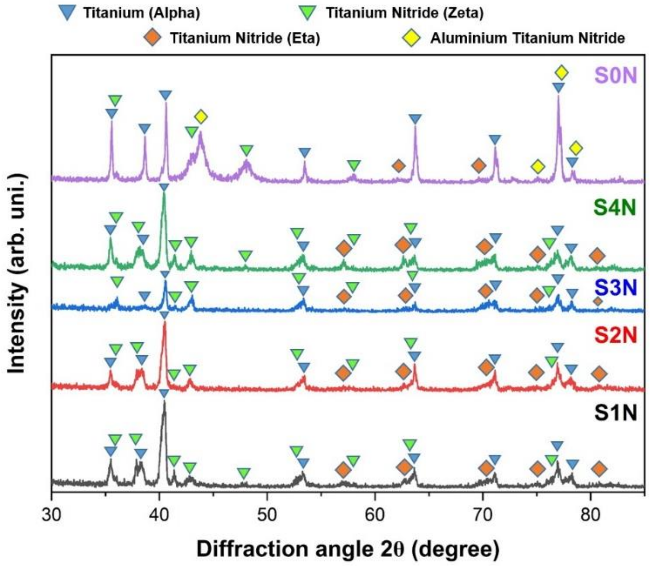

Fig. 5. X-ray diffraction (XRD) patterns of Ti-6Al-4V alloy after IPN with various UNSM treatment parameters.

3.3. Hardness

The parameters of the ion-plasma nitriding process were selected based on experimental data, which showed that the maximum surface microhardness of the Ti-6Al-4V alloy was achieved at a temperature of 500 °C and a duration of 2 h (Fig. 6). When the treatment time was increased to 3 h, a decrease in hardness was observed, which was likely associated with grain coarsening, possible recrystallization, relaxation of internal stresses, the formation of defects (such as cracks and delamination), and the homogenization of the nitrogen concentration gradient due to extended diffusion saturation. Similar effects were reported by Minoru Umemoto [13], where excessive nitriding time resulted in a reduction in hardness due to grain growth and the release of residual stresses. Therefore, the temperature of 500 °C provides active nitrogen diffusion without structural degradation of the material, making it optimal for the treatment of the Ti-6Al-4V alloy. The results presented in Fig. 7 clearly demonstrate the pronounced effect of the combination of UNSM and subsequent nitriding on the enhancement of surface hardness. Specifically, the combined treatment under the S1N regime resulted in more than a 25% increase in microhardness compared to the specimen subjected to nitriding alone (S0N). This indicates that preliminary UNSM treatment facilitates the active diffusion of nitrogen atoms into the material and the formation of hard nitride phases [20]. Such an enhancement is consistent with the findings of Sun et al. [21] and Mogucheva [22], which emphasize the significant role of prior plastic deformation in improving the conditions for nitrogen diffusion and the formation of the nitride layer. The S2N specimen, processed under similar conditions but with an increased amplitude (30 μm), exhibited a 17% increase in microhardness, further confirming the effectiveness of more intense surface deformation. In the S3N regime, at a temperature of 400 °C, the microhardness increased by 23%, confirming the activation of nitrogen diffusion processes due to enhanced atomic mobility. This effect has been reported in several studies, which have also emphasized the role of thermal activation in increasing the thickness and density of the nitride layer [23]. The least pronounced effect was observed for the S4N specimen, where UNSM was performed at 400 °C with a load of 60 N. The increase in microhardness was only 9.6%, which may be attributed to partial recrystallization, a reduction in defect density, or local overheating that diminished the plastic deformation effect [13].

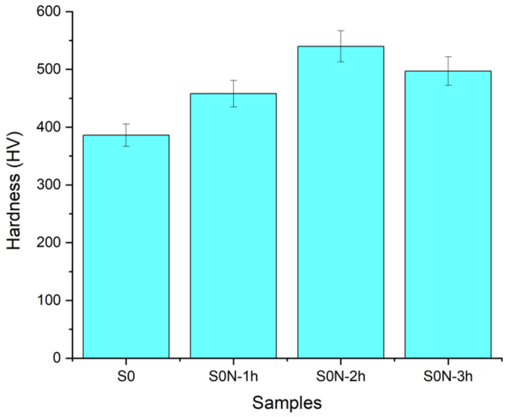

Fig. 6. Variation in the hardness of Ti-6Al-4V specimens at 500 °C with different nitriding durations.

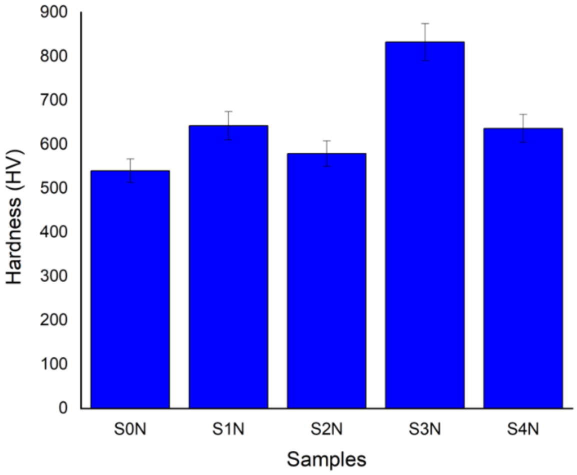

Fig. 7. Effect of different UNSM regimes on the hardness of Ti-6Al-4V alloy after nitriding.

The greatest improvement in microhardness (over 25%) was achieved under moderate UNSM conditions (amplitude of 20 μm, load of 30 N, RT), which provided an optimal degree of plastic deformation that enhanced nitrogen diffusion. Although increasing the UNSM temperature combined with high loads may further activate diffusion processes, in some cases it leads to reduced hardening efficiency, likely due to recrystallization or the partial relaxation of internal stresses [13,23]. Preliminary UNSM treatment prior to nitriding contributes to an increase in both the elastic modulus and microhardness due to the activation of diffusion processes and the formation of a nanostructured subsurface layer. Repeated high-frequency impacts induce intense plastic deformation, increase dislocation density, and generate crystal lattice defects (dislocations, sub-boundaries, vacancies), which create internal resistance to deformation and thus enhance resistance to elastic strain. This phenomenon, known as dislocation hardening, effectively increases the apparent elastic modulus during surface-sensitive localized measurements [17]. In addition, the presence of a greater number of high-angle grain boundaries and the formation of nanostructures further restrict atomic movement, contributing to localized stiffness enhancement [24]. The experimental results obtained in this study confirm this effect. As shown in Fig. 8, the maximum values of microhardness and elastic modulus were achieved under UNSM parameters that provided controlled plastic deformation without overloading the surface (amplitude of 20 µm, load of 30 N, RT). Such treatment promotes a uniform distribution of residual stresses and the formation of a structurally active surface favorable for nitride phase formation during subsequent ion nitriding. Thus, a clear correlation was established between the parameters of the preliminary treatment and the mechanical properties of the modified layer, enabling the targeted design of surface structure and properties for the development of wear-resistant functional coatings [25]. The microhardness values obtained in this study (e.g., up to 25% increase after UNSM + IPN treatment) demonstrate a noticeable improvement compared to those reported for Ti-6Al-4V alloys treated using conventional PVD, plasma ion implantation, and laser nitriding methods, where hardness enhancement typically ranges from 10% to 15% [22]. This suggests that the pre-treatment by UNSM significantly enhances nitrogen diffusion and contributes to the formation of a more hardened surface layer.

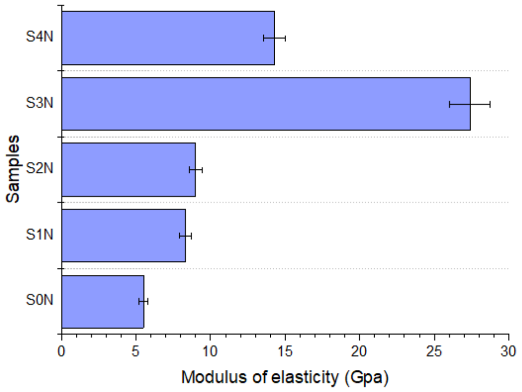

Fig. 8. Elastic modulus of Ti-6Al-4V specimens under different UNSM treatment parameters after nitriding.

4. Conclusion

UNSM pre-treatment boosts microhardness by up to 25% and the elastic modulus by 18% compared to samples without this initial activation. Moreover, UNSM improves the tribological performance of the nitrided layer. The most favorable outcomes were observed in samples treated at an amplitude (A) of 30 µm, a static load (F) of 60 N, and a temperature (T) of 400 °C, which exhibited a stable coefficient of friction of approximately 0.55. SEM and XRD analyses confirmed that UNSM promotes the formation of a more uniform and dense nitride layer. This indicates that UNSM is an effective method for enhancing both the structural integrity and functional performance of nitrided layers on Ti-6Al-4V alloy. Given the improved surface hardness, refined microstructure, and enhanced wear resistance, UNSM-treated Ti-6Al-4V alloy is poised for use in advanced industries. This includes biomedical engineering (e.g., hip implants, dental screws), aerospace systems, and precision mechanical components that face high contact stresses and corrosive environments.

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Conflict of interest
The authors declare that they have no competing interests.

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