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Nano gradient structuring at Ti-6Al-4V surface induced by ultrashort-pulse laser peening

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ABSTRACT

We have successfully fabricated gradient micro/nano structuring on the surface top layers of Ti-6Al-4V alloy using a shock-peening technique facilitated by femtosecond and picosecond laser pulses, without the need for coatings or confinement. These micro/nano structures encompass ultrafine grains, extensive subgrain boundaries, hierarchical nanotwins, and complex dislocation morphologies. The ultrafine grains, ranging in size from hundreds of nanometers to a few micrometers, were predominantly located in the grain-refined regions, whereas the micro/nano dislocation structures were predominantly found in regions of severe plastic deformation. These observations suggest a promising avenue for achieving high-precision gradient structuring in the field of metallic surface engineering.

1. Introduction

Tailoring the micro and nano structures of metallic materials, such as refined and lamellar grains, high-order nanotwins, and dislocation architectures, can effectively improve their mechanical performances [1]. Hence, the manipulation of the micro/nano structures is emerging as a pivotal technological approach for metal reinforcement to overcome the strength-ductility trade-off dilemma. Interestingly, the mechanical properties of metals can be further optimized by integrating multiple advanced micro/nanostructures to create a gradient. The underlying mechanism is that the microscopic structural heterogeneities induce non-synchronous and non-uniform deformation behaviors under a loading [2], which in turn modifies the macroscopic mechanical performances, leading to improved strength-ductility synergy, fatigue resistance, corrosion resistance, and wear resistance [3–5]. Over the past two decades, gradient-structured micro/nanomaterials have exhibited extraordinary mechanical properties, a phenomenon observed across various engineering alloys [6].

Laser-shocked peening is a developing advanced technology for metal surface strengthening and gradient structuring. Laser-driven shock wave, capable of inducing ultrahigh strain rate ($> 10^6$ s^{-1}) and compressive residual stress into metal surface layers, can create a grain gradient and improve the surface performances including fatigue and corrosion resistance [7–9]. Traditional high-energy nanosecond laser peening typically requires the sacrifice of a protective coating under water confinement to generate a high-temperature plasma explosion [10]. However, this method is associated with several drawbacks, including a complex process, significant thermal diffusion, and potential surface damage. Ultrashort-pulse lasers, with pulse durations typically less than ten picoseconds, provide substantial advantages for high-precision material processing and surface modification. For instance, femtosecond laser can induce a peening effect on metal surfaces without the need for coating and confinement, thanks to its ultrahigh peak intensity [11–13].

Although the optimization of surface mechanical performance through structural gradient tailoring is effective, current manufacturing approaches, such as conventional mechanical methods and nanosecond laser-shock peening [6], are limited for high-precision high-quality gradient structuring. Hence, it is of paramount importance to investigate the effects of ultrashort-pulse laser peening on micro/nano structural

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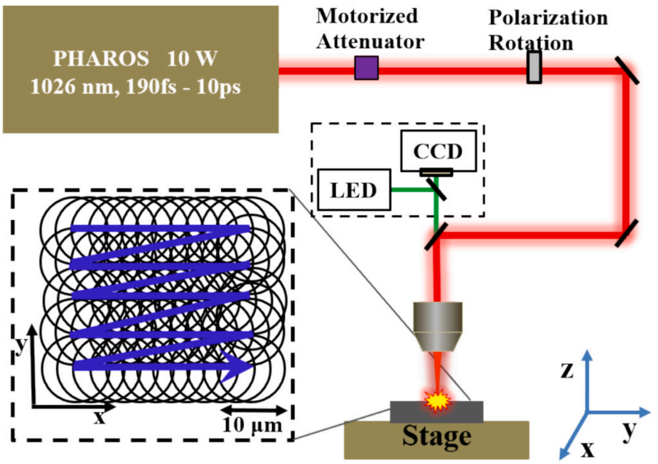

Fig. 1. Schematic of ultrashort-pulse laser-peening experimental setups and the laser scanning model.

modification in surface layers [14]. This method is renowned for its unique and highly reproducible outcomes, rendering it a reliable option for both research and engineering applications. Microstructural characterization resulting from laser-shock strengthening typically employs electron microscopy techniques [10], including scanning electron microscopy (SEM), transmission electron microscopy (TEM), and electron backscatter diffraction (EBSD). Among these methods, TEM offers the advantage of directly observing crystal micro/nanostructures, albeit requiring complex sample preparation.

In our previous work, we have successfully achieved the nearly nondestructive surface strengthening by femtosecond laser peening with low energy and ultrahigh pulse density [15]. Here, we have further explored the gradient micro/nano structuring on the surface of Ti-6Al-4V using ultrashort-pulse laser peening, employing a process free from coating and confinement. Based on the TEM observations, we conducted a comparative analysis of the peening effects and gradient structures generated by femtosecond and picosecond lasers. Moreover, a comparison was carried out between ultrashort-pulse laser peening and convention mechanical methods, as well as nanosecond laser-shock peening, with respect to the gradient structuring effect.

2. Experimental setups

The ultrashort-pulse laser peening experiments were conducted using a femtosecond laser micromachining system (FemtoLAB, Workshop of Photonics). As illustrated in Fig. 1, a Yb: KGW laser (PHAROS) operating at 200 kHz repetition rate was employed for the laser peening process. Ti-6Al-4V samples in dimensions of 10 × 10 × 1 mm^3 were mounted on a high-precision positioning stage. The lasers were normally irradiated at the mirror-polished sample surface without coating and confinement in air after computer-controlled motorized attenuator and polarization rotation. The incident Gaussian laser spot size was approximately 10 μm ($1/e^2$ intensity level), generated by an Olympus microscope objective (5×, NA = 0.13). The laser-peening procedure was monitored using a CCD device. To achieve effective laser peening effect, we adopt a novel low-energy ultrahigh-pulse-density laser scanning model. The pulse durations for the femtosecond and picosecond lasers were 190 fs and 10 ps, respectively, with corresponding fluences of 0.6 J cm^{-2} and 0.96 J cm^{-2}. The overlapping rate in the y-direction is approximately 50 %. While in the x-direction, the laser pulse density is 6×10^8 pulses/mm^2.

The surface morphology of Ti-6Al-4V samples was characterized using a 3D noncontact optical profilometer (NewView 9000, ZYGO Corp., USA), renowned for its sub-nanometer accuracy along the normal direction due to its advanced coherence-scanning interferometry technology. The gradient structuring features were characterized by a JEOL JEM-F200 transmission electron microscope (TEM) operated at an accelerating voltage of 200 kV. TEM foils were extracted from the laser-peening treated regions of the Ti-6Al-4V samples using ion-beam cutting techniques.

3. Results and discussion

Fig. 2 presents a comparative analysis of surface morphology and microstructural changes induced by ultrashort-pulse laser peening on Ti-6Al-4V alloy. The results show that femtosecond laser peening causes minimal material removal and slightly altered surface roughness

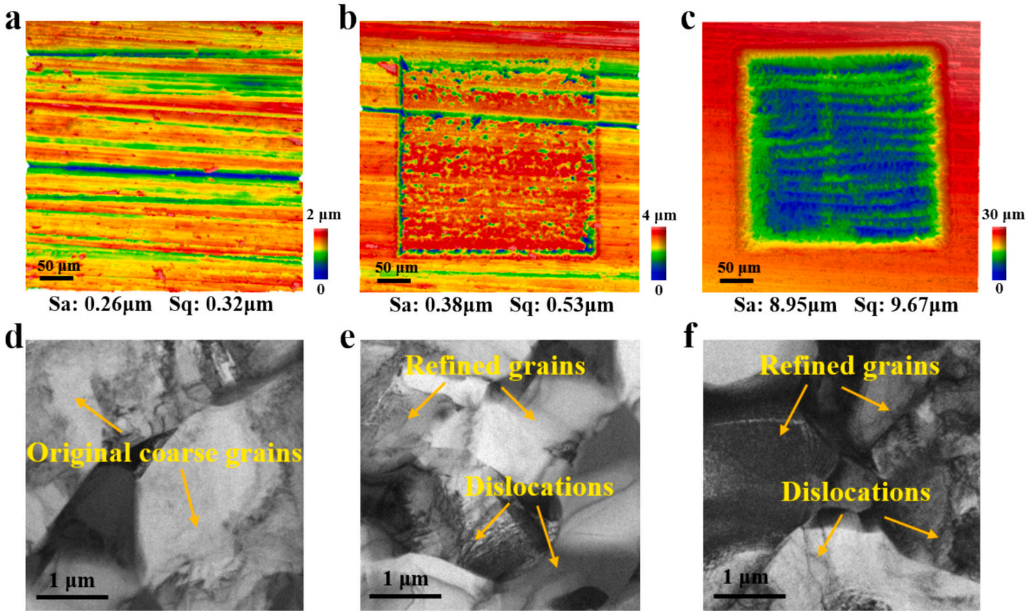

Fig. 2. Comparisons of surface morphology and microstructures between pristine and ultrashort-pulse laser-peened Ti-6Al-4V alloy samples. Surface morphology of (a) pristine, (b) femtosecond laser peened, and (c) picosecond laser peened samples. TEM images of (d) pristine, (e) femtosecond laser peened, and (f) picosecond laser peened sample surfaces. Sa: arithmetic mean deviation. Sq: root mean square deviation.

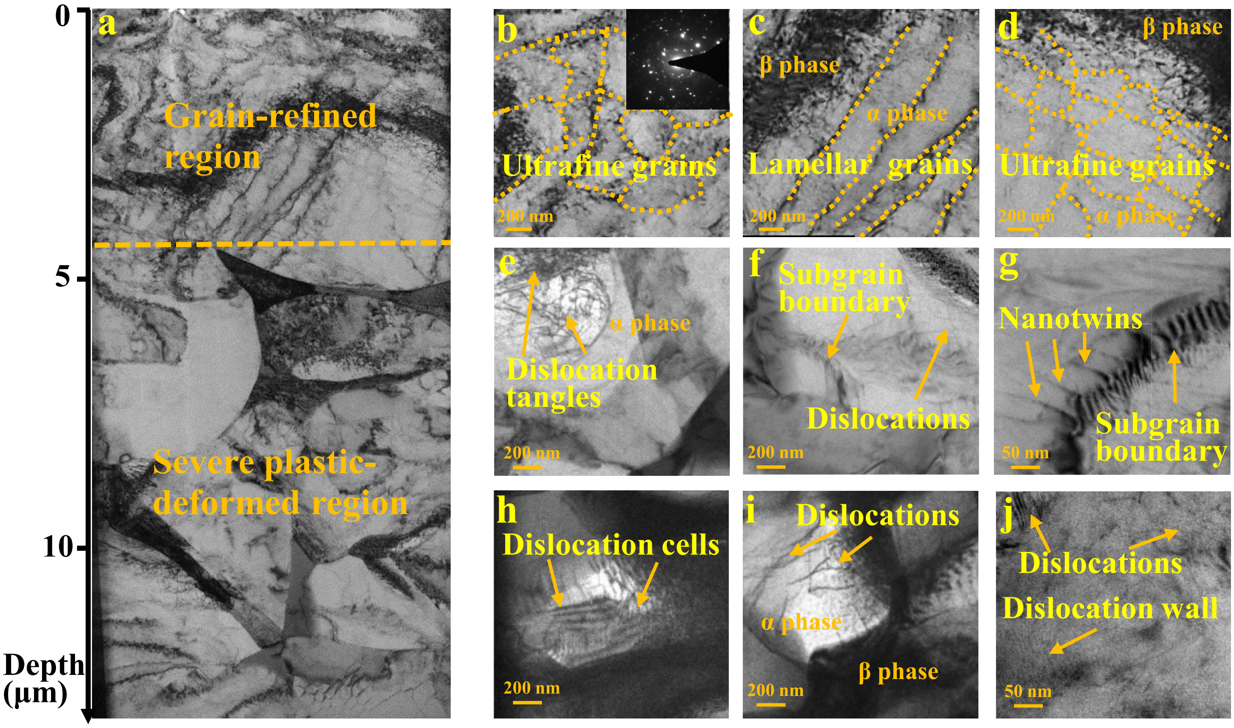

Fig. 3. Gradient micro/nano structural features on the top surface (at a depth of ~15 μm) of the studied Ti-6Al-4V alloy produced by femtosecond laser peening. (a) Microscopic observation by TEM at depth direction. (b–d) Ultrafine grains at grain-refine regions close to the laser-peened surface. The inset in (b) represents a SAED pattern of refined grains. (e–j) Micro and nano structures at severe plastic deformed region induced by femtosecond laser peening, including dislocation tangles, subgrain boundary, nanotwins, dislocation cells and dislocation walls.

compared to the pristine surface, owing to the adoption of a low-energy laser scanning model. In contrast, picosecond laser peening results in more pronounced phase transformation and material removal, reaching a maximum removal depth of nearly 30 μm. This distinction arises from the picosecond laser's greater thermal effects compared to femtosecond lasers. Specifically, femtosecond lasers induce phase explosion and particle ejection due to their ultrahigh peak intensities, while picosecond lasers primarily induce thermal accumulation and phase changes on the metal surface [16,17]. Compared to conventional nanosecond laser shock peening, which typically induces surface roughness on the scale of dozens of micrometers [18,19], ultrashort-pulse laser peening can achieve significantly improved surface quality through a low-energy ultrahigh-pulse-density approach. This method utilizes highly repetitive low-energy laser pulses to minimize surface damage while maximizing the peening effect through multiple impacts.

Furthermore, the microstructural changes following ultrashort-pulse laser peening treatment are notable. The coarse grains undergo refinement, and there is a substantial proliferation of dislocation patterns. Transmission electron microscopy observations reveal microstructural characteristics in the Ti-6Al-4V alloy similar to those induced by nanosecond laser shock peening [20–22]. However, a significant limitation of ultrashort-pulse laser peening is its restricted impact depth. It is indicated that femtosecond lasers can induce higher strain rates compared to nanosecond lasers, making them particularly suitable for precise structural modification in micro parts [23]. This characteristic underscores the potential of femtosecond lasers in achieving fine-scale structural modulation.

Fig. 3 presents the gradient micro/nano structures generated by femtosecond laser peening in the Ti-6Al-4V alloy surface, extending along the depth direction. The laser-peened surface layer, characterized by a hierarchical structure and approximately a dozen micrometers in thickness, is discernibly divided into two distinct regions: a grain-refined region and a severely plastic-deformed region. In the nanograined top layer with a thickness nearly 3 μm, ultrafine grains, measuring hundreds of nanometers in size, were identified (Fig. 3b–d). Those nanograins are capable of effectively enhancing the strength and deformation-bearing capacity of the metal matrix [24]. The inserted SAED pattern in Fig. 3b confirms the formation of nanoscale polycrystals within the grain-refined region. Besides, lamellar structures, featuring numerous low-angle grain boundaries, exist in low-energy states, conductive to structural stabilization and refinement [25,26]. Beneath the nanograined layer lies the coarse-grained region, marked by severe plastic deformation. A multitude of dislocation morphologies, such as tangles, dense dislocation cells, dislocation walls and lines, as well as nanotwins and subgrain boundaries, are observed (Fig. 3e–j). Those micro and nano structures play an important role in improving the mechanical properties of the metal matrix. To date, dislocation architectures and twinning engineering remain significant technological approaches for overcoming the trade-off between strength and ductility in metal strengthening [27,28].

The evolution of microstructures and the formation of gradient structures are intimately linked to the propagation of laser-shock energy. When subjected to sufficient shock energy and ultrahigh compressive strain rates, the dislocation multiplication in β phase and deformation twinning in α phase are activated in Ti-6Al-4V alloy. Dislocation motion and accumulation lead to the formation of numerous dislocation nanostructures within the original grains, such as dislocation tangles and irregular blocks consisting of dense dislocation walls. These structures subsequently transform into subgrain boundaries and eventually evolve into refined grain boundaries [29]. Hence, the top layer exhibits a significant generation of ultrafine grains following laser-peening treatment. As the shock energy diminishes with depth, there is insufficient driven energy to significantly deform and refine the coarse grains. Instead, a variety of dislocation tangles, nanotwins, and subgrain boundaries are formed to accommodate the relatively severer plastic deformation. Furthermore, with a further reduction in shock energy and strain rate, only mild dislocation activities are initiated, including sliding, interaction, and slight accumulation, leading to the formation of

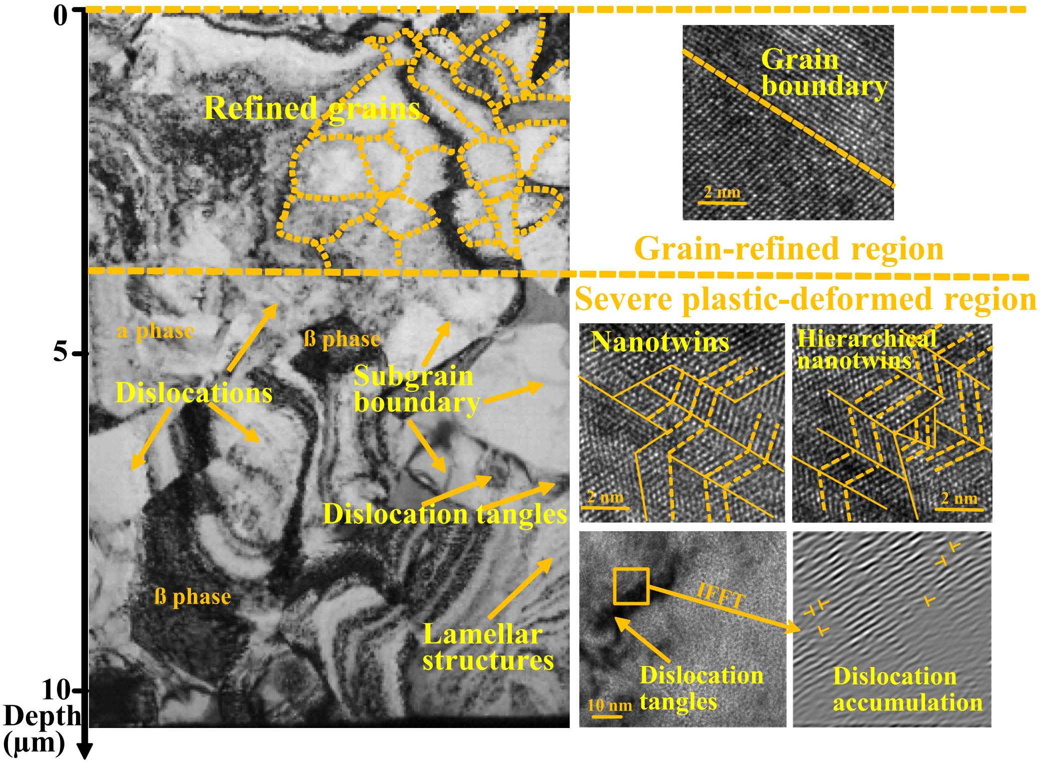

Fig. 4. TEM characteristics of gradient micro/nano structures along depth direction at surface of the studied Ti-6Al-4V alloy produced by picosecond laser peening, including refined grains, lamellar structures, subgrain boundary, hierarchical nanotwins, dislocation tangles and accumulation. The high-resolution TEM pictures present the grain boundary, nanotwins and dislocation tangles, respectively. The local dislocation distribution and accumulation are revealed by inverse fast Fourier transform (IFFT) image of the magnified square region.

tangles.

Those gradient micro and nano structures are capable of significantly enhancing the surface mechanical performance of the metal matrix. Upon an external loading, distinct plastic deformation mechanisms are activated within those gradient structures, including dislocation movement in coarse grains and grain boundary migration in refined grains [30]. The pre-existed micro and nano structures impede dislocation slip and pin the dislocations, thus enhancing the load-bearing capacity and strength of the metal matrix. Meanwhile, the suppression of intergranular stress and strain localization in gradient grains undergoing plastic deformation mitigates the generation of cracks at grain boundaries, thereby significantly increasing the ductility [2].

Several perspectives have been put forward to elucidate the fundamental mechanisms responsible for the exceptional strength-ductility synergy of gradient-structured metal materials. These include the concept of heterogeneous deformation-induced back stress [31], plastic strain gradient [32], twinning-dislocation interaction [33], and grain coarsening [34]. Specifically, a widely recognized unifying principle that counts for these strengthening mechanisms is the unique patterning of geometrically necessary dislocations (GNDs), which occurs during inhomogeneous plastic deformation. The accumulation and concentration of GNDs at the boundaries of structural domains lead to the generation of a back stress field and the establishment of a plastic strain gradient.

Compared with corresponding homogeneous counterparts and other heterogeneous microstructures such as bimodal grains embedded with micro-nano reinforcements [35], gradient structures demonstrate significantly enhanced surface mechanical performance. This enhancement is attributed to their superior strain-hardening capability, which is a result of the induction of GNDs [36]. It is reported that the formation of long-range GNDs in gradient grains under tensile loading contributes to an additional work hardening. Consequently, gradient specimens exhibit a more favorable balance of strength and ductility over their homogeneous counterpart [37]. A dislocation density-based continuum plasticity model has quantitatively validated that the strain hardening capability of gradient layers can rival that of coarse grains. This is due to the prolific generation of abundant GNDs during non-uniform deformation in gradient layers [38]. A recent research, combining experiment and strain gradient plasticity modeling by Cheng et al., has demonstrated that an increase in structural gradient leads to a progressive plastic strain gradient, facilitated by the accumulation of GNDs. This results in an elevated level of back stress and strength [39].

Laser peening offers an effective pathway for inducing gradient micro/nano structuring, thereby enhancing the strength-ductility performance of metal surfaces. The shock-peening effect induced by ultrashort-pulse lasers in the absence of coating and confinement, originates from rapid plasma expansion when laser pulses interaction with metal surfaces. Hence, laser peak intensity and pulse width are the primary factors influencing the peening effect. The former determines the intensity of plasma explosion, whereas the latter has a direct impact on the duration of peening and the extent of thermal diffusion. In addition to femtosecond laser peening, there is a scarcity of reports on laser-shock surface strengthening produced via picosecond lasers.

Compared to femtosecond lasers, picosecond laser ablations exhibit a more pronounced thermal effect, as the picosecond pulse width approaches or exceeds the electron-phonon coupling times of metallic lattices. This means that picosecond lasers result in a thicker ablated layer and more severe thermal damage at target surface. However, picosecond lasers are capable of inducing a sufficiently intense plasma explosion, as well as a longer acting time. With low energy density and comparatively narrower pulse widths (a few picoseconds), picosecond lasers have vast potential in producing an excellent shock-peening effect

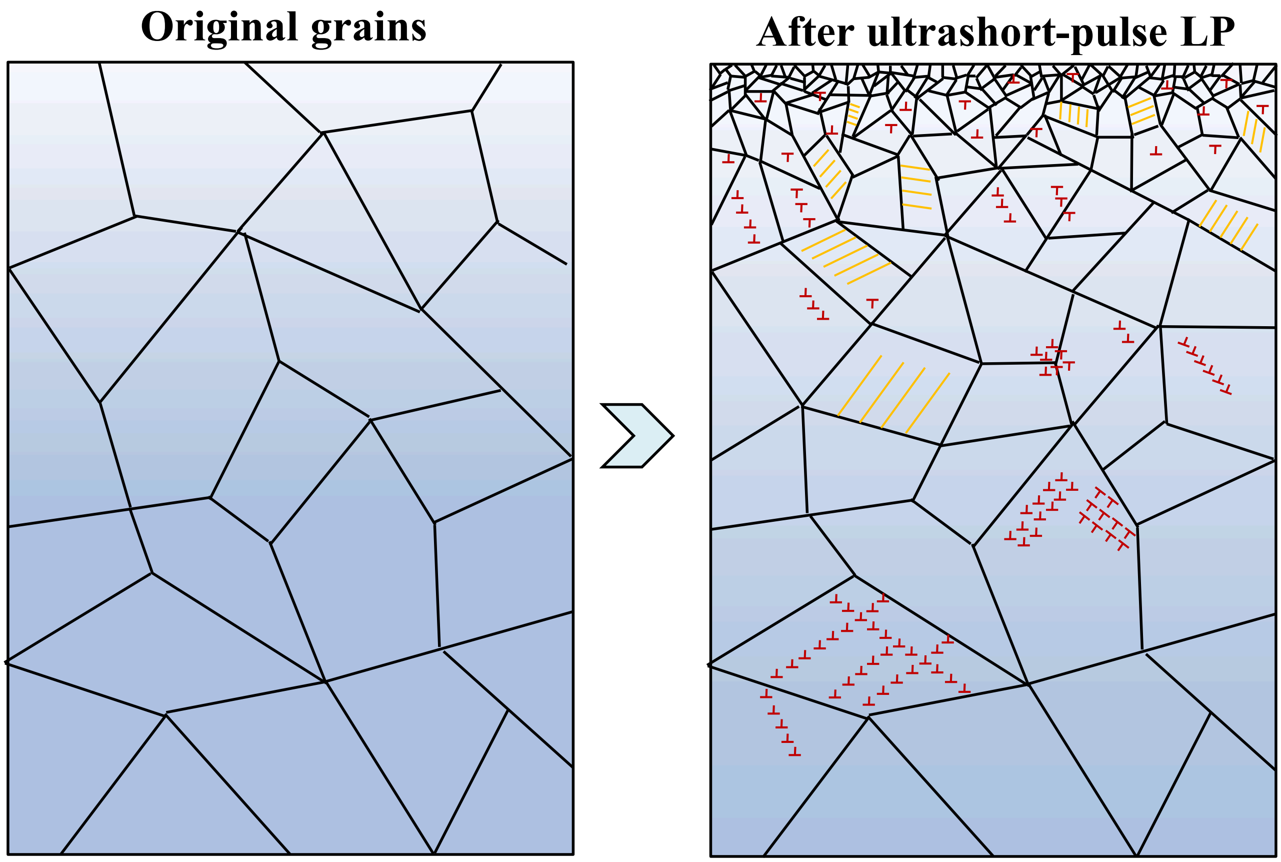

Fig. 5. Schematic diagram of the micro/nano structures before and after ultrashort-pulse laser peening (LP) treatment.

[40]. Therefore, we have conducted the picosecond laser peening experiments to validate its shock-peening effect and explored the new technical pathway for metallic surface strengthening and gradient structuring.

Fig. 4 presents the gradient structural features on the top surface of Ti-6Al-4V alloy with a thickness of approximately 10 μm achieved through picosecond laser peening. The compressive deformed top surface layers are divided into grain-refined and plastic-deformed regions. The depth of grain refinement is comparable to that observed in femtosecond laser treatments. However, the refined grains produced by picosecond laser peening typically have a diameter size nearly one micrometer, which is moderately larger than the nanometer-scale grains produced by femtosecond lasers. Despite the fact that extraordinary strength can be obtained with extremely fine grains (dozens of nanometers) according to typical Hall-Petch relation, an optimum grain size of a few micrometers was recently reported by Wang et al. for strength-ductility synergy of metals [41]. Therefore, it may be imprudent to persistently seek higher peak shock intensities and finer grains through the use of shorter laser pulses in the field of laser peening. An optimal laser pulse width may exist, and picosecond laser peening holds significant potential for metal surface strengthening.

Subgrain boundaries, nanotwins and various dislocation morphologies are evident in picosecond laser-treated specimen, as displayed at severe plastic-deformed region in Fig. 4. The compressive shock waves generated by picosecond lasers facilitate dislocation movement and multiplication, resulting in the formation of diverse dislocation structures. A localized square region with gradient dislocation densities (as shown in Fig. 4) was selected for the analysis of dislocation distribution and evolution.

The inverse fast Fourier transform (IFFT) image of the square region reveals a distribution of scattered dislocations and their accumulations within intricately tangled structures. Moreover, deformation twinning is commonly observed at high stress conditions, especially with high strain rate [42]. The compressive strain rate induced by laser shock is markedly higher than that achieved in conventional mechanical testing, differing by several orders of magnitude. Hence, hierarchical nanotwins are generated and dispersedly distributed in the severe plastic-deformed coarse grains. Twin boundaries effectively impede dislocation movement and serve as slip planes to accommodate dislocations under mechanical loading, thus promoting the strain hardening [43]. The hierarchical nanotwin structures contribute to a complex three-dimensional boundary architecture, which more effectively hinders dislocation motion, providing a novel design for highly promising metallic materials.

The aforementioned TEM observations confirm the viability of shock-peening strengthening techniques employing ultrashort-pulse lasers. In contrast to the complex technical procedures associated with conventional nanosecond-laser shock peening, ultrashort-pulse lasers are capable of inducing a significant shock peening effect on metal surfaces, eliminating the necessity of coating and confinement. This is attributable to their ultrahigh transient intensity, which significantly simplifies the technical process. However, while the gradient structuring on Ti-6Al-4V surfaces induced by ultrashort-pulse lasers resembles that achieved by nanosecond laser shock peening [44,45], the penetration depth is noticeably shallow. This limitation arises from the need to preserve surface integrity, necessitating constrained laser energy application to prevent substantial laser ablation and material removal. Consequently, the peening depth achieved by ultrashort-pulse lasers, typically in the range of tens of micrometers, is significantly lower than that achieved by conventional high-energy nanosecond lasers, which can reach depths of hundreds of micrometers [46]. This renders ultrashort-pulse lasers particularly suitable for applications that require precise and shallow peening depths.

Furthermore, ultrashort-pulse lasers are capable of inducing a substantially higher strain rate and peak pressure compared to nanosecond lasers. This, in turn, promotes the formation of lattice defects with high-forming energy, such as complex dislocation arrangements, profuse twins, and stacking faults [23]. The ultrahigh strain rate reduces the time for dislocation slip, and the high peak pressure supplies sufficient energy for the formation of these defects. Consequently, ultrashort-pulse laser peening presents a distinct advantage in achieving gradient structuring characterized by hierarchical and heterogeneous micro/nano structures, which has significant potential for enhancing exceptional mechanical properties.

The gradient micro/nano structuring illustration at metal surface by ultrashort-pulse laser peening is presented in Fig. 5. After ultrashort-pulse laser-peening treatment, gradient grains are generated in size from hundreds of nano meters to a few micro meters, accompanied with various dislocation morphologies and a dense array of nanotwins. Those tough micro/nano gradient structures effectively improve the mechanical properties and functionality of peened metal surfaces. In comparison with traditional mechanical methods for gradient structuring, such as attrition [47], rolling [48], and grinding [49], ultrashort-pulse laser peening takes the advantages of high-precision, stability, and spatial flexibility. However, due to the limitations imposed by the restricted energy output and small beam size, ultrashort-pulse laser peening encounters challenges in achieving macroscopic surface engineering. Therefore, it is suitable for strengthening the surface and interface of micro components and for creating localized gradient structures, which are particularly relevant in automotive, aerospace, and medical applications.

Ultrashort-pulse laser peening technique offers a novel and effective approach for gradient structuring and strengthening of metal surfaces, particularly for micro or complex spatially shaped industrial components that are not amenable to conventional nanosecond-laser peening or mechanical strengthening methods. Future research endeavors should focus on a comprehensive understanding of the impact of laser parameters on the formation of the micro/nano structures. This will enable precise manipulation over gradient structuring, especially at the nanometer scale, and further enhance the peening depth and distribution of micro/nano structures, where preserving an optimal surface finish.

4. Conclusions

In conclusion, we have successfully implemented gradient micro/nano structuring on the surface of Ti-6Al-4V alloy through ultrashort-pulse laser peening, eliminating the need for coatings and confinement. Femtosecond and picosecond lasers are capable of inducing gradient grains and a variety of micro/nano structures, including subgrain boundaries, nanotwins, dislocation arrays, and tangles. Notably, the femtosecond laser is particularly effective at generating ultrafine grains within the nanometer scale, a result of its superior peak intensity. In contrast to traditional nanosecond laser shock peening techniques and mechanical methods typically employed for microstructural gradient formation, ultrashort-pulse laser peening presents significant advantages in precision and spatial adaptability, particularly for the surface strengthening of microcomponents.

CRediT authorship contribution statement

Haimin Ding: Writing – review & editing, Investigation, Funding acquisition, Conceptualization. **Qing Peng:** Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare no conflict of interest.

Acknowledgments

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Data availability

All data generated or analyzed during this study are included in this published article.

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