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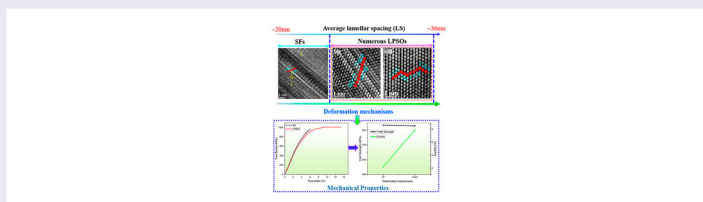
Long-period stacking ordering induced ductility of nanolamellar TiAl alloy at elevated temperature

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ABSTRACT

This work reports that the plastic deformation mechanism of lamellar microstructure (LM) in Ti-43.5Al-4Nb-1Mo-0.1B (TNN) alloy transforms from stacking faults (SFs)-dominated process to long-period stacking ordering (LPSO) structures-mediated process with a slight increase in lamellar spacing (LS) (20–36 nm). Multiple LPSO bands significantly enhance the work hardening response and trigger the transformation-induced plasticity (TRIP) effect, causing a four-fold higher ductility than SF-deformed LM at 750°C without compromising yield strength (YS). This phenomenon provides novel insights into the development of high-performance TiAl alloys with extremely nano-LM (LS < 55 nm) at elevated temperatures.



IMPACT STATEMENT

The development of multiple nanometer-sized LOPS bands during deformation in nanolamellar TiAl alloys can significantly improve ductility without resulting in a reduction of yield strength.

ARTICLE HISTORY

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KEYWORDS

TiAl alloy; Long-period stacking ordering; Mechanical properties; Stacking faults

According to the classical Hall–Petch equation, a decrease in the grain size or lamellar spacing (LS) of the lamellar microstructure (LM) often increases the (YS) of metals because the smaller grain size increases the concentration of grain boundaries, providing more barriers to dislocation motion. However, when the grain size is reduced to a critical size, the plastic deformation mechanism switches from the dislocation-mediated process to other mechanisms, such as grain boundary slipping [1] and strain-driven grain growth [2], invalidating the Hall–Petch relationship. This transformation exerts an important influence on the mechanical properties of metals. For example, grain boundary slipping significantly improves the plasticity of materials [3,4].

TiAl alloys are promising intermetallic compounds that are expected to replace heavier nickel-based superalloys in the temperature range of 650°C–800°C for aerospace applications [5,6]. Among four classical microstructures of TiAl alloys, the LM, which consists of parallel α_2 (Ti₃Al) and γ (TiAl) lamellae, has always been considered the most promising microstructure for high-temperature engineering applications [7,8]. The YS (δ_y) of LM and its lamellar spacing λ are in accordance with the Hall–Petch formula at both room temperature and high temperature [9–11], namely:

$$\delta_y = \delta_0 + \kappa/\sqrt{\lambda},$$

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where δ_0 and κ are material constants. Hence, a higher YS of LM can be obtained by refining lamellar thickness. However, YS was found to saturate as λ decreased to a critical value [12,13]. At the nanoscale, activation of dislocation slipping and mechanical twinning becomes difficult [14], and the deformation is dominated by other mechanisms. Since 1990s, LPSO structures, such as 9R and 6H, have been occasionally observed in severely deformed TiAl alloys [15,16], which could be considered as a novel deformation mechanism of nano-LM. Exploring the effect of LPSO on mechanical properties is of great significance for regulating mechanical properties of TiAl intermetallics. Nonetheless, this is extremely challenging because the variability of LS causes various deformation mechanisms to operate simultaneously.

This work reveals a novel plasticizing mechanism associated with the development of numerous LPSO bands during high-temperature deformation in nanoscale LMs (with maximum LS < 80 nm). The proposed mechanism implies that the ductility is considerably improved without compromising the YS, rendering an unprecedented combination of ultrahigh strength and tensile ductility. The deformation mechanisms of nano-LMs with various LSs were characterized via high-resolution transmission electron microscopy (HRTEM).

The Ti-43.5Al-4Nb-1Mo-0.1B (TNM) alloy was forged at 1170°C with a total engineering deformation strain of 74%. The maximum service temperature of the TNM alloy is 750°C. The detailed microstructure of the as-forged TNM has been reported elsewhere [17]. To obtain LM with the desired LS, a two-step solution and aging heat treatment process was devised. Initially, cylindrical specimens with a diameter of 10 mm and a length of 70 mm were cut from the forged billets using electrical discharge machining. Then, these samples were subjected to a solution treatment at 1280°C for 1 h and air-cooled to room temperature. Afterwards, they were aged at 850°C for 6 h (LM₁), 12 h (LM₂), 24 h (LM₃), and 40 h (LM₄) and then furnace-cooled to room temperature (RT). One should note that prolonging the aging time increases both the LS and the lamellar colony size. Thread circle-shaped tensile specimens with a gauge diameter of 3 mm, gauge length of 15 mm, and total length of 50 mm were fabricated from the heat-treated materials according to the HB-5143 96 national standard (Metal tensile test method). Then, tensile tests were performed at 750°C with a strain rate of 10^{-3} s^{-1} using an electronic universal testing machine (Instron 3351). The microstructures before and after tensile tests were characterized via scanning electron microscopy (SEM, Zeiss Sigma 300) using backscattered-electron (BSE) mode, equipped with an electron backscattering diffraction (EBSD) system. The collected EBSD data were post-treated using Channel

5TM software. To clarify the deformation mechanisms, thin foils were cut perpendicular to the tensile loading axis via electron discharge machining after tension tests and then investigated via TEM and HRTEM using a field-emission transmission electron microscope (FEI Talos F200X) operating at 200 kV. Thin slices were first ground to $\sim 60 \mu\text{m}$ in thickness and twin-jet electropolished using a solution consisting of 60% methanol, 35% butanol, and 5% perchloric acid at -20°C under 30 V.

Figure 1 shows the tensile true stress-strain curves of LMs with various LSs at 750°C. The nano-LMs exhibit YS and ultimate tensile strength (UTS) up to $\sim 835 \text{ MPa}$ and $\sim 1010 \text{ MPa}$, as shown in Figure 1(a). Such a high strength surpasses all of the reported TiAl alloys and is comparable to the Ni-Co based superalloy (TMW) which is generally used in engine disks [18–35], indicating the superiority of extremely fine LM, as shown in Figure 1(b). LMs with LSs of ~ 15 and $\sim 20 \text{ nm}$ exhibit similar deformation behavior and almost the same YS/UTS and ductility. Interestingly, increasing LS from 20 to 35 nm results in a dramatic enhancement of ductility without compromising YS. In particular, LM₃ exhibits a comparable YS with LM₁ and a four-fold increase in plasticity. One should note that this phenomenon contradicts the Hall-Petch relationship. The corresponding work-hardening rates of LM₁ and LM₂ in Figure 1(c) decrease dramatically until catastrophic failure. In contrast, the work-hardening rates of LM₃ and LM₄ can be divided into two stages: In stage I, the non-linear work-hardening rate declines rapidly with the true strain, followed by stage II, in which the work hardening rate slows down with the true strain. The transition from stage I to stage II indicates an increase in accumulation of defect density during deformation, which retards the rapid decline of work hardening rate. The fracture morphologies of LM₁ and LM₃ in Figure 1(d,e) present cleavage planes and numerous dimples, indicating brittle and ductile fractures, respectively.

To clarify the underlying reason for the dramatic improvement in ductility when LS changes from ~ 20 to $\sim 35 \text{ nm}$, the microstructures of LM₁ and LM₃ were investigated before and after tensile tests using EBSD and TEM, respectively. The Euler orientation maps shown in Figure 2(a,b) indicate that the crystallographic orientation distribution of the lamellar colonies is almost random, and the average lamellar colony sizes of LM₁ and LM₃ are ~ 41.6 and $\sim 60.5 \mu\text{m}$, respectively. Thus, prolonging soaking time results in an increase in the average size of the α_2/γ lamellar colonies. Moreover, the bright-field (BF)-TEM images in Figure 2(c,d) indicate that the LSs of LM₁ and LM₃ are ~ 20.1 and $\sim 35.9 \text{ nm}$, respectively, and their maximum thicknesses are ~ 35 and $\sim 65 \text{ nm}$, respectively. The corresponding selected area

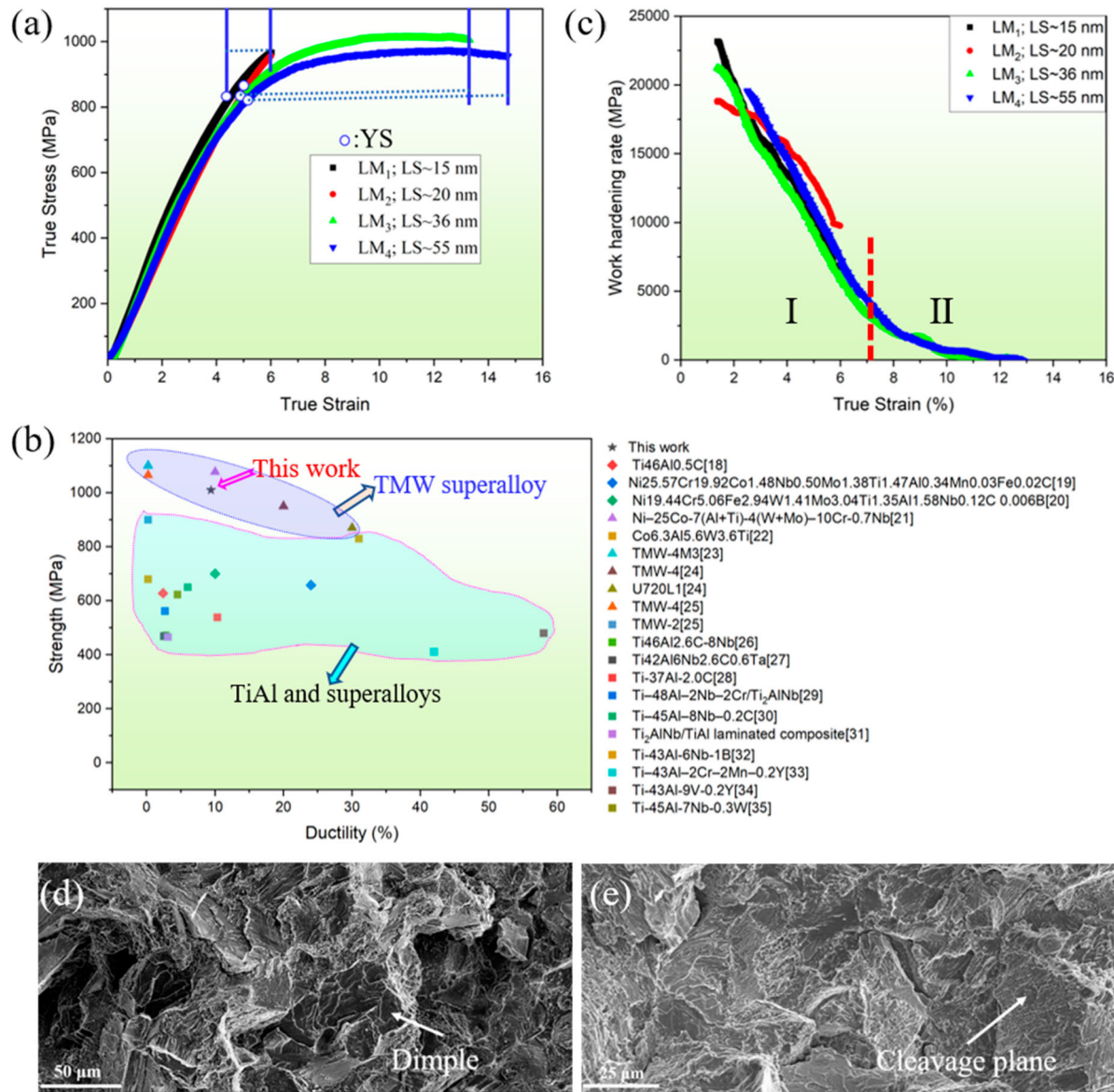


Figure 1. Tensile properties and fracture morphology of LMs with various LS at 750°C: (a) Tensile true strain–stress curves; (b) Comparison of strength and ductility between the designed TNM alloy with LM₃ structure and other alloys used at elevated temperature, including TiAl alloys, Ni-based, Co-based and TMW superalloys [18–35]. (c) Work-hardening rate curves. (d) and (e) fracture morphology of LM₁ and LM₃, respectively.

electron diffraction (SAED) patterns of LM₁ and LM₃ reveal the Blackburn orientation relationship between α_2/γ lamellae and γ twin symmetry. The parallel zone axis of $\langle 01\bar{1} \rangle_{\gamma} // \langle 1\bar{1}0 \rangle_{\gamma}$ also indicates the existence of a 120° rotational boundary. In fact, three types of γ/γ twist grain boundaries with different misorientations exist in the form of twins, namely, true twin (misorientation 180°), rotational boundary (120°) and pseudo-twin (60° misorientation), due to the tetragonality of the L1₀ structure [10].

According to the Hall–Petch effect, the YS of LM₃ should be less than that of LM₁. It is counterintuitive that a four-fold increase in ductility can be obtained without

compromising YS. The inability of Hall–Petch effect in regulating the YS of nano-LM indicates that different mechanisms dictate the tensile deformation. Extensive TEM observations were carried out for deformed LM₁ and LM₃. Herein, BF-TEM images indicate that no dislocations or twins operate during tensile deformation of LM₁, as shown in Figure 3(a). Thus, dislocation and twin multiplication are confined by nano-scale geometries, while plastic deformation occurs via other mechanisms. Moreover, the HRTEM image of deformed LM₁ in Figure 3(b) reveals step-like characteristics along γ/γ and α_2/γ interfaces. The high-magnification HRTEM images of A and B regions illustrate the occurrence of SFs,

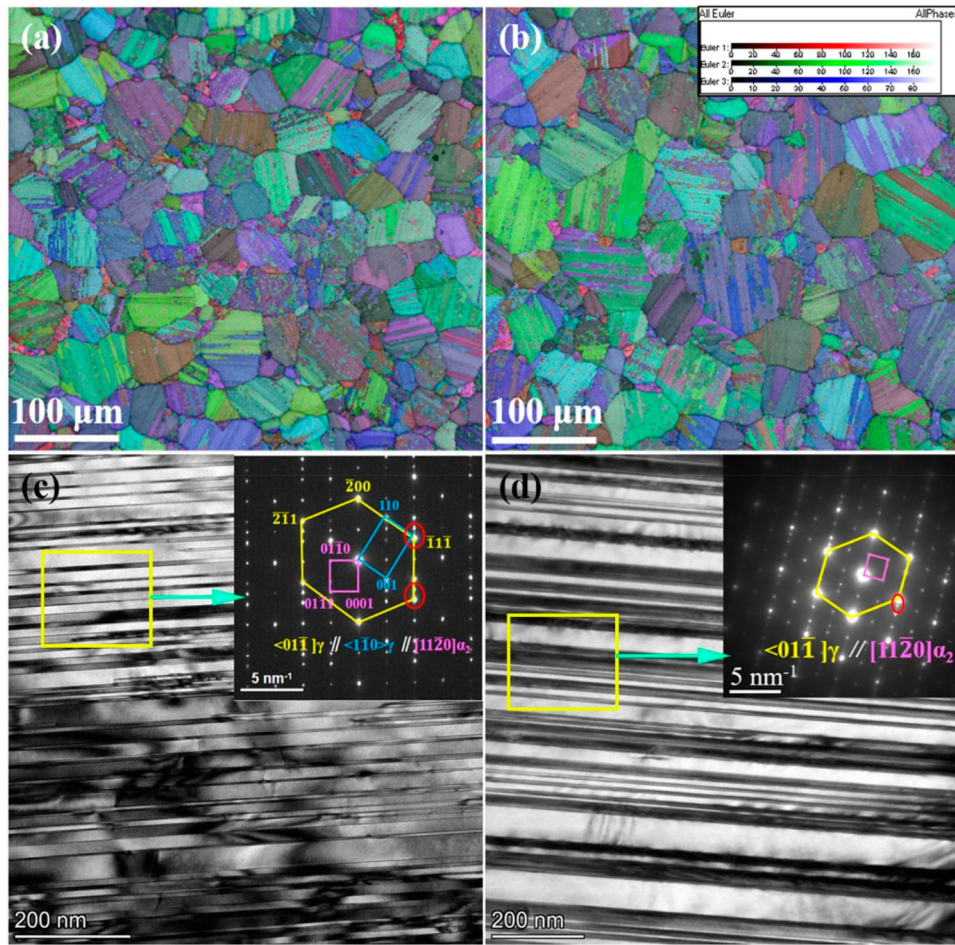


Figure 2. Microstructural details of LM₁ and LM₃: (a) and (b) Euler orientation maps superimposed on the BC images of two LMs, where all results are attained with a step size of 0.15 μm; The Euler angle-based color scale attached in (b) shows that similar colors indicate similar orientations; (c) and (d) BF-TEM images of LM₁ and LM₃, where the insets show SAED patterns, elaborating Blackburn orientation relationship between α_2/γ lamellae and γ twin symmetry.

as presented in Figure 3(c,d). Therefore, the development of SFs along the lamellar boundaries governs the plastic deformation of LM₁.

Furthermore, numerous BF-TEM observations also indicate that dislocation slipping and mechanical twinning failed to control the deformation of LM₃. Different from LM₁, fine blade-shaped bundles that are perpendicular to the lamellar interface are observed in deformed LM₃, as shown in Figure 4(a). The corresponding HRTEM image presents an LPSO structure, as shown in Figure 4(b). The inverse fast Fourier transform (FFT) pattern in Figure 4(d) shows extra Bragg diffraction spots at $1/3(\bar{1}\bar{1}1)\gamma$ and $2/3(\bar{1}\bar{1}1)\gamma$, confirming the formation of 9R-LPSO structure. The 9R structure is composed of a repeated unit with the periodicity of three adjacent $\{111\}$ planes, and the stacking sequence is ABC/BCA/CAB/A..., as presented in Figure 4(c).

Planar deformation and multiple deformation bands can be identified along the lamellar boundary, as

indicated in Figure 4(e,f). The high-magnification Region-D presents atoms stacking order of... ABC/BAC/A..., whereas the corresponding FFT pattern indicates that the angle between (006) and (100) diffraction planes is 90°. Moreover, five spots can be clearly identified between the transmission spot and (006) spot, demonstrating the 6H-LPSO structure with six stacking layers. Owing to the strong strain field, HRTEM image shows a complex and ambiguous atomic stacking order of LPSO bands in some regions. There may exist other types of LPSO structures, such as 9R. Therefore, the planar deformation features of LM₃ contain abundant nanometer-sized LPSO bands containing a combination of 9R and 6H. That is to say, the multiple LPSO bands accommodate the plastic deformation of LM.

The formation of both SFs and LPSO requires the gliding of $1/6[11\bar{2}]$ partial dislocations on $\{111\}$ planes [5,36]. Therefore, slipping of $1/6[11\bar{2}]$ interfacial partial dislocations along the lamellar interface governs the

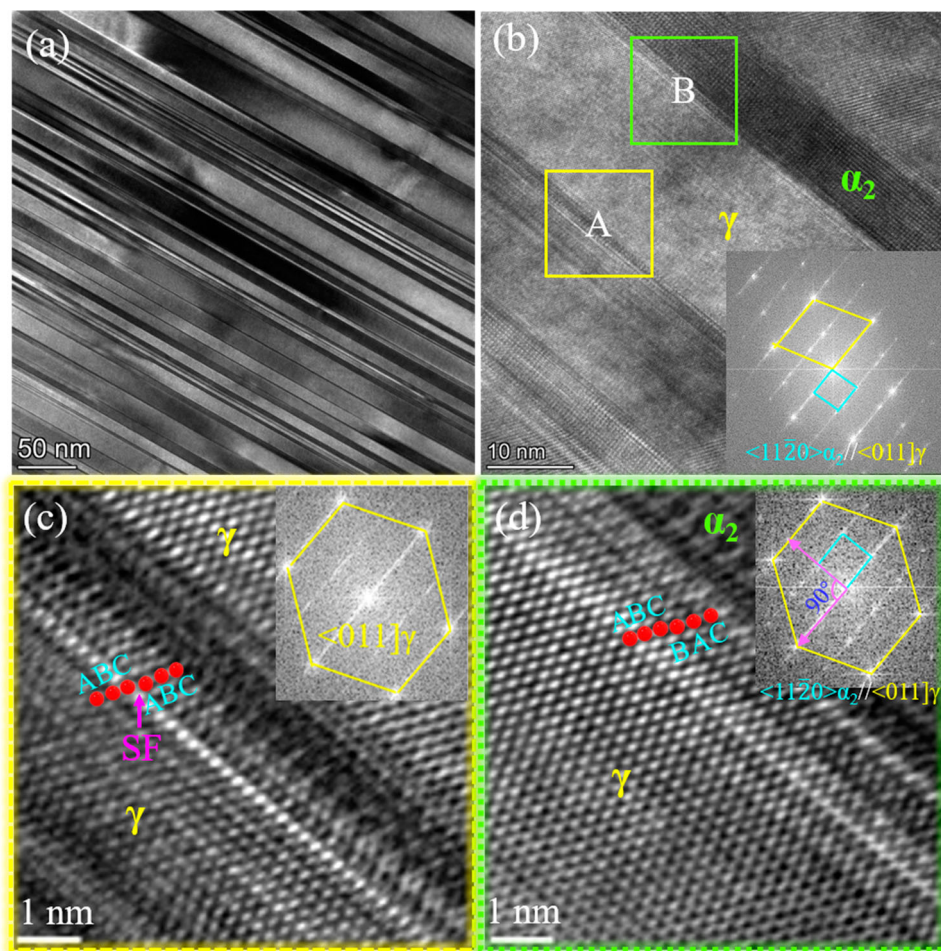


Figure 3. Deformation mechanism of LM₁: (a) BF-TEM image indicates that dislocations and twins do not develop during the tensile process; (b) HRTEM image of deformed LM₁; and (c) high-magnification HRTEM images of (b) Area-A and (d) Area-B.

plastic deformation of nano-LM. The movement of interfacial partial dislocations is extremely difficult due to the high SF energy and they can only occur along lamellar interfaces under the aid of thermal activation [37]. As a consequence, the nanoscale LM shows a much higher YS than coarse LM, which is deformed by dislocation gliding. This work reveals that sliding of $1/6[11\bar{2}]$ interfacial partial dislocations along the $\{111\}$ planes of lamellar interface is strongly dependent on LS and results in two structures. Firstly, the shearing of partial dislocations along a layer of atoms leads to the occurrence of SFs, as demonstrated in the case of LM₁ [10]. Secondly, the periodic sliding of interfacial partial dislocations on $(111)\gamma$ planes alternatively results in the LPSO structures, as shown in LM₃. Moreover, when the pileup of interfacial dislocations cannot be effectively dissipated, they will be emitted from the interfaces into lamellae to relieve the local stress concentration, resulting in an LPSO structure perpendicular to the lamellar interface, as illustrated by the formed blade-shaped bundles in LM₃. The underlying reasons for such a distinctive deformation mechanism with an increase in the LS from ~ 20 to ~ 35 nm

require further exploration. These two different deformation mechanisms are certainly associated with the failure of Hall–Petch effect in regulating the YS of LM.

It is worth noting that increase in LS converts the deformation mechanism from SFs-dominated process to numerous LPSO bands-mediated process, indicating easier interfacial partial dislocation sliding along LM₃, which makes lower YS of LM₃ than LM₁. However, the opposite trend is observed in this work, which can be attributed to the following reasons:

- (1) The formation of large-area LPSO bands is a highly efficient process for developing and, most importantly, prolonging strain hardening to restrict the localization of deformation. The outstanding strain hardening capacity increases the strength and delays the onset of necking, which contributes to a larger elongation. Moreover, the formation of LPSO structures perpendicular to the interface can also relieve part of the accumulated stress at the interface, effectively preventing the premature failure of the material.

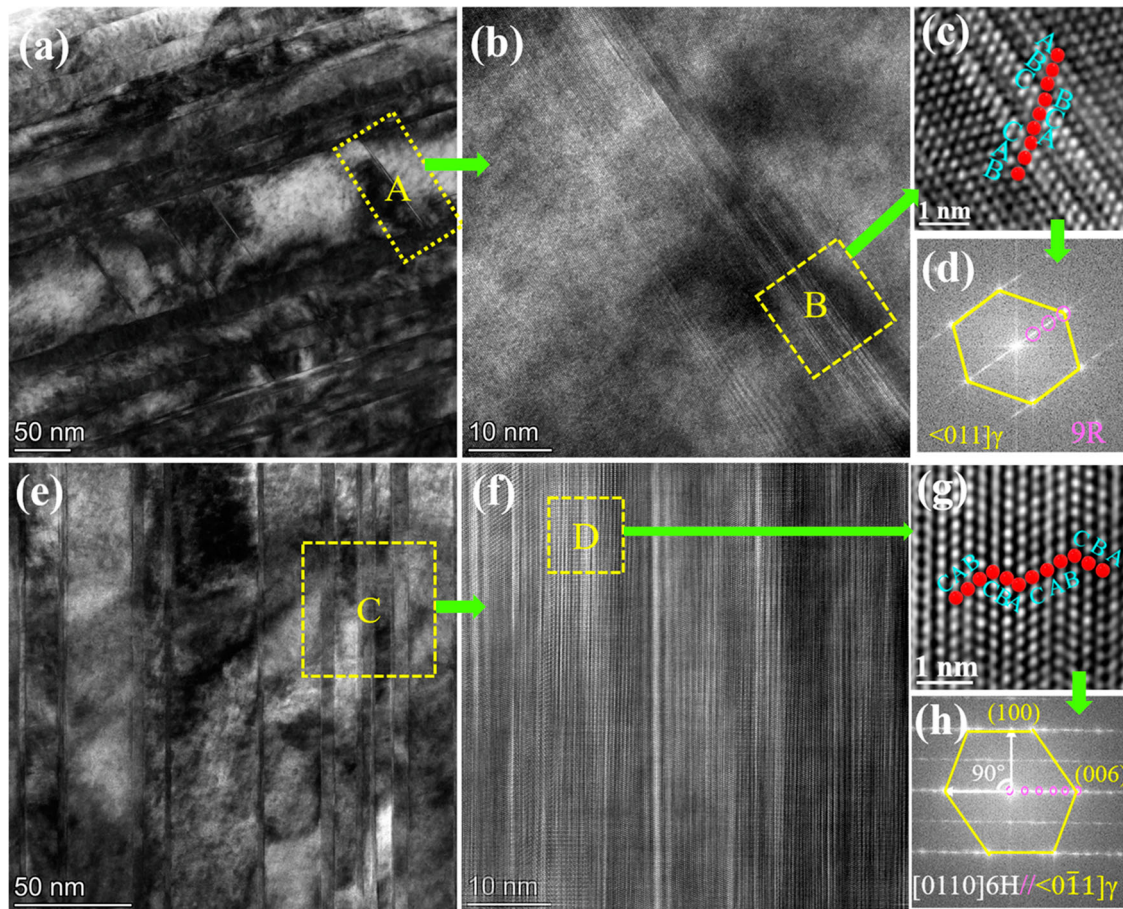


Figure 4. Deformation mechanism of LM₃: (a) BF-TEM image exhibits fine bundles perpendicular to the lamellar interface; (b) HRTEM image of the Area-A in (a); (c) high-magnification HRTEM image of the Area-B indicates a 9R stacking sequence; (d) FFT pattern of (b); (e) BF-TEM image shows the deformed microstructure of HT₃-ed alloys without dislocations or twins; (f) HRTEM shows the multiple nanometer-sized LPSO bands; (g) high-magnification HRTEM image of the Area-B revealing a 6H-LPSO stacking sequence; and (h) FFT pattern reveals 6H-LPSO diffraction spots.

- (2) The formation of LPSOs is a local phase transformation process. Numerous LPSO bands cause prominent TRIP effect, which can simultaneously improve the strength and ductility [38,39].

Overall, the current work indicates that dislocation slipping and mechanical twinning are confined during plastic deformation when LS is reduced to the nanometer scale, and plastic deformation proceeds via interfacial partial dislocation gliding along lamellar interfaces. A slight increase in LS of LM from ~ 20 to ~ 36 nm causes SFs- and numerous LPSOs-accommodated deformation, respectively. The formation of large-area LPSO structures triggers the TRIP effect and causes a prominent strain hardening rate, resulting in a dramatic enhancement of ductility without compromising yield strength compared to fine LM₁ deformed by SFs at 750°C. These findings provide insights into the development of advanced TiAl alloys with comprehensive high-temperature mechanical performance.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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