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ORIGINAL REPORT



Microtwinning avalanches induced the Protevin–Le Châtelier effect in PWA1483 at 750 °C

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ABSTRACT

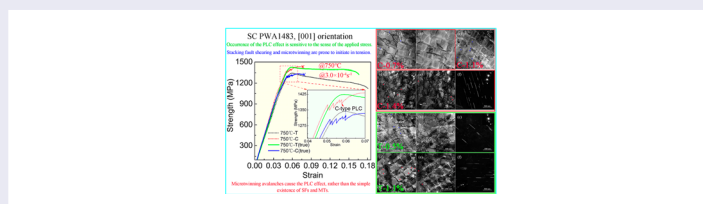
The flow behavior and deformation mechanisms of the [001]-oriented single-crystal nickel-based superalloy PWA1483 are investigated under quasi-static compressive and tensile loading conditions at 750°C. We found that the tensile yield strength is larger as compared to the compressive strength, and the serrated flow only appears on the compressive stress–strain curves. Microstructural observations uncover that stacking fault shearing and microtwinning are prone to initiate in PWA1483 during tensile deformation, and the appearance of the Protevin–Le Châtelier effect in compression derives from the abrupt frequent bursts of the two mechanisms on a mesoscale, rather than the simple occurrence of these procedures.

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KEYWORDS

Nickel-based superalloy; Protevin–Le Châtelier effect; tension/compression asymmetry; stacking faults; microtwins



1. Introduction

As one kind of important high-temperature structural materials, single-crystal (SC) nickel-based superalloys have been widely used in the key components of advanced aircraft engines and industrial gas turbines attributed to their excellent combination of good mechanical properties and corrosion resistance at high temperatures [1,2]. Generally, over 10 alloying elements have been added to these alloys, giving rise to the unique γ/γ' microstructure and therefore conferring many interesting mechanical response on these alloys during plastic deformation, including the anomalous yield behavior, orientation-dependent strength, Protevin–Le

Châtelier (PLC) effect and tension–compression asymmetry during plastic deformation [1,2].

Originally, it was generally accepted that the ‘cross-slip’ and ‘core width’ effects accounted for the larger tensile yield strength as compared to the compressive yield strength for [001]-orientated SC nickel-based superalloys [3–7]. However, in later work, Sondhi et al. [8] ascribed the tension/compression creep asymmetry in IN100 to the initial internal stress. Moreover, recently, it has been widely reported that the activation of deformation modes in SC nickel-based superalloys was sensitive to the sign of the applied stress, which in turn explained the tension/compression asymmetry of these superalloys

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[9–14]. For instance, Yamashita and Kakehi [11] pointed out that, for the SC nickel-based superalloy PWA1480 oriented near the [001] direction, stacking fault shearing and microtwinning were prone to occur as compared to anti-phase boundary (APB) shearing during compressive deformation, and thus the tensile yield strength was larger.

With respect to the PLC effect, previous researchers are also at variance about the microscopic mechanisms conferring the phenomenon in nickel-based superalloys [15–27]. Traditionally, the PLC effect in nickel-based superalloys was attributed to the dynamic interaction of diffusing solute atoms and mobile dislocations, i.e. dynamic strain aging (DSA) [17,20,21]. Later, Cai et al. [19] proposed that the C-type serrations on the stress–strain curves of nickel-based superalloys were associated with the second-phase particles, as initially reported in Al-based alloys [15,16]. In contrast to these findings, Cui et al. [18] reported that a high density of stacking faults (SFs) gave rise to the appearance of the C-type PLC effect in the Ni-Co-based superalloy TMW-2. Subsequently, Zhang et al. [26] and Huang et al. [27] expanded this theory and concluded that apart from SFs, the existence of microtwins (MTs) also played a vital role in the occurrence of the C-type serrations in these superalloys.

In this paper, we report the flow behavior of the SC nickel-based superalloy PWA1483 oriented near the [001] direction under quasi-static compressive and tensile loading at 750 °C. Particularly, the work focuses on the analysis of the influence of the sense of the applied stress on the deformation modes and their possible consequences on the PLC effect and tension–compression asymmetry.

2. Materials and methods

The nominal chemical composition of the experimental alloy PWA1483 is given in Table 1. SC rods, which had a diameter of around 14 mm and a length of 210 mm, were grown along the $\langle 001 \rangle$ direction at a constant withdrawal rate of 300 mm h^{−1} using an ALD Vacuum Technologies furnace. All the rods were subjected to a standard heat treatment (SHT), which consisted of a two-step solution treatment, 1204°C/1 h + 1265°C/1 h/air cooling (AC) and a coating heat treatment, 1080°C/4 h/AC [28]. After SHT, the misorientation of each bar was evaluated using a Zeiss Gemini 500 field emission scanning electron microscope equipped with a symmetry electron backscattered diffraction (EBSD) detector from Oxford Instruments. As demonstrated in Figure 1(a), the deviation angle from the ideal [001] direction is smaller than 5° for one of these bars. Then, all the specimens

Table 1. The nominal chemical composition of the experimental alloy (wt.%).

Alloy	Cr	Co	W	Ta	Mo	Al	Ti	C	Ni
PWA1483	12.2	9.0	3.8	5.0	1.9	3.6	4.1	0.07	Bal.

were sectioned from the bar using a wire-cut electric discharge machine along the growth direction. Meanwhile, as demonstrated in Figure 1(b), the SHT results in a bimodal size distribution of γ' precipitates for the experimental alloy. The average edge length of coarse γ' precipitates and the diameter of fine γ' precipitates are 292 ± 70 nm and 29 ± 12 nm, respectively. The volume fraction of γ' precipitates is $54.2 \pm 0.8\%$. Moreover, from this figure and Figure S-1 in Electronic Supplementary Materials, one can get that dislocations and planar faults, such as SFs and MTs, are rarely visible in the experimental alloy.

Threaded specimens, which had a diameter of 3.0 mm and a gauge length of 15.0 mm, and cylindrical samples, which had a diameter of 5.0 mm and a height of 12.5 mm, were separately machined from the heat-treated bar. Quasi-static plastic deformation in tension and compression was performed on an MTS 793 testing machine at 750 °C. All the tests were carried out at a nominal strain rate of 3.0×10^{-4} s^{−1}. Besides the highly strained and fracture tests, some tests were also interrupted at some typical points on the stress–strain curves to clarify whether the operative deformation mechanisms would evolve with proceeding plastic deformation in the experimental alloy using a transmission electron microscope (TEM).

Thin slices with the normal parallel to the $\langle 001 \rangle$ direction were cut from the heat-treated samples by a wire-cut electric discharge machine. Meanwhile, some slices were also extracted from the gauge sections of the deformed and fracture specimens along the desired crystallographic orientation to fully characterize the deformation-induced microstructures. After mechanically thinned to a thickness of around 50 μ m, thin lamellae of 3 mm in diameter were electropolished in a solution of C₂H₆O with 10.0 vol.% HClO₄ at around −30 °C. Conventional microstructural observations were performed on a JEM-2100Plus TEM at 200 kV. Additionally, atomic resolution high-angle annular dark-field (HAADF) imaging of planar faults was performed using an aberration-corrected JEM-ARM200F NeoARM TEM at 200 kV.

3. Results and discussion

Figure 1(c) shows the engineering stress–strain curves of the experimental alloy under quasi-static compressive

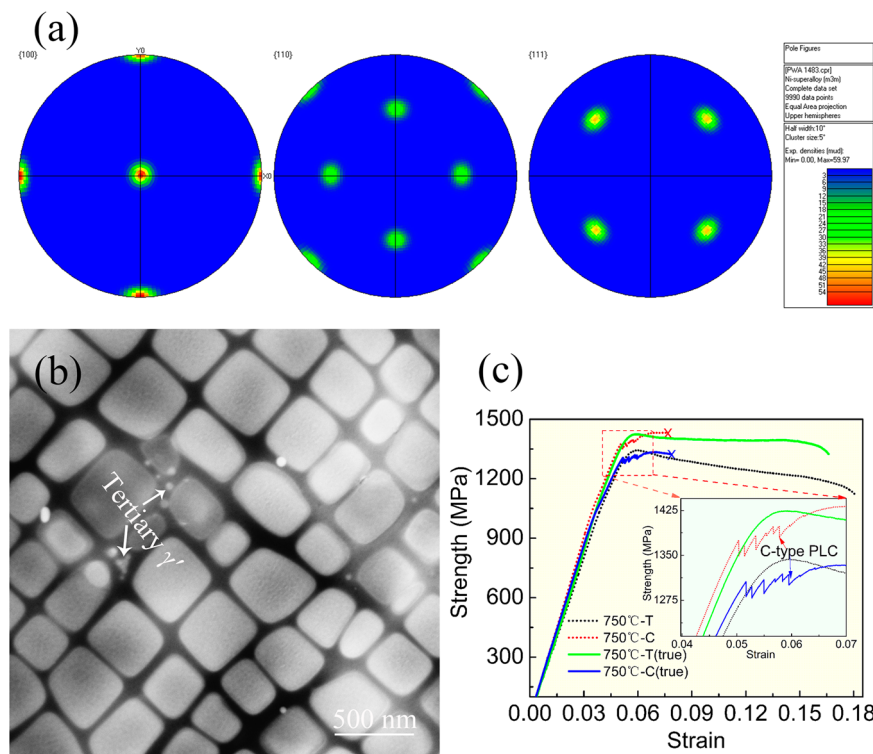


Figure 1. (a) EBSD figure showing the deviating angle between the growth direction of the bar and [001] direction; (b) high-angle annular dark-field image illustrating the γ/γ' microstructure in the experimental alloy after SHT; (c) engineering stress–strain curves of the experimental alloy at 750°C, together with the true stress–strain curves. The marks indicate that the tests were interrupted.

(750°C-C) and tensile loading (750°C-T) conditions at 750°C, together with the true stress–strain curves obtained following the procedures reported in Ref. [29]. In the elastic region, the stress–strain curves coincide in compression and tension. However, once the plastic deformation initiates, remarkable divergences appear on the curves, and a higher flow stress is obtained for the experimental alloy in tension. The tensile yield strength $\sigma_{0.2}$ is 1210 ± 40 MPa, which is almost 100 MPa larger than the compressive yield strength. Meanwhile, from this figure, one can also get that, as reported in Refs. [20,22], the C-type serrations start to appear on the stress–strain curve after around 1.4% plastic strain in compression, whereas such a phenomenon is invisible on the whole tensile stress–strain curve. Evidently, the DSA, which has been widely accepted as the underlying mechanism of plastic instability of numerous alloys [17,18], cannot lead to the appearance of the jerky flow for the experimental alloy. For this reason, the deformation-induced microstructures are investigated in detail in the following sections.

Figure 2(a,b) illustrates the typical microstructures in the experimental alloy after 0.7% compressive plastic strain at 750°C. A few superlattice intrinsic/extrinsic stacking faults (SISFs/SESFs) and numerous dislocation pairs are generated in the experimental alloy. This

demonstrates that APB shearing governs the plastic deformation, although SF shearing takes place occasionally during plastic deformation. A close inspection of these dislocation pairs reveals that cross-slip of some dislocation pairs from octahedral planes to cube planes has occurred, as indicated by the blue arrowheads in Figure 2(a). This is in strong contrast to previous results found in other SC nickel-based superalloys in Refs. [30–37], which showed that the plastic deformation of these superalloys was achieved mainly by SF shearing and microtwinning at intermediate temperatures. The apparent inconsistency might be explained by noting that the operative deformation mechanism in nickel-based superalloys evolved with proceeding deformation [38,39]. Then, after around 1.1% (before the appearance of the C-type serrations), 1.4% (after the C-type serrations have occurred) and 4.0% plastic strain, the microstructures in the experimental alloy are also investigated. Indeed, as plastic deformation continues, the operative deformation mechanism changes in the experimental alloy. After the C-type serrations occurred, a high density of SISFs/SESFs and MTs was generated in the γ' precipitates, apart from considerable dislocation pairs, as demonstrated in Figure 2(d)–(i). The changing tendency is distinctly different from those found in nickel-based superalloys in Refs. [38,40], which showed that

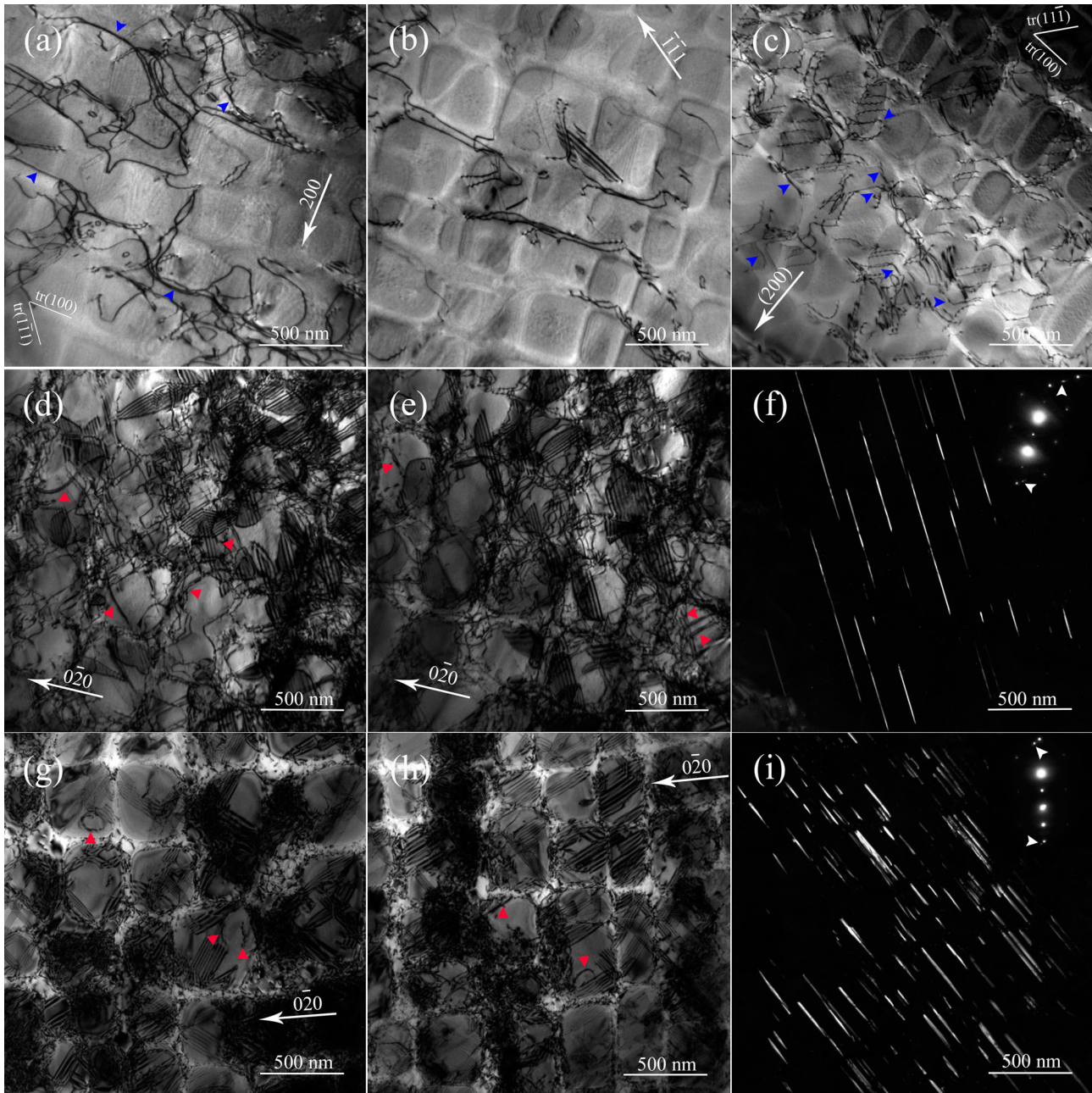


Figure 2. TEM images displaying the typical microstructures in the experimental alloy after various compressive plastic strain at 750°C: (a) 0.7% plastic strain, dislocation pairs; (b) 0.7% plastic strain, dislocation pairs and extended stacking faults; (c) 1.1% plastic strain, dislocation pairs; (d)–(f), 1.4% plastic strain; (g)–(i) 4.0% plastic strain; (d,e) and (g,h) dislocation pairs and isolated SISFs/SESFs; (f) and (i) MTs. The blue arrowheads indicate the cube cross-slip of dislocation pairs within γ' precipitates. The red arrowheads indicate dislocation pairs. The inset figures on the upper right corner of (f) and (i) are the selected area diffraction (SAD) patterns of MTs, and the white arrowheads indicate the spots of MTs.

the transform in the deformation mechanism from SF shearing to APB shearing usually occurred during plastic deformation arising from producing a high density of dislocations at the two-phase interface and therefore losing the coherent two-phase interface with strain. Note that, although some SISFs/SESFs are occasionally observed in the experimental alloy undergoing 1.1% compressive plastic strain (see Electronic Supplementary Figure S-2),

APB shearing is still the dominant deformation mechanism, as demonstrated in Figure 2(c). This result seems to demonstrate that the occurrence of the PLC effect is ascribed to the existence of SISFs/SESFs and MTs for PWA1483, as reported in Refs. [18,24,27].

Figure 3(a)–(c) illustrates the typical microstructures in the experimental alloy after 0.5% tensile plastic strain at 750°C. Analogous to the deformation microstructures

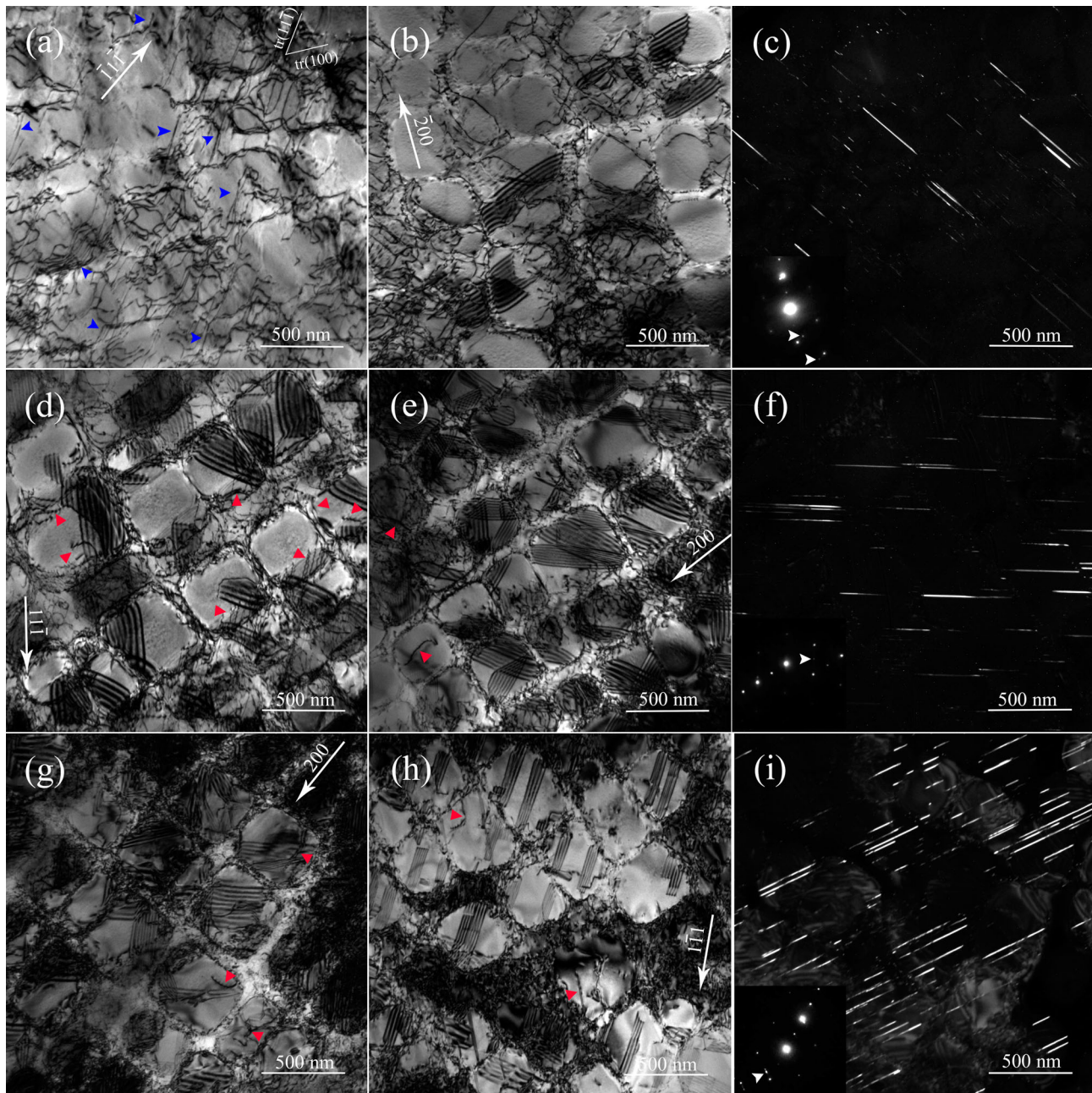


Figure 3. TEM images displaying the typical microstructures in the experimental alloy stretched to various tensile plastic strain at 750°C: (a)–(c), 0.5% plastic strain; (d)–(f), 1.1% plastic strain; (g)–(i) fracture; (a) dislocation pairs; (b) isolated SISFs/SEFs; (d,e) and (g,h) dislocation pairs and SISFs/SEFs; (c), (f) and (i) MTs. The blue arrowheads indicate the cube cross-slip of dislocation pairs within γ' precipitates. The red arrowheads indicate dislocation pairs. The inset figures on the bottom-left corner of (c), (f) and (i) are the SAD patterns of MTs, and the white arrowheads indicate the spots of MTs.

created in the experimental alloy undergoing slight plastic deformation in compression, numerous APB-coupled dislocation pairs are observed, suggesting that APB shearing controls the initial tensile plastic deformation. However, as compared to the deformation patterns in compression, cube cross-slip of APB-coupled dislocation pairs is more frequently observed, which is consistent with the results found in some SC nickel-based

superalloys and $\text{Ni}_3(\text{Al}, \text{Nb})$ SCs oriented the [001] direction and might partly induce the higher tensile yield strength [3–5,7]. Meanwhile, apart from these substructures, it is interesting to note that profuse SFs and MTs are also formed, and the content of SISFs/SEFs and MTs within γ' precipitates is much higher as compared to that in compression, as demonstrated in Figure 3(b,c). This indicates that the two mechanisms are easier to

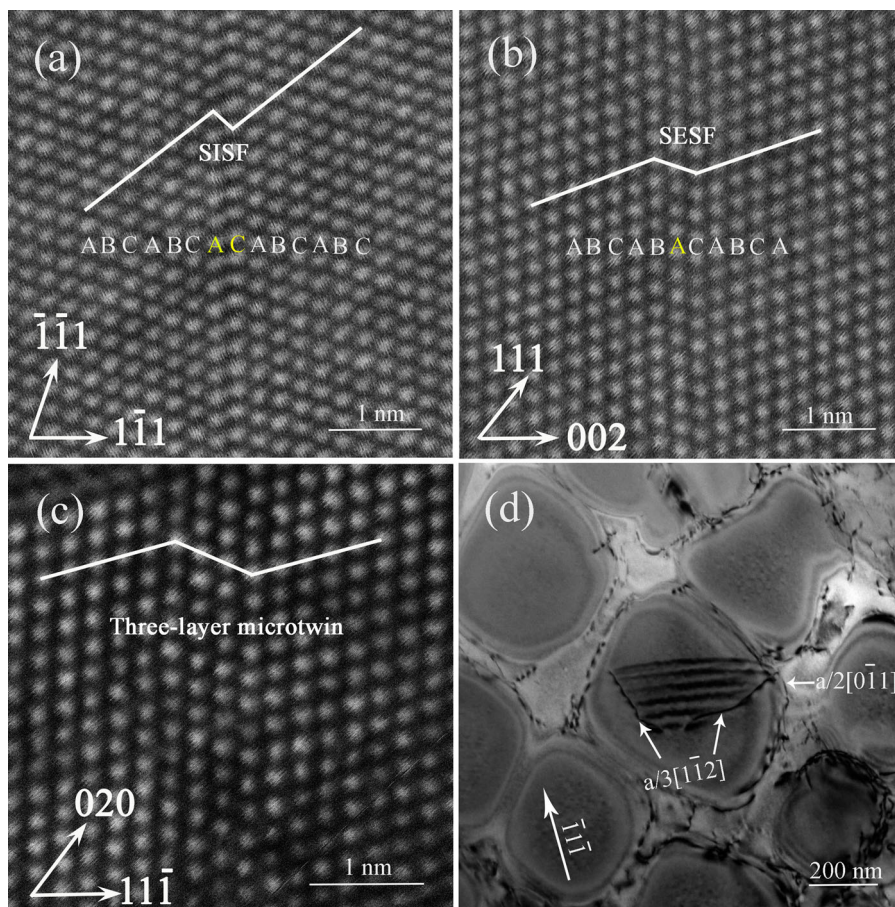


Figure 4. (a)–(c) HAADF images showing SISFs, SESFs and an MT in the experimental alloy after 1.1% tensile plastic strain at 750°C: (a) SISF; (b) SESF; (c) a three-layer MT; (d) TEM bright-field image showing one isolated SESFs enclosed by $a/3 \langle 112 \rangle$ partial dislocations and connected by one single matrix dislocation.

operate in tension, and the operation of the two mechanisms cannot cause a decrease in the yield strength of the experimental alloy. On the other hand, after around 1.1% plastic deformation and fracture, the microstructures are also analyzed. As displayed in Figure 3(d)–(i), many dislocation pairs as well as a higher density of SISFs/SESFs and MTs are visible in these specimens, further corroborating the above statement. Moreover, in order to observe SISFs/SESFs and twin lamellae more clearly, atomic resolution HAADF micrographs of these faults are obtained on the $\langle 011 \rangle$ axis. As shown in Figure 4(a)–(c), there are separately one SISF and one SESF as well as a three-layer MT. Then, following the procedures reported in Refs. [41–43], rigorous diffraction contrast analyses were conducted using TEM under different two-beam conditions. Different from those previously reported in the turbine disk superalloys [18,44–46] and some blade superalloys [36,47–49], it is found that isolated SISFs/SESFs are usually connected by single matrix dislocations, and the partial dislocations, which lead to the creation of these faults within γ' particles,

are the $a/3 \langle 112 \rangle$ type, as illustrated in Figure 4(d) (For more details, please refer to Electronic Supplementary Figure S-3). Such a pattern was also found in other nickel-based superalloys after plastic deformation at low and intermediate temperatures [9,41,42,50–52]. Thus, it is believed that the formation process of SISFs/SESFs in the experimental alloy follows the same schedule. With respect to the microtwinning process, it is not clear yet. Nevertheless, given the fact that the partials, which give rise to SISFs/SESFs, are the $a/3 \langle 112 \rangle$ type, and SISFs, SESFs and MTs are observed in the same specimens (see Figure 4(a)–(c)), it is believed that the successive movement of the $a/3 \langle 112 \rangle$ partial dislocations on adjacent 111 planes causes the formation of MTs [10,53,54].

Then, a natural question arises as to why SF shearing and microtwinning are prone to initiate in PWA 1483 in tension. In fact, the phenomenon can be interpreted in the terms of the detailed formation procedures of the two planar faults. As stated above, due to the low stacking fault energy of the matrix, the single perfect dislocation $a/2 \langle 011 \rangle$ is inclined to dissociate into two

Shockley partials on the 111 planes to decrease the self-energy [55–57], which can also be demonstrated in the Electronic Supplementary Figure S-4. So, the two Shockley partials must first constrict into a perfect dislocation before it can decompose into one $a/3 \langle 112 \rangle$ partial dislocation and one $a/6 \langle 211 \rangle$ partial dislocation [9,41,50,53,56]. For the [001]-oriented SC nickel-based superalloys, the recombination of the two Shockley partials was promoted by the resolved shear stress pushing them together against their repulsive interaction during tensile deformation [3,4,6,11,12,58], and for this reason, it is favorable for the latter process to occur. But, in compression, the opposite is true. Thus, these two kinds of planar faults are more frequently observed in the specimens after slight tensile plastic deformation. Note that, the latter decomposition process of single matrix dislocations is prone to take place in compression as compared to in tension [13,14]. Thus, the present experimental results seem to indicate that the former constriction process of the two partials plays a more important role than the subsequent decomposition process at the two-phase interfaces in the initiation of the two processes for PWA1483 at 750°C.

With respect to the PLC, someone might argue that since the serration flow does not appear on the tensile stress–strain curve, but SF shearing and microtwinning take place frequently during tensile deformation, seeming to suggest that the occurrence of the C-type serrations in PWA1483 is not associated with the two processes as well as the Suzuki segregation, as reported in other superalloys [24,59,60]. Actually, the phenomenon might be understood in light of the evolution of the deformation mechanisms with plastic strain and the critical resolved shear stresses (CRSSs) required for these deformation mechanisms to initiate. As demonstrated in Figure 3, SF shearing and microtwinning launch at the very beginning of plastic deformation during tensile deformation, and the frequencies of these procedures increase gradually with strain. Moreover, the CRSS needed for APB shearing to initiate is much higher than those needed for the two processes to operate when shearing of γ' particles is accomplished by $a/3 \langle 112 \rangle$ dislocations (For more details, please see the Electronic Supplementary.) [53,54]. Thus, the gradual increase in the frequencies of these two processes with strain would not induce a considerable drop in the flow stress, and for this reason, the serrated yielding is rarely observed in tension. With respect to the compressive deformation, only when the compressive plastic strain reaches around 1.4%, SF shearing and microtwinning start to operate actively. The instantaneous, frequent synchronisation of the two processes decreases the stress required for plastic deformation to proceed. Thus, the serrated flow on the stress–strain

curve appears within a certain strain range. However, as plastic deformation proceeds, it might be difficult for SF shearing and microtwinning to occur again on the mesoscale simultaneously, since a high density of dislocations, SFs and MTs has been created. As a result, the jerky flow disappears on the stress–strain curves.

4. Conclusion

In summary, we found that: (i) as the plastic deformation proceeds, the dominant deformation mechanism in PWA1483 changes from APB shearing to SF shearing and microtwinning at 750 °C; (ii) the latter two procedures are easy to launch in the experimental alloy during tensile deformation, which is because the dislocations which result in the formation of these planar faults are the $a/3 \langle 112 \rangle$ type and the constriction process of two Shockley partials is dominant over the latter dissociated procedure of single matrix dislocations at the γ/γ' interfaces; (iii) the serrated flow is sensitive to the sense of the applied stress, and the simultaneous sudden bursts of SF shearing and microtwinning on a large scale are responsible for the occurrence of the PLC effect during plastic deformation, rather than the simple occurrence of these processes.

Disclosure statement

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

Data availability

The data that supports the findings of this study are available from the corresponding author, [P. Zhang], upon reasonable request.

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