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Pore-induced defects during thermo-mechanical fatigue of a fourth-generation single crystal superalloy

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ABSTRACT

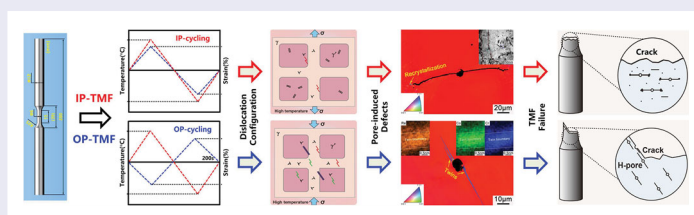
Comparable investigation of pore-induced defects during in-phase (IP) and out-of-phase (OP) thermo-mechanical fatigue (TMF) were conducted on fourth-generation single crystal superalloy. It was discovered that recrystallizations and deformation twins would take shape near micro-pores at IP and OP cycling, respectively. High-resolution observation uncovered that the increasing $a/6\langle 112 \rangle$ twinning dislocations and more frequent activation of $\langle 112 \rangle \{111\}$ viscous slipping during OP-TMF accounted for twinning nucleation. Atom-scale mapping additionally disclosed the dual effect of Re, Co, and Cr in facilitating twinning formation and impeding of partial dislocations movement. It was deemed that the accumulation of pore-induced twins caused the premature fracture of alloy during OP-TMF.

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KEYWORDS

Fourth-generation single crystal superalloy; micro-pores; deformation twins; element segregation; thermo-mechanical fatigue



IMPACT STATEMENT

This paper firstly uncovers the formation and evolution mechanisms of pore-induced defects during thermo-mechanical fatigue of fourth-generation single crystal superalloy under different phase angles.

1. Introduction

Decades of investigations and composition optimization have generated single crystal (SX) Ni-based superalloys, which are distinguished by the incorporation of satisfactory mechanical properties and oxidation resistance at elevated temperatures [1–3]. Up to now, the fourth-generation SX alloys with Ru addition, viz., ideal microstructural stabilizer, have gradually been manufactured as turbine blades of advanced aero-engines [4]. During the real service of SX turbine blades, the iterative start, acceleration, deceleration and stop of aero-engines would unavoidably give rise to the cyclic thermal and mechanical stress, thereby resulting in the potential thermo-mechanical fatigue (TMF) failure of the alloys [5,6].

In nature, the cycling modes of in-phase (IP) and out-of-phase (OP) are most commonly introduced in TMF process, which are basically referred to the cold-spots on turbine blades like *ektexine* and hot-spots like inner wall of the blade, respectively [7]. To date, the effect of phase angle on the fatigue behavior, crack initiation, fracture and damage mechanisms of 1st ~ 3rd generations SX superalloys have been basically clarified [8–13]. However, the constant development and application of advanced fourth-generation SX alloys would bring about two new challenges.

The first challenge lies in the rising inhomogeneity of chemical composition and dendritic segregation in Ru-containing SX alloys. Hence, the time and temperature of solution treatments have been markedly prolonged

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and elevated compared to 1st ~ 3rd generations SX alloys [4,14–16]. Therefore, the number of homogenization pores (H-pores) in the alloys would be dramatically increased. During fatigue deformation in various forms, the micro-pores could exert remarkable influence and limitation on the fatigue life of SX alloys [17–20]. More locally, it has been reported that the LCF/HCF/VHCF life of SX superalloys are more likely to be controlled by the large solidification pores (S-pores) in the alloy, while the smaller H-pores seem to exert a slight impact on fatigue behavior of the alloy [17,19,21,22]. To date, only a few investigations have reported the influence of micro-pores on the TMF behaviors of SX superalloys. Zhang et al. [23,24] discovered that deleterious deformation twins could be induced by pores during OP-TMF of TMS-series SX alloys. In their work, the considerable stress concentration in the vicinity of pores was regarded to account for the occurrence of twins [23,24]. Nevertheless, the detailed mechanisms for nucleation and propagation of those pore-induced defects have not been figured out. Additionally, the compared study of IP experiments is also required to elucidate the influence of phase angle on the formation of these defects. The second challenge rests with the reduced stacking fault energy (SFE) of fourth-generation SX alloys due to the partition of refractory elements (especially Re and Ru) into γ matrix [4]. This variation may promote the activation of $\langle 112 \rangle$ $\{111\}$ slip system and the formation of deformation twins during TMF process, in comparison to previous 1st ~ 3rd generations SX alloys [5,25]. Further, what is more serious is that the increasing pores are potential to serve as advantageous nucleation sites of twins, thereby exerting considerably adverse effects to the alloy during fatigue deformation.

Therefore, this work is focused on the formation and evolution of defects induced by micro-pores (especially H-pores) during the TMF of Ru-containing SX alloy. The TMF property of fourth-generation SX alloy was evaluated under both IP and OP conditions, while the characteristic of pore-induced defects and resultant effects on the fatigue performance were investigated.

2. Materials and methods

The fourth-generation SX superalloy developed by our research group was selected in this work, and the nominal composition of the experimental alloy in weight percentage was as follows: 5.8Al, 12.0Co, 5.4Re, 3.0Ru, 4.0Cr, 15.2(Mo + W + Ta), 0.15Hf and balance Ni. Only SX bars along the $[001]$ direction (within the deviation of 8°) were chosen to go through standard heat treatments: 1325°C/16 h + 1333°C/16 h, air cooling (AC); 1150°C/4 h, AC; 870°C/24 h, AC. Specimens with

the gage length and diameter of 15.0 and 6.0 mm were then machined and carefully polished along the axial direction. The required surface roughness of the specimen was $R_a = 0.2 \mu\text{m}$. IP- and OP-TMF experiments were conducted under the same mechanical strain amplitude of $\pm 0.6\%$ ($\Delta\epsilon_{\text{mech}}/2$), while the temperature scope was ranging from 600°C to 1000°C. The detailed schematic of specimens and testing conditions were displayed in Figure S1. More than two parallel specimens were prepared and applied for each testing condition. After TMF fracture, an optical microscope (OM) of LEICA DMi8 and a scanning electron microscope (SEM) of Tescan MIRA4 equipped with electron back-scattering diffraction (EBSD) detector were utilized for microstructure observation. The deformed structures were investigated through Talos F200X microscopy at 200 kV and aberration-corrected Titan³™ G2 60–300 microscopy with SuperX-energy dispersive spectrometer (EDS) detector at 300 kV. The specimens for transmission electron microscope (TEM) observation were prepared via the method of focused ion beam (FIB).

3. Results and discussion

Figure 1(a) displayed the heat-treated structure of the experimental alloy, the γ' phase with average size of approximately $0.29 \mu\text{m}$ and uniform distribution was observed. After IP-TMF failure, the fracture surface was macroscopically flat and the fatigue cracks were basically perpendicular to stress axis, as shown in Figure 1(b). However, the undulating plane as well as crystallographic fracture was observed when the specimen was subjected to OP cycling, as seen in Figure 1(c). The insets of macroscopic fracture from cross-sectional view were coincident with the above OM observations. Notably, the inner fatigue cracks were generated from both H-pores and the accumulation of tiny deformation pores (D-pores) under IP cycling, however, only H-pore-induced cracks were observed at OP condition. As for fatigue behavior, the alloy exhibited consecutive cyclic softening and hardening at high-temperature and low-temperature half cycles throughout the entire IP-TMF process. However, the cyclic saturation stage was interrupted due to the early fracture of specimen at OP condition (Figure 1(d)). From the evolution of hysteresis loops in Figure 1(e,f), it was noticed that these loops were almost closed at different stages during IP-TMF. But, the hysteretic energy (especially during high-temperature half cycles) had increased when approaching fatigue failure under OP cycling. The above observation had collectively manifested that during OP-TMF, the propagation of the main crack would be accelerated along the specific crystallographic

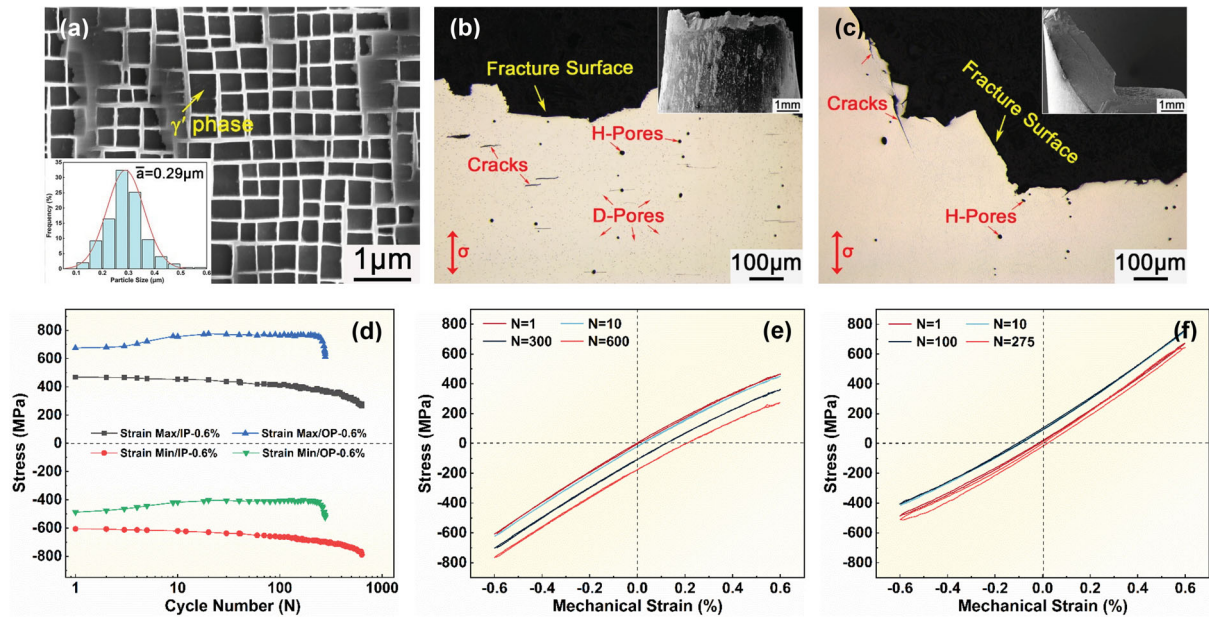


Figure 1. (a) γ/γ' -structure and distribution of γ' -size (in-set image), cross-sectional view of fracture surfaces and macroscopic fracture morphology (in-set images) after TMF failure at (b) IP-TMF and (c) OP-TMF, (d) cyclic stress response curves, evolution of hysteresis loops of the experimental alloy during (e) IP-TMF and (f) OP-TMF.

plane, thereby leading to the premature fracture of the specimen.

Figure 2 visualized the magnified morphologies of the typical H-pores (very close to fracture surface) and the corresponding inverse pole figure (IPF) as well as Kernel average misorientation (KAM) maps. At IP condition, the accumulated stress around H-pores was released through the nucleation of cracks, seen in Figure 2(a). The cracks propagated almost perpendicular to the direction

of external stress, viz., the dislocation deposition and the consequent plastic deformation were more prone to occur in the horizontal γ channels of alloy [10,26]. Subsequently, the horizontal propagation of these pore-induced cracks would in turn lead to an increasing degree of plastic deformation on their tips (Figure 2(c)), which ultimately resulted in the occurrence of recrystallization grains (Figure 2(b)). Actually, the recrystallization had been previously noticed in OP-TMF of SX alloys, which

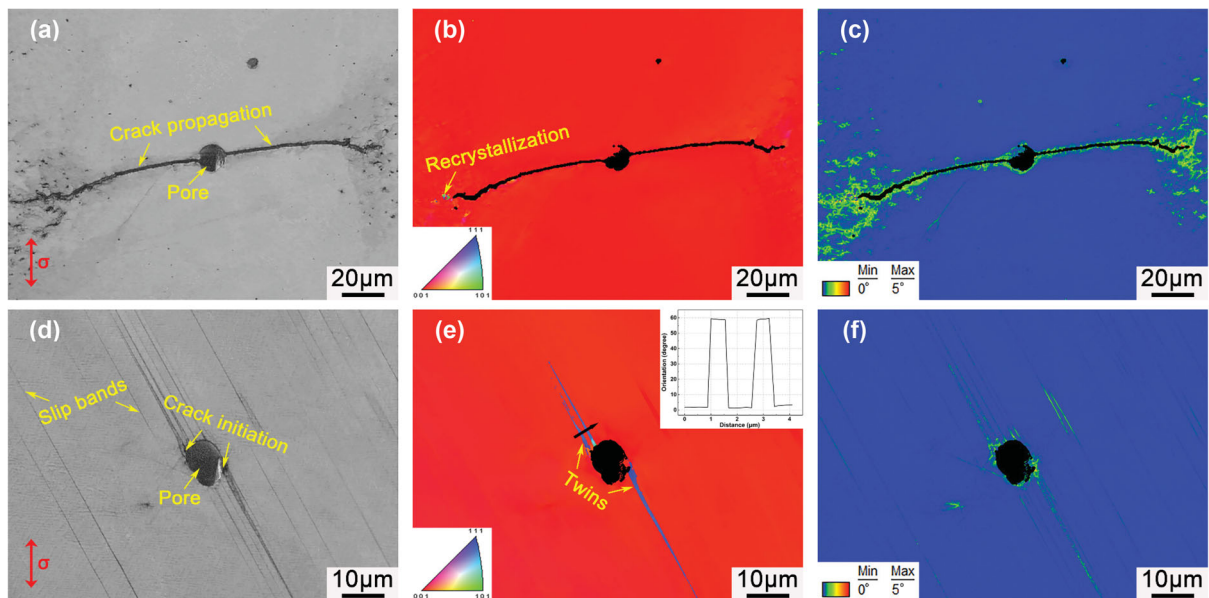


Figure 2. The EBSD analysis of typical pores near fracture surface after (a-c) IP-TMF and (d-f) OP-TMF cycling. (a) and (d) BSD maps, (b) and (e) IPF, (c) and (f) KAM maps. Inset of (e) displaying the typical orientation angle of the twinning bands.

was primarily pertinent to the deformation twins or slipping bands [12,27]. In this work, it was found that during the creep-dominant IP-TMF process, the recrystallization grains could also take shape, meanwhile, their nucleation site was different from previous results [27,28]. Nevertheless, it was also noticed that the crack propagation ended up with occurrence of recrystallization, i.e. no further damage was caused to the alloy.

When it was converted to OP cycling, widespread slipping bands were visible beneath the fracture surface (Figure 2(d)) and the deformation twins that nucleated at the edge of pores could be found in Figure 2(e). Consequently, the accompanied generation of nascent cracks was observed at the position of twinning nucleation, which would rapidly grow along the high-energy twinning boundary (Figure 2(f)), thereby accelerating the crystallographic fracture of alloy [5,23,29]. In this category, additionally, it was also noted that the twinning plates were promptly getting thinner during propagation due to the reduced stress level, detailed explanation would be given in later section.

In order to make a comparison of the deformed structures and disclose the root cause of the above

pore-induced defects, TEM specimens were accurately obtained from the edge of H-pores through FIB method. Figure 3(a) illustrated the dislocation configurations after IP-TMF, where the matrix dislocation segments (marked by triangle) and antiphase boundary (APB)-coupled superpartials were paramount in γ and γ' phases, respectively. The prevailed deformation mechanism of APB-shearing was verified through high-resolution observation (blue circle in Figure 3(a)) of Figure 3(b). Apart from that, stacking faults (SFs) were occasionally observed in γ channel, while the corresponding high-resolution image (green circle in Figure 3(a)) illustrated that the majority of these defects were constrained by γ/γ' interface (Figure 3(c)). Note that although the entrance of SF into γ' particle was not completely forbidden, only one-layer structures were observed throughout the γ and γ' phases. Also, the concomitant $a/6\langle 112 \rangle$ Shockley partials were generated via the decorrelation reaction of matrix dislocation as follows [5,30,31]:

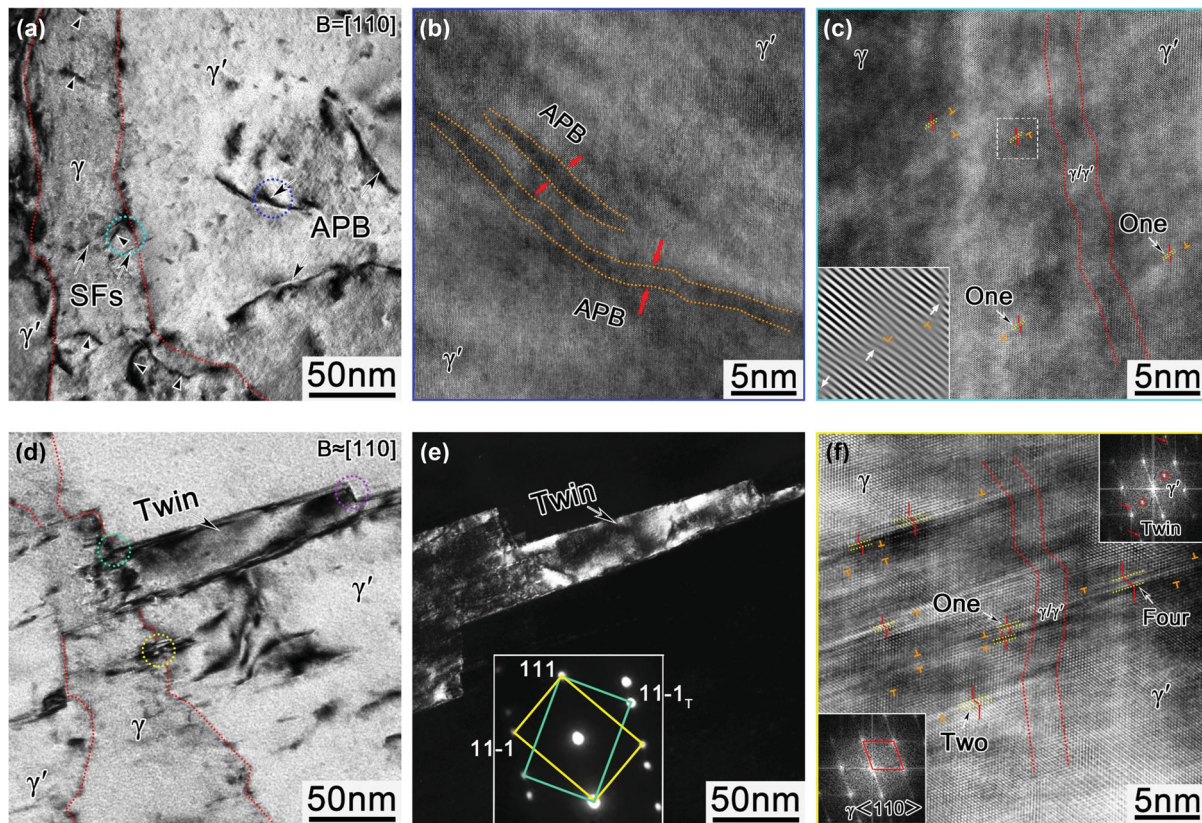
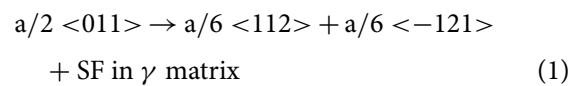


Figure 3. (a) Bright TEM image of the deformed structure close to micro-pore after IP-TMF, (b) high resolution images of APB (relatively low Z-contrast) in γ' phase, (c) high resolution image of dislocation configuration crosses the γ/γ' -interface at IP-TMF, (d) bright TEM image of the pore-induced twin after OP-TMF, (e) contrast observation of dark TEM as well as selective diffraction patterns of the pore-induced twin, (f) high resolution image showing the nucleation of deformation twin at OP-TMF.

Bright field (BF) and contrasted dark field (DF) figures of pore-induced twin formed during OP-TMF were demonstrated in Figure 3(d,e). High-resolution image (Figure 3(f)) which was magnified from the yellow circle in Figure 3(d) showed that, on the one hand, the SFs in matrix as well as accompanied partial dislocations (PDs) had visibly increased in amount compared to IP cycling. On the other hand, two-layer external stacking fault (ESF) and four-layer twin had taken shape in γ and γ' phases, respectively. It was thus deduced that the $\langle 112 \rangle \{111\}$ viscous slipping of groups of two successive Shockley partials had been advantageously activated under OP cycling condition [32,33].

Virtually, the nucleation of deformation twins in SX superalloys requires the consecutive activation of $\langle 112 \rangle \{111\}$ viscous slipping as well as adequate $a/6\langle 112 \rangle$ twinning dislocations [5,34–36]. As the temperature increased from 600°C to 1000°C, the dominant tensile deformation mechanism of SX superalloys with low SFE was gradually converted from SFs-shearing to superdislocations shearing the γ' phase [37]. However, this transformation was completely reversed during the compressive deformation under same temperature range [38]. Therefore, when subjected to IP cycling, the decomposition of matrix dislocations would not be promoted throughout the whole deformation process. Else, it was previously assumed that during IP-TMF, the split-partial dislocations were prone to constrict and recombine into $a/2\langle 110 \rangle$ dislocation in the next high-temperature half cycle [39,40]. Herein, through inverse fast Fourier transformation (IFFT), a strong correlation between the extended dislocations with leading screw partial segments (marked by white arrow in Figure 3(c)) in γ matrix was observed correspondingly, which further contributed to the reduction of twinning dislocations during IP-TMF.

During OP-TMF, however, $a/6\langle 112 \rangle$ twinning dislocations could be generated at both high-temperature or low-temperature half cycles, thereby satisfying the first requirement for twinning nucleation. On the other hand, according to the asymmetry of tensile and compressive deformation, the $\langle 112 \rangle \{111\}$ directional shearing is more preferential in $\langle 001 \rangle$ compression [41–43]. Considered as thermal-activated process, the viscous slipping of $a/6\langle 112 \rangle$ pairs on successive $\{111\}$ planes would be favorably enhanced at high-temperature half cycles during OP-TMF, thereby facilitating the nucleation of deformation twins [32,41].

After the formation of these pore-induced twins, they would gradually be lengthened and thickened driven by the movement of dislocations and segregation of solute atoms [44,45]. Both previous and experimental observations have indicated that the considerable stress concentration near pores was indispensable to twinning

propagation [23]. While in this work, it was discovered that the thickness of twin lamella would be dramatically reduced when encountered γ/γ' -interface or was stacked in γ' particles. Figure 4(a) was magnified from green circle in Figure 3(d), it was clearly seen that the γ/γ' -interface acted as a strong barrier by capturing the leading partials and thus hindering their movements. As a consequence, the atomic layers of twins had decreased, while the $\Sigma 3\{111\}$ coherent twinning boundary (CTB) began to enter into the γ' particle. Figure 4(b) revealed that during the elongation of twinning band in strengthening phase, the movement of front $\Sigma 3\{112\}$ incoherent twinning boundary (ITB) had been partially impeded, viz., the twinning band was further narrowed down [46]. According to the periodic contrast in high-resolution image, it was supposed that the ITB comprised the periodic arrangement of three types of PDs on three successive $\{111\}$ planes (marked by white arrows). Therefore, the contraction of twins in γ' phase could be partially rationalized by the hindrance effect of $L1_2$ -ordered structure to these leading PDs.

Another aspect that needed to be considered was the element segregation behavior, given its role in the formation of twins through reordering and long-ranged diffusion as well as its potential benefit for impeding the motion of dislocations [44,47]. High-resolution scanning TEM (HRSTEM)-EDS mapping of Figure 4 demonstrated the element distributions at γ/γ' -interface and CTB (the magnification was properly lowered compared to Figure 4(a) for better observation), a moderate concentration of Co, Cr, Re and Ru as well as evident depletion of Al and Ni were detected at γ/γ' -interface. Inside the γ' particle, however, a strong distribution of Co, Cr and Re were detected around the CTB. In the meanwhile, the separated Al/Ni-rich γ' and Al/Ni-deficient CTB were also detected.

Actually, the enrichment of Cr and Co on the twins, SFs, APB, etc. have been previously acknowledged, nevertheless, it was discovered that the element Re might also participate in the formation of twins [44,48,49]. Figure S2 showed a more intuitive atom-scale mapping of element distribution across the CTB, the distinct Re/Cr/Co-segregations and a mild enrichment of Ru were found near the twinning boundary. Considering the relatively lower diffusion coefficients of these solute elements in SX alloys, it was reasonable to believe that their long-ranged diffusion into γ' phase was extremely energy-demanded process [50]. From this perspective, the elevated temperature was of vital significance to stimulate the long-range movement of these slow diffusers in γ matrix. Additionally, the required process for twinning formation prior to this segregation scenario, viz., the preceding short-range reordering between Al and Ni atoms, would be

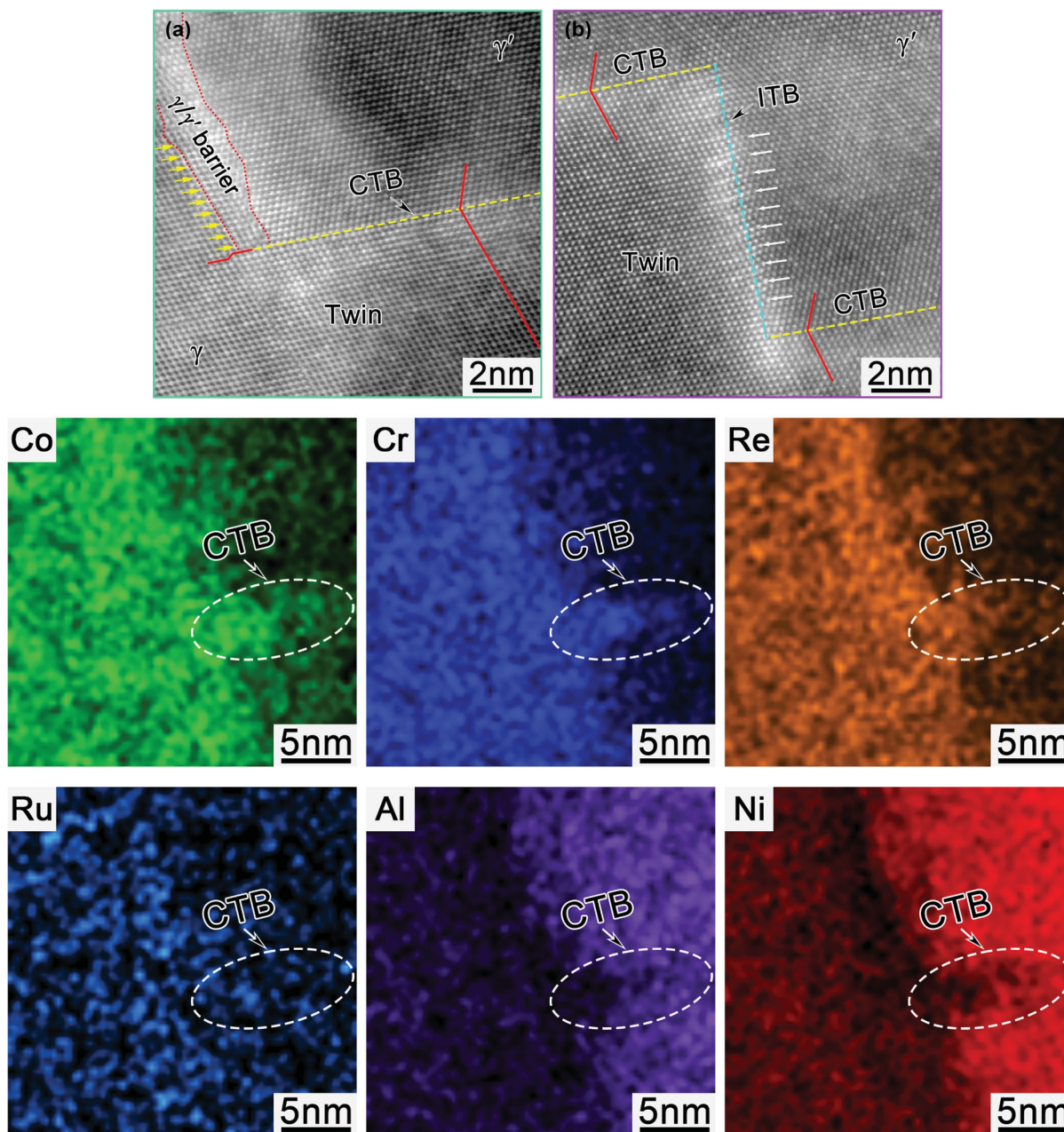


Figure 4. (a) High resolution image illustrating that the extension of deformation twin was partially impeded by γ/γ' -interface, (b) characteristic of typical CTB and ITB in the γ' particle. HRSTEM-EDS element mapping displaying the element segregation at the γ/γ' -interface and CTB in γ' phase (white cycle).

synchronously impelled [47]. Therefore, in comparison to IP-TMF, the coupling of compressive stress and high temperature under OP cycling would not only enhance the dislocation-shearing, but assisted in the essential element diffusions, thereby synergistically promoting the formation of pore-induced twins.

Apart from the encouraging influence of these critical elements on twinning formation, additional attention was also paid to their impeding effects on the dislocation movement. Previous reports have proposed that

the Re-concentration to crystalline defects like γ/γ' -interface and PDs could resultantly stabilize the interface or impose a strong drag-effect to dislocations [51–53]. In the present work, it was striking to find out that during the propagation of twins in γ' phase, the PDs and superlattice intrinsic stacking fault (SISF) were enriched with higher content of Co, Cr and Re, as shown in Figure S2. These solute atmospheres could interact with these defects and exert obstacles to dislocation gliding, which provided a supplementary strengthening effect and well

explained the constant contraction of twinning bands. Nevertheless, it needed to be pointed out that regardless of the remarkable resistance to twinning propagation (including interface strengthening, γ' -strengthening and pinning effect of refractory elements), the surging H-pores would provide considerable shortcuts for twinning nucleation in the alloy. Hence, during OP-TMF process, the extension of primary crack would be accelerated by

the occurrence of excessive pore-induced twins, resulting in the rapid fatigue fracture along typical $\{111\}$ plane.

Eventually, the schematic illustrations of the formation and evolution mechanisms of pore-induced defects as well as resultant fracture characteristic were drawn in Figure 5. It could be reasonably assumed that the pore-induced recrystallizations formed at IP-cycling were much less detrimental to the alloy, compared

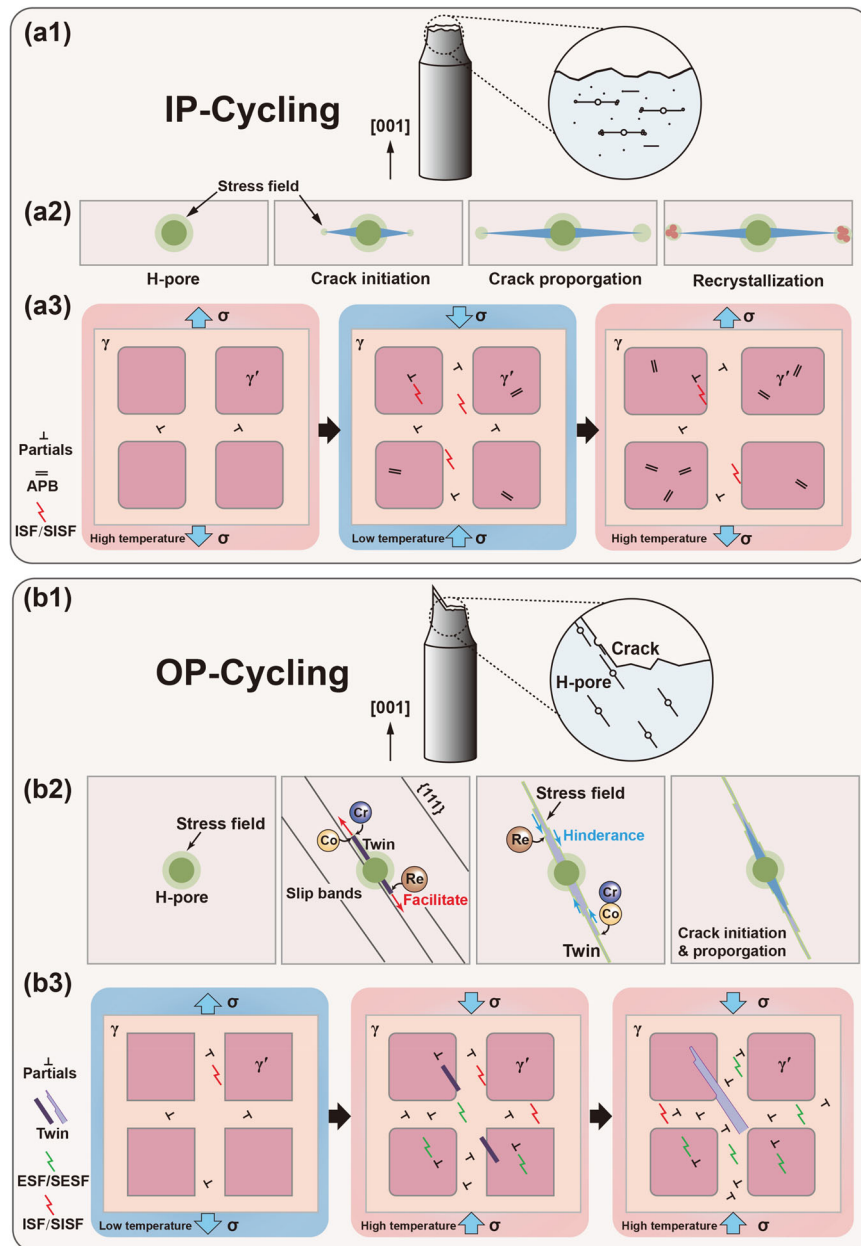


Figure 5. Schematic illustration of the formation and evolution of pore-induced defects as well as TMF fracture features in fourth-generation SX superalloy during IP- and OP-TMF processes. (a1) characteristic of flat fracture and horizontal cracking at IP-cycling, (a2) procedures of crack initiation and propagation as well as introduction of recrystallizations, (a3) evolution of dislocation configurations during consecutive high-temperature and low-temperature half cycles, (b1) characteristic of crystallographic fracture and cracking at OP-cycling, (b2) mechanisms of twinning formation and propagation as well as cracking along twinning boundary, (b3) evolution of dislocation configurations and contraction of twins during OP-TMF.

to the pore-induced twins formed under OP-cycling. Therefore, in the future design and application of Ru-containing fourth-generation SX superalloys, it is suggested to appropriately reduce the holding time of high-temperature solution treatment, for the sake of lowering the number of H-pores in the alloy and accomplishing a co-enhancement of TMF property under different cycling phase conditions.

4. Conclusions

To sum up, conclusions could be drawn as following:

- (i) Recrystallizations and deformation twins would be induced in the vicinity of pores during IP- and OP-TMF of fourth-generation SX alloy, respectively.
- (ii) The accumulation of pore-induced twins accelerated fatigue fracture of alloy; no evident adverse effect of pore-induced recrystallization was found.
- (iii) The increasing density of $a/6 \langle 112 \rangle$ twinning dislocations and more frequent activation of $\langle 112 \rangle \{111\}$ viscous slipping accounted for nucleation of twins during OP-TMF.
- (iv) The contraction of twins basically occurred at the γ/γ' -interface or in γ' particles. Re/Co/Cr-segregations could on the one hand contribute to the formation of twins, on the other hand hinder the movement of PDs.

These above findings provided new insight and experimental evidence into improving the service reliability of fourth-generation SX superalloys and preventing the alloys from untimely TMF fracture failure.

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Disclosure statement

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

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