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Microtwinning mechanism revealed by its orientation dependence and tension-compression asymmetry in γ' precipitate-strengthened Ni-based superalloys

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ABSTRACT

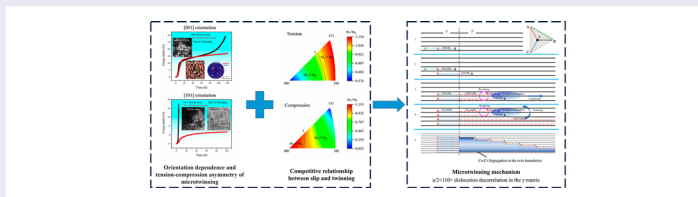
Microtwinning is observed as an important mechanism in γ' precipitate-strengthened Ni-based superalloys during service at intermediate temperatures. Here, the effect of crystal orientation and stress direction on microtwinning was investigated. The result indicates that the formation of microtwins exhibits orientation dependence and tension-compression asymmetry. Based on the analysis of slip and twinning competition behavior under different conditions, it is deemed that the $a/2\langle 110 \rangle$ matrix dislocations would dissociate into $a/6\langle 112 \rangle$ Shockley partials in the γ matrix and then a Shockley partial sheared γ' phase and formed microtwins.

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KEYWORDS

Ni-based superalloys; microtwinning; orientation dependence; tension-compression asymmetry



IMPACT STATEMENT

For the first time, this work explains why microtwinning exhibits orientation dependence and tension-compression asymmetry while stacking faults do not, although they both originate from the activity of $\{111\} \langle 112 \rangle$ slip systems.

1. Introduction

γ' precipitate-strengthened Ni-based superalloys are an important class of materials that are used in demanding high temperature structural applications such as the hot zone of aircraft gas turbine engines [1]. Their special two-phase microstructure, the combination of the FCC γ matrix and the coherent L_{12} γ' precipitate, was known to be the origin of unusual anomalous high-temperature mechanical properties [2–5]. The precipitation hardening from γ' precipitates is one of the main strengthening mechanisms, in which γ' precipitates can hinder the movement of matrix dislocations [6–10]. Planar defects such as antiphase boundaries (APB), complex stacking faults (CSF), superlattice intrinsic stacking faults (SISF), superlattice extrinsic stacking faults (SESF) and microtwins, will form due to different types of dislocations shearing γ' precipitates [11–23]. Microtwinning is

one of the typical characteristics at intermediate temperatures, which has a significant impact on alloy properties.

Over the past few decades, researchers have conducted extensive research on the microtwinning mechanism, and the development of microtwinning formation mechanism can be seen in **S1 of Supplementary Materials**. A consensus does seem to be formed around that microtwins are caused by the propagation of $1/6\langle 112 \rangle$ twinning partials on every consecutive $\{111\}$ plane [24–26]. However, there is a big controversy surrounding the dissociation of $1/2\langle 110 \rangle$ matrix dislocations and the formation of $1/6\langle 112 \rangle$ Shockley partials. Unocic et al. [25] thought the matrix dislocation decorrelation appeared to be a key precursor to microtwinning, which was the $a/2\langle 110 \rangle$ matrix dislocations should first dissociate into $a/6\langle 112 \rangle$ Shockley partials in the γ matrix channel. However, Smith et al. [26] thought the $a/2\langle 110 \rangle$

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dislocation decorrelation in the γ matrix was not a necessary precursor for micro-twinning and $a/2\langle 110 \rangle$ dislocation should dissociate at the γ/γ' interface. Figure 1 compares the differences in these microtwinning mechanisms. Figure 1(a) is the microtwin formation model where $a/2\langle 110 \rangle$ dislocation decorrelation at γ/γ' interface. First, a single $a/2\langle 110 \rangle$ dislocations move to γ/γ' interface and then is decomposed into two $a/6\langle 112 \rangle$ Shockley partial dislocations. Then, a $a/6\langle 112 \rangle$ partial shear into γ' phase, which would result in the formation of a single-layer CSF [17,27]. When another $a/2\langle 110 \rangle$ matrix dislocation dissociates on the adjacent slip plane at γ/γ' interface in the same way, a two-layer CSFs would be created in γ' phase. The two-layer CSFs are transformed into an SESF by short-range diffusion through the reordering mechanism, and the SESF could be regarded as the embryonic twin [28]. When more $a/2\langle 110 \rangle$ dislocations decompose in the above way, microtwins will form in γ' phase. The presence of Co- and Cr-rich Cottrell atmospheres around leading dislocations would promote the long-range diffusion of critical elements such as Cr and Co, which is beneficial for the lengthening and thickening of microtwins [14,15,29]. The segregation behavior of Co and Cr at the microtwin interface was a common phenomenon, which was also found in this study (See S2 of Supplementary Materials). Finally, due to the asynchrony of $a/6\langle 112 \rangle$ Shockley partial dislocations shearing in the γ' phase, microtwins having varying thicknesses are eventually formed [30,31]. Different from the microtwin formation model shown in Figure 1(a), the process of $a/2\langle 110 \rangle$ dislocation dissociating into $a/6\langle 112 \rangle$ Shockley partials occurs in the γ matrix channel rather than at γ/γ' interface for the model shown in Figure 1(b).

In addition to the controversy over the formation location of $1/6\langle 112 \rangle$ Shockley partial dislocations, microtwins seem to be closely related to crystal orientation and stress direction [32–36]. However, existing research still cannot explain the impact of microtwinning mechanism on this phenomenon. Thus, the current work investigates the effects of crystal orientation and stress direction for microtwinning in Ni-based superalloys. Finally, we reveal the microtwinning mechanism based on its orientation dependence and tension-compression asymmetry. This work also explains why microtwinning exhibits orientation dependence and tension-compression asymmetry while stacking faults do not, although they both originate from the activity of $\{111\}\langle 112 \rangle$ slip systems.

2. Experimental

A third-generation Ni-based single crystal superalloy is used in the present experiment [37], and the nominal

composition is as follows(wt.%): 3.5Cr, 12Co, 1.5Mo, 6W, 8Ta, 4Re, 6Al, and the balance Ni. Further details experimental procedure and analysis method are available in the **Supplementary Material (S3)**.

3. Results and discussion

Figure 2 analyzes the microstructures of [001] oriented single crystal superalloy after tensile and compressive creep at 750 °C/750 MPa. The black curve in Figure 2(a) is the tensile creep curve, which shows a typical three-stage creep [38]. The red curve in Figure 2(a) shows the compressive creep curve, which underwent a 120-h compressive creep interruption test at 750 °C/750 MPa. It can be observed that the creep curve of the [001] oriented specimen in the primary stage does not exhibit significant tension-compression asymmetry, and the secondary stage of the tensile creep curve also has a significant overlap with the compressive creep curve. However, in the tertiary stage, the strain of the tensile specimen increases sharply, while the compressive creep specimen has a longer secondary stage.

Figure 2(b) shows the TEM microstructure of the [001] oriented specimen after tensile creep, where the deformation microstructure includes large number of matrix dislocations and stacking faults in γ' phases. Similar to tensile creep, there are also a large number of stacking faults during compressive creep in Figure 2(c). Figure 2(d) indicates microtwins also exist in the [001] oriented compressive creep specimen. The EBSD map of the compressive creep specimen indicates there are two micro stripes with significant changes in orientation. The misorientation angle (60°) on both sides of the typical stripe area in Figure 2(e) and the EBSD pole figure in Figure 2(f) indicate that the stripe structure is microtwins [39]. Therefore, for the [001] oriented specimen, not only the creep behavior but also the microstructure exhibits tension-compression asymmetry. Regardless of tensile or compressive creep, there are a large number of stacking faults, while microtwins are related to deformation conditions. There are no microtwins in the deformation microstructure after tensile creep, but microtwins exist in the specimen after compressive creep. In other superalloys, [001] oriented samples also exhibit the similar law [33,40,41].

The creep behavior and microstructures of single crystal superalloys with other orientations also exhibit tension-compression asymmetry at intermediate temperatures and high stress. Figure 3 analyzes the microstructures of [101] oriented single crystal superalloy after tensile and compressive creep at 750 °C/750 MPa. The black curve represents the tensile creep curve and is magnified in the purple rectangle, as shown in Figure 3(a).

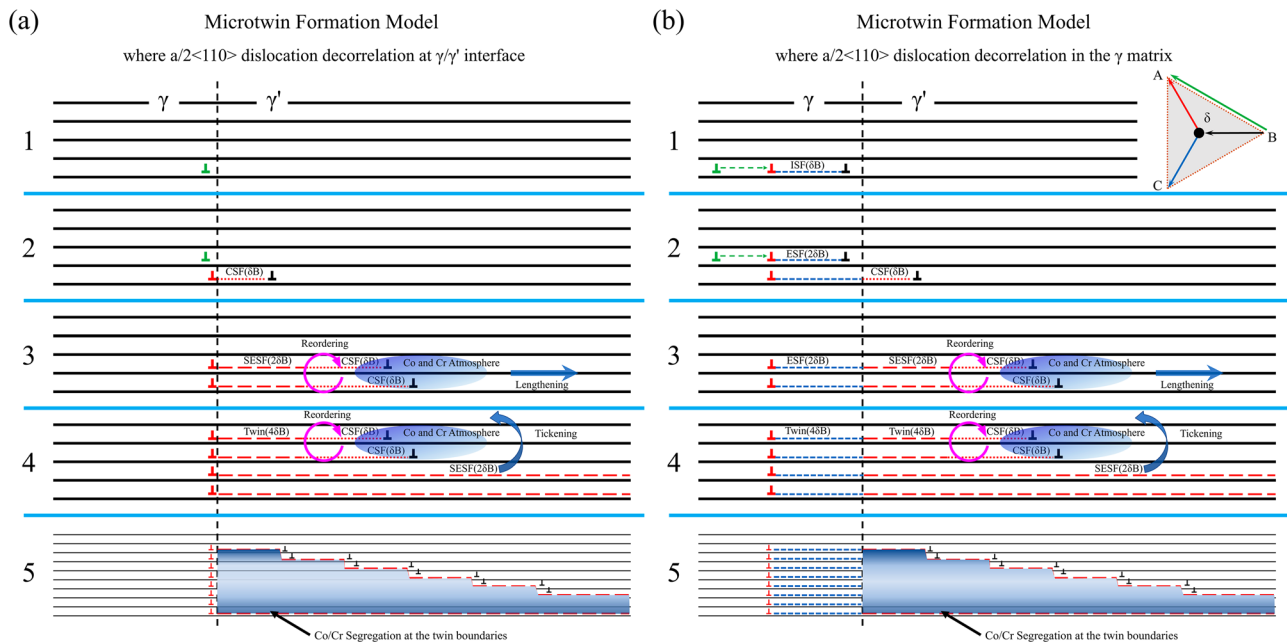


Figure 1. Two microtwin formation models in superalloys: (a) where $a/2\langle 110 \rangle$ dislocation decorrelation at γ/γ' interface [26]; (b) where $a/2\langle 110 \rangle$ dislocation decorrelation in the γ matrix [25].

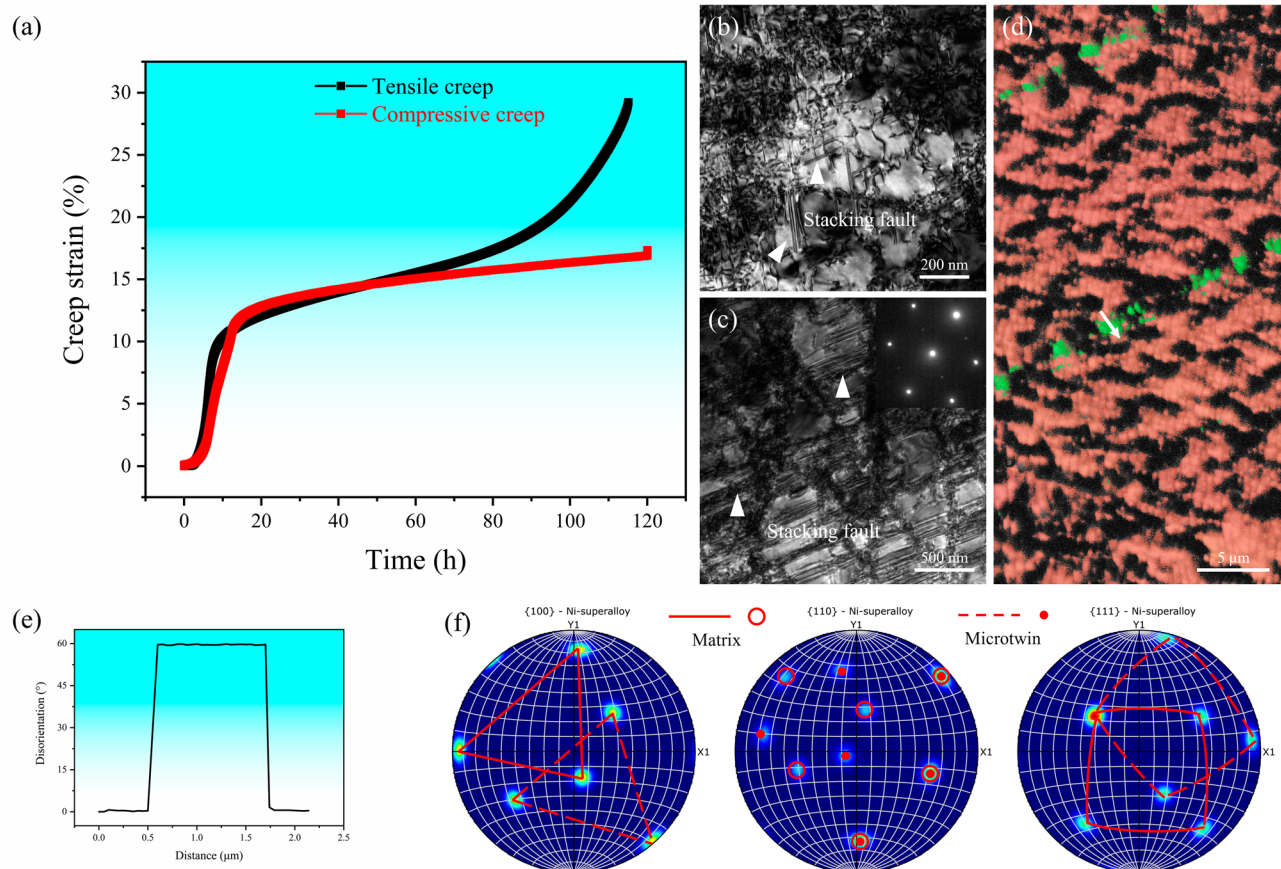


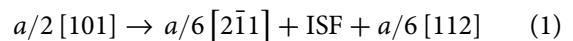
Figure 2. Analysis of microstructure of [001] specimens after tensile and compressive creep at 750 °C/750 MPa: (a) Creep curves; (b) TEM microstructure of the tensile creep specimen; (c) TEM microstructure of the compressive creep specimen; (d) EBSD map of the compressive creep specimen; (e) The disorientation along the white arrow in (d); (f) Corresponding EBSD pole figure map of (d).

Similar to some previous research [30,42], the [101] specimen does not exhibit a typical three-stage creep and has a poor creep life of approximately 4.3 h. The red curve in Figure 3(a) is the creep curve of the [101] specimen after 100 h of compressive creep at 750 °C/750 MPa. It can be found that the specimen has a longer secondary stage in compressive creep. These creep curves indicate that the tension-compression asymmetry of creep behavior of the [101] oriented specimen is more severe than that of the [001] oriented specimen. Figure 3(b) indicates stacking faults remain the main deformation microstructure in the [101] tensile creep specimen. There are also some microtwins in the [101] tensile creep specimen, and the bright and dark field images of the same microtwins are shown in Figure 3(c) and (d), respectively. The microstructure of the [101] oriented specimen after compressive creep is shown in Figure 3(e), and the most typical microstructure still is stacking faults. Compared with the tensile creep specimen, the number of stacking faults is smaller and the density of matrix dislocations is also lower in the compressive creep specimen, which is related to its smaller creep strain. The compressive yield strength of [101] oriented single crystal superalloys is higher than the tensile yield strength [43], which is also the main reason for their smaller strain. Although the deformation microstructure of [101] oriented single crystals also exhibits tension-compression asymmetry, the formation law of microtwins is completely opposite to that of [001] oriented single crystals. For [101] oriented single crystals, microtwins can form during tensile creep, while there is no microtwins during compressive creep. Therefore, the formation of microtwins in Ni-based superalloys is not only related to crystal orientation but also stress direction.

The formation of microtwins in Ni-based superalloys exhibits significant orientation dependence and tension-compression asymmetry, as shown in Figures 2 and 3. Except for superalloys, the formation of twins in other alloys with FCC structure also exhibits similar characteristics. Figure 4(a) summarizes the orientation dependence of twinning formation in several typical FCC alloys and superalloys during tensile deformation. Yang et al. [44] conducted a statistical analysis for the grain orientation of deformation twinning in a high manganese steel after tensile deformation, and the results showed that the most twinning was seen for grains with orientations near [111] during tensile deformation, as shown by the black square in Figure 4(a). For Fe-22 wt.% Mn-0.6 wt.% C TWIP steel [45], the grain orientations that can form deformation twinning under tensile deformation are generally enriched near the [111] pole, and deformation twinning is also found at the [101] orientation, as shown by the red solid circle in Figure 4(a). For other FCC alloys,

the orientation distribution formed for twinning during tensile deformation is also the same [46–48]. Figure 4(b) shows the orientation distribution map that can form twinning in several typical FCC alloys and superalloys in compression. For FeMnNiCoCr high entropy alloys [48] and TWIP steel [49,50], twinning can be formed in compression in the sample with an orientation close to the [001] pole. For single crystal superalloys, the orientation dependence and tension-compression asymmetry of microtwinning formation is consistent with FCC alloys, as shown by the orange ellipse in Figure 4(a) and (b). Table 1 further provides statistics on the formation conditions of microtwins in different single crystal superalloys. It can be found that microtwins tend to occur in [101] oriented and [111] oriented single crystal superalloys during tension [14,15,51–55], while microtwins tend to form in [001] oriented single crystals during compression [26,33,34,36,40,41,52,53,56–58]. In addition, microtwins can also form in CMSX-4 alloys with [165] and [256] orientations during tension [34,53], both of which are located near the [101]–[111] boundaries.

The consistency of twinning formation laws between Ni-based superalloys and typical FCC alloys may be related to the FCC matrix of superalloys. In FCC alloys, slip and twinning are two typical deformation modes. Slip is mainly caused by the movement of $a/2\langle 110 \rangle$ dislocations, which is dominated by $\{111\}\langle 110 \rangle$ slip systems. Twinning is dominated by $\{111\}\langle 112 \rangle$ slip systems, and the formation of twins is caused by multiple $a/6\langle 112 \rangle$ Shockley partial dislocations shearing through adjacent $\{111\}$ slip planes in sequence. The decomposition reaction is always described as follows:



Where $a/2 [101]$ dislocation dissociates into two $a/6\langle 112 \rangle$ Shockley partial dislocations, $a/6 [2\bar{1}1]$ and $a/6 [112]$. There is an intrinsic stacking fault (ISF) sandwiched between two Shockley partial dislocations. Then, the twins will form, when multiple $a/2\langle 110 \rangle$ dislocations dissociate on the adjacent $\{111\}$ planes in the same way. During the deformation process, slip and twinning would compete with each other, and twinning usually occurs when slip is less likely to occur. Due to the prerequisite for the activity of $\{111\}\langle 112 \rangle$ systems is that $\{111\}\langle 110 \rangle$ slip systems are first activated, there must be slip when twinning deformation occurs but twinning may not necessarily occur when slip exists. Therefore, the orientation dependence and tension-compression asymmetry of twinning relate to the competition between these two deformation modes. Figure 4(c) describes the Schmid factor contours for slip and for twinning of FCC alloys with different orientations during tensile deformation. The black dashed line in Figure 4(c) indicates that the

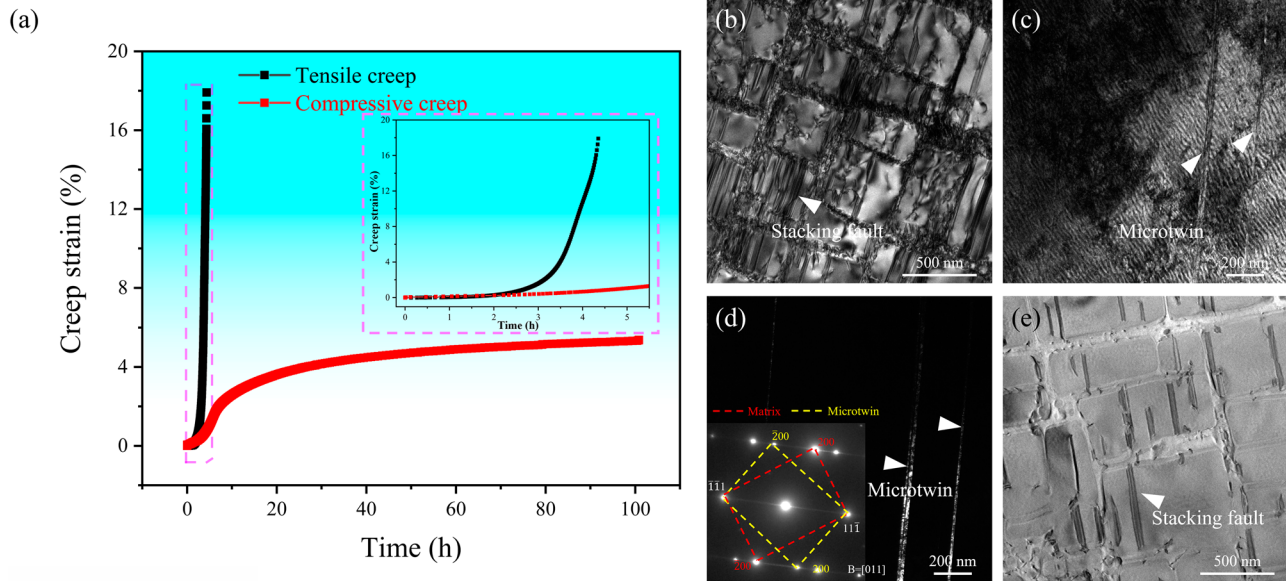


Figure 3. Analysis of microstructure of [101] specimens after tensile and compressive creep at 750 °C/750 MPa: (a) Creep curves; (b)–(d) TEM microstructure of the tensile creep specimen; (e) TEM microstructure of the compressive creep specimen

Table 1. Formation conditions of microtwinning in different single crystal superalloys.

Alloy	Orientation	Temperature (°C)	Stress (MPa)	Type of Stress	Reference
MD2	011	800	625	Tensile	[14]
MD2	011	800	675	Tensile	[14]
MD2	011	825	625	Tensile	[14]
MD2	011	800	650	Tensile	[14,15,51,52]
MD2	001	825	625	Compressive	[52]
CMSX-4	111	750	750	Tensile	[53]
CMSX-4	165	750	750	Tensile	[53]
CMSX-4	001	750	750	Compressive	[33,34,53]
CMSX-4	256	950	350	Tensile	[34,53]
CMSX-4	001	950	350	Compressive	[34,53]
NKH-304	001	750	750	Compressive	[36]
NKH-510	001	750	750	Compressive	[36]
ME3	001	760	414	Compressive	[26]
SRR99	111	850	450	Tensile	[54]
ERBOCo-2Ta	001	975	150	Compressive	[56]
PWA1480	001	750	750	Compressive	[33,40]
TMS-75	001	750	750	Compressive	[33]
ERBOCo-1	001	850	400	Compressive	[41]
DD33	111	850	650	Tensile	[55]
PWA1483	001	1000	-	Compressive	[57,58]
DESC-1	001	1000	-	Compressive	[57]
Rene N5	001	1000	-	Compressive	[57]

Schmid factor for slip at this position is the same as that for twinning. Figure 4(d) shows the ratio distribution of the Schmid factor for twinning m_T to the Schmid factor for slip m_S at different orientations during tensile deformation, in which the dashed line indicates that m_T/m_S is 1. Comparing these two figures, it can be observed that the m_T/m_S of the [111] and [101] poles, as well as the regions near the [101]–[111] boundaries, is larger than 1, which means the Schmid factor for twinning is greater than the Schmid factor for slip. In the region formed by the black dashed line and [001] poles, the m_T/m_S is less than 1, hence the Schmid factor for twinning is smaller than the Schmid factor for slip. Therefore, the areas where

$m_T/m_S > 1$ are prone to twinning during tensile deformation. The orientation of twinning formation in several typical FCC alloys shown in Figure 4(a) is also consistent with the areas prone to twinning deformation shown in Figure 4(c) and (d). Figure 4(e) and (f) are the Schmid factor contours for slip and for twinning and the ratio distribution of the Schmid factor for twinning m_T to the Schmid factor for slip m_S of FCC alloys with different orientations during compressive deformation, respectively. Similar to Figure 4(c) and (d), the black dashed line also indicates the Schmid factor for slip at this position is the same as that for twinning. Unlike tensile deformation, the Schmid factor for twinning near the [001] orientation is

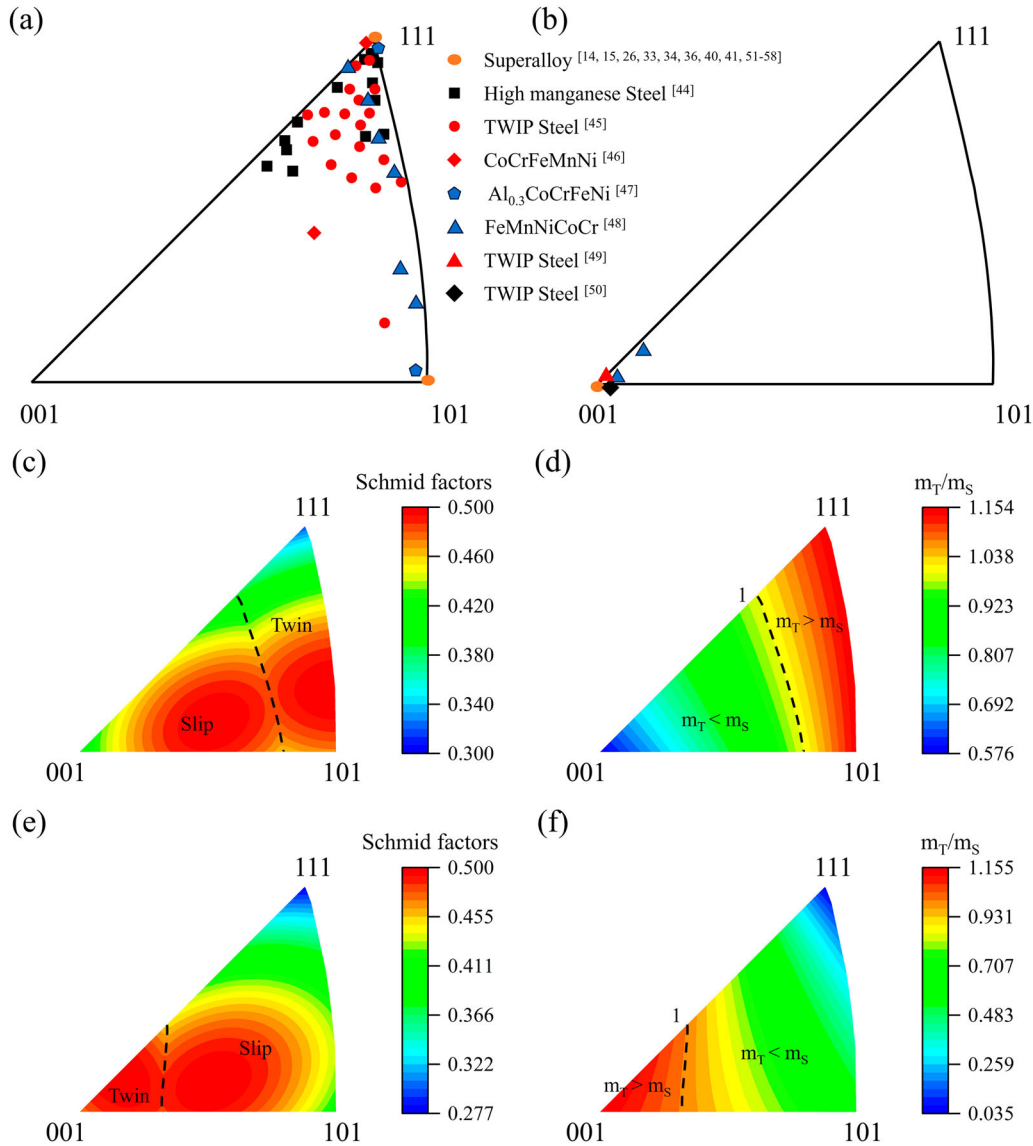


Figure 4. (a) Orientation dependence of twinning formation in several typical FCC alloys and superalloys during tensile deformation; (b) Orientation dependence of twinning formation in several typical FCC alloys and superalloys during compressive deformation; (c) Schmid factor distribution for slip and twinning during tension; (d) Distribution map of the ratio of Schmid factor for twinning to Schmid factor for slip under different orientations during tension; (e) Schmid factor distribution for slip and twinning during compression; (f) Distribution map of the ratio of Schmid factor for twinning to Schmid factor for slip under different orientations during compression.

larger than the Schmid factor for slip. Therefore, the orientation of twinning that can be formed in FCC alloys during compressive deformation is around the [001] pole, as shown in Figure 4(b) and Table 1.

In superalloys, both stacking faults and microtwins originate from the activity of $\{111\}\langle 112 \rangle$ slip systems, hence microtwins should be theoretically formed on the condition for the formation of stacking faults. However, it has been proven that only the formation of microtwins exhibits orientation dependence and tension-compression asymmetry. For stacking faults, the commonly accepted mechanism is two $a/2 \langle 110 \rangle$ matrix dislocations with different Burgers vectors dissociating at

γ/γ' interface and the $a/3\langle 112 \rangle$ dislocation shear in γ' phase [16]. During the deformation, $\{111\}\langle 110 \rangle$ slip systems would be first activated, resulting in the formation of a large number of $a/2\langle 110 \rangle$ matrix dislocations. Then, the $a/2\langle 110 \rangle$ matrix dislocations would slip to γ/γ' interface and react to form stacking faults. Due to the fact that stacking faults are formed at the γ/γ' interface rather than in the FCC matrix through formula (1), the competition between slip and twin in the FCC matrix would not influence the formation of stacking faults. Therefore, the formation of stacking faults is independent of crystal orientation and stress direction.

For microtwins, if $a/2\langle 110 \rangle$ dislocation decorrelation occurs at the γ/γ' interface as shown in Figure 1(a), the movement of $a/2\langle 110 \rangle$ dislocations in the γ matrix is still dominated by slip. Hence, the formation of microtwins is also not affected by crystal orientation and stress direction, just like stacking faults. If $a/2\langle 110 \rangle$ dislocation decorrelation occurs at the γ/γ' interface as shown in Figure 1(b), due to the competitive relationship between slip and twinning in the FCC γ matrix, the formation of microtwins is affected by crystal orientation and stress direction. The formation conditions of microtwins in superalloys are also consistent with the twinning formation pattern in FCC structured crystals shown in Figure 4. During tensile conditions, only samples near the [101] and [111] poles, as well as near the [101]–[111] boundaries, exhibit microtwinning formation, while during compressive deformation, microtwins only form in the areas near the [001] orientation. Meanwhile, microtwins observed in experiments often penetrate multiple γ matrix and γ' phases. Therefore, the formation of microtwins in superalloys first occurs in the γ matrix channel, $a/2\langle 110 \rangle$ dislocation decorrelation in the matrix is a prerequisite for the formation of microtwins.

4. Conclusion

In this work, the orientation dependence and tension-compression asymmetry of microtwinning in γ' precipitate-strengthened Ni-based superalloys were studied to reveal the mechanism of microtwin formation. In tensile deformation, the orientation that can form microtwinning was mainly concentrated at the [101] pole, [111] pole, and near the [101]–[111] boundary. In compressive deformation, microtwinning was easily formed near the [001] orientation. The competitive relationship between slip and twinning in the FCC γ matrix leads to the orientation dependence and tension-compression asymmetry of microtwinning. Therefore, the $a/2\langle 110 \rangle$ matrix dislocations would dissociate into $a/6\langle 112 \rangle$ Shockley partials in the γ matrix. Then, $a/6\langle 112 \rangle$ Shockley partials shear γ' phase and form microtwins.

Disclosure statement

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

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Data availability statement

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of other ongoing studies.

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