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The nucleation of δ phases triggered by the stacking faults in a single crystal superalloy

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ABSTRACT

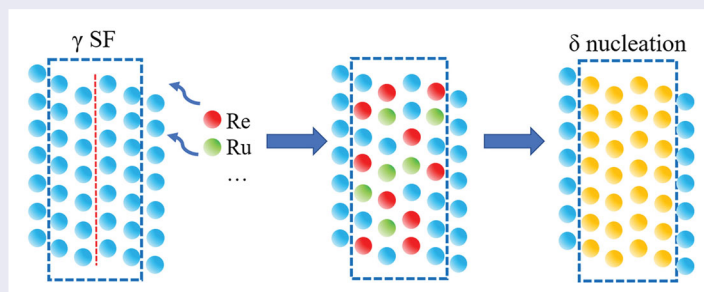
Here we investigated the nucleation of the Re, Ru-rich δ phase affected by stacking faults in the γ phase in a 4th generation Ni-based single crystal superalloy during the service at 800°C. The stacking faults in the γ phase were rich in Re and Ru, similar to the observed δ phase. And the stacking fault regions possessed a similar structure to the δ phase, promoting the transformation from γ phase to δ phase. Furthermore, the calculation results indicated that Ru had more significant promoting effects on the δ phase precipitation than Re, providing a reference for the alloy design.

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KEYWORDS

Ni-based single crystal superalloy; δ phase; stacking faults; nucleation



1. Introduction

Ni-based single crystal superalloys (SCs) have been applied widely on turbine blades of aero and gas turbine engines for their excellent mechanical properties at elevated temperatures [1–3]. The increasing demands for inlet temperature necessitate the development of new generation Ni-based SCs with improved temperature capability. The past decades applied increasing contents of refractory elements such as Mo, W, and, Re as common strategy for enhancing the temperature capability of the material [4–7]. However, these refractory contents, especially Re, generally facilitate the precipitation of topologically close-packed phases (TCP phases), which damage

the alloy performance greatly by dissolving the surrounding microstructure and inducing microcrack initiation [8,9]. Element Ru, restricting TCP phases precipitation effectively, has thus increasingly been applied in the new generation alloys [10,11]. But the increased addition of Ru is unfavorable for the density and cost of the alloy [12]. Moreover, excessive addition of Ru even lead to the precipitation of other secondary phases, such as δ -ReRu [13,14], bringing further problems to the processing of the alloys. Thus, it is vital to determine the precipitation mechanism of these secondary phases for alloy design.

δ phases that are rich in Re and Ru have received much less attention. The hexagonal δ phase is typically rich in

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Re and Ru, easy to precipitate during the solidification process when the content of Re and Ru is high [13,14]. Typically, the dendrite of the δ phase nucleated during the solidification of Ni-based superalloys at temperatures well above the liquidus of γ -Ni [14]. However, the nucleation of the δ phase during the service at high temperatures is rarely reported. With the addition of high content of Re and Ru in the new generation of Ni-based SCs, the δ phase can precipitate during service, which could further influence the processing and mechanical properties of SCs. Therefore, the investigation of the nucleation mechanism of the δ phase becomes necessary for the purpose of alloy design.

During the creep process at intermediate temperature (about 700°C ~ 850°C) [1,15,16], multiple stacking faults (SFs) emerge in the γ channel and γ' phase [17]. In this paper, we discovered that the δ phase emerged during the creep process at intermediate temperatures along the direction of SFs. This phase transformation is analogous to other ones previously reported in Ni-based superalloys and other alloy systems [18,19]. For instance, the η phase and χ phase transformation along the SFs have been proven beneficial to the creep strength [19–22]. The local phase transformation in the SF regions is induced by the structural change caused by SF shearing and solute segregation along the SFs [19,23]. The emphasis on the nucleation mechanism of the δ phase provides a perspective for the alloy design and regulation of the Re and Ru contents.

2. Experimental

The composition of the investigated SC was listed in Table 1. The experimental alloy was casted by the Bridgman method. And the typical three-step heat treatments were applied to process the as-cast material, including a high-temperature solution heat treatment (1350°C) and two aging treatments at relatively low temperatures (1100°C and 870°C). Isothermal tensile creep tests were carried out at 800 °C and 735 MPa. The load direction was along the direction of [001] orientation of SC. The creep strain reached 16.61% when the sample ruptured. Moreover, the interrupted sample at 550 h (creep strain ~0.69%) was applied to investigate the SF in the γ matrix. The samples of the as-cast state and heat-treated state were applied to compare with the samples after loading in terms of microstructure. The samples for transmission electron microscope (TEM) observation were cut perpendicular to the direction of $\langle 110 \rangle$ from the crept specimen. The samples were mechanically grinded and polished and then prepared by electrochemical double twin jet thinning at around — 30 °C and a voltage of near 15 V. The electrolyte included 7 vol.% HClO₄ and 93 vol.% ethanol. Ordinary TEM observation was carried out on Tecnai F20. The atomic figure was obtained on Titan G2 80-200 and Thermofisher Spectra

Table 1. Composition of the investigated alloy (wt.%).

Al + Ta	W + Cr + Co	Re	Ru	Hf	Mo	Ni
12	14.8	5	2.5	0.1	4	Bal.

300. The TEM images were captured along the direction of [110]. Specifically, high-resolution energy dispersive spectrum (EDS) was performed on Thermofisher Spectra 300 and the corresponding sample was cut by focused ion beam (FIB). Thermodynamic calculation on the δ phase was carried out on the software ThermoCalc, using the database TCNi9: Ni-Alloys v9.1.

3. Results and discussion

No δ phase emerged in the as-cast and heat-treated samples (Figure S1 in the supplementary materials). After rupture over 2000h, rod-like precipitates with a length of about 100 nm and a width of 10 nm appeared in the γ channel (marked by white arrows in Figure 1a). The view direction of Figure 1 was parallel to $[110]_{\gamma}$ and the loading stress axial was along $[001]_{\gamma}$. Many straight bright traces parallel to the direction of $[\bar{1}12]$ or $[1\bar{1}2]$ emerged within the γ channel. The enlarged image in the left corner of Figure 1(a) reveals these traces are SFs typically formed after the intermediate-temperature creep of Ni-based SCs [24]. In Figure 1(a), the growth direction of δ particle was parallel to the direction of $[\bar{1}12]$, which is the projection direction of SFs on the $(\bar{1}\bar{1}1)$ and $(\bar{1}11)$ planes. The elemental distribution of δ particle was shown in Figure 1(b). Re and Ru segregated in the δ particle, while the segregation tendency of Mo, Co and Cr in the particle was insignificant. Re and Ru are typical δ phase-forming elements in Ni-based superalloys, consistent with the former studies. Moreover, Re and Ru mainly partitioned in the γ matrix in Figure 1(b). In new-generation SCs, Re acts as a main strengthener in the γ matrix and increases the lattice misfit between γ and γ' phase significantly, which provides strong creep resistance [25,26]. However, Re is also one of the main TCP-forming elements [27]. Ru is added to suppress the formation of TCP phases [10], and Ru could also decrease the stacking fault energy (SFE) in the matrix, providing creep resistance [28]. In Figure 1(b), the absence of other typical TCP-forming elements, such as Mo and Cr, excluded the possibility of TCP phases [8,29]. Furthermore, Figure 1(c and d) showed the fine structure of the precipitate. The high-resolution TEM (HRTEM) images (Figure 1d) roughly revealed the structure of the particle precipitated in the γ phase. The fast Fourier transform (FFT) image in Figure 1(e) confirmed the type of this precipitate as δ phase, indicating the orientation relationship between δ phase and γ phase as $[110]_{\gamma} // [\bar{1}\bar{2}10]_{\delta}$. The

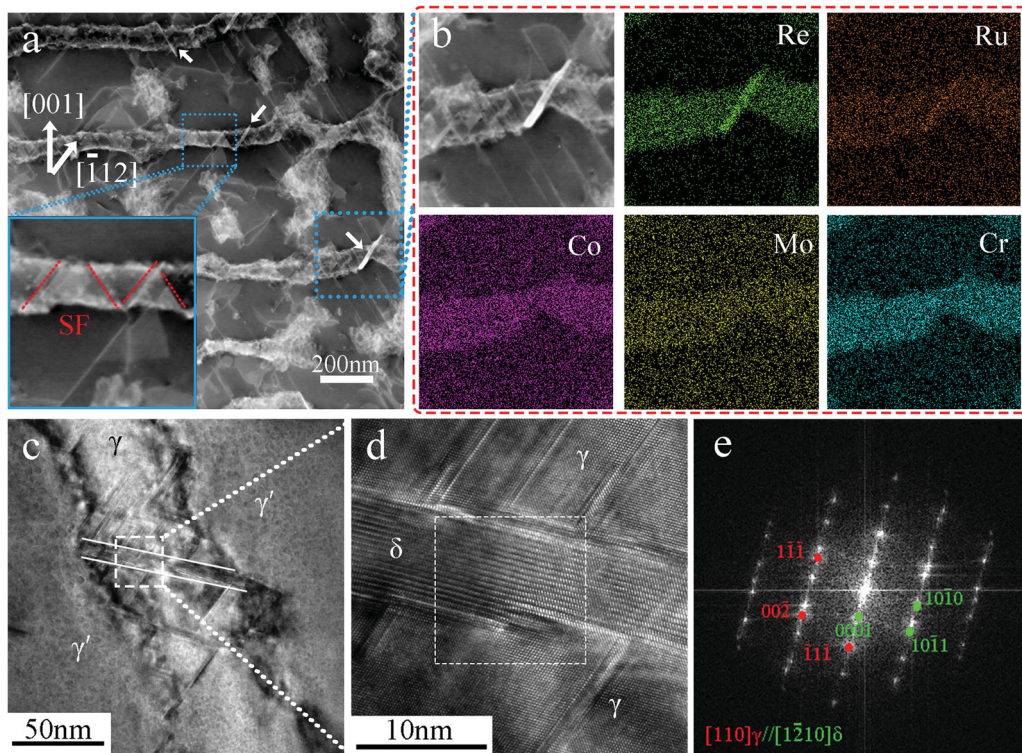


Figure 1. The morphology of δ phase precipitated within γ phase. (a) The scanning transmission electron microscopy (STEM) image of the morphology of δ phase in γ phase after creep rupture. The δ phase were marked by white arrows. The detailed morphology of stacking faults and δ phase were enlarged in the left corner. (b) EDS mapping results of the region marked by blue frame in (a). (c) The TEM image of δ phase in γ phase. White lines indicated the position of δ phase and the area marked by white frame was enlarged in (d). (e) The FFT image of the selected area in (d) marked by white frame.

crystal information of δ phase was extracted from Ref. [30], which was listed in Table 2. Additionally, the interplanar spacing of two phases was followed by $(\bar{1}1\bar{1})_{\gamma} // (0001)_{\delta}$, $d_{\gamma}(\bar{1}1\bar{1}) = d_{\delta}(0002)$.

The elemental segregation behavior in the SFs in the γ phase was investigated in the interrupted (550 h) sample in Figure 2. The white arrows in Figure 2(a) marked two SFs in the γ phase with a lock, which were further enlarged in Figure 2(b). The SF regions possessed brighter contrast compared to other regions in the γ phase, indicating that the segregation of refractory elements occurred in the SF regions. The EDS mapping results revealed that Re and Ru were segregated in the SFs. Such segregation was energy-favored and improves the creep strength [20,31]. Though Co and Cr could decrease the SF energy in γ phase, the stacking faults did not exhibit significant Co and Cr segregation. The elemental segregation tendency was related to the reduction degree of elements to the stacking fault energy. Calculation results have pointed out that Re possessed the most significant effect on decreasing the SFE in the γ phase [32,33] (see more details in Supplementary Note 1). Therefore, the thermodynamic driving force of Re segregating along the stacking faults in the γ phase was larger than Cr and Co. Due to the existence of the interaction between Re and Ru and the fact that Ru also decreases the SFE significantly [10,33,34], Ru segregated slightly along

Table 2. The crystal information of δ phase [30].

System	Space group No.	Space group	Lattice parameter	
			a	c
Hexagonal	194	P6 ₃ /mmc	2.73 Å	4.32 Å

the stacking faults. The segregation behavior in the lock was not more significant than in the SF regions, which differed from the results in Ref. [35]. With prolonged creep time and accumulated strain, the elemental segregation behavior might become more pronounced [35]. The elemental segregation behavior in the SF regions was similar to the δ phases in Figure 1, proving that the δ phases nucleated at the SFs. Moreover, the probability of δ phase precipitation near the lock might not be higher than in the other region of SFs, due to the similar segregation behavior between the lock and the SFs. Furthermore, the composition of SF and δ phase was compared in Figure S2. The Re and Ru content were higher in the δ phase than in the SF regions, indicating that further segregation in the SF could trigger the δ particle nucleation. The content of Cr and Co in the δ phase was slightly lower than in the SF regions. The calculated composition of the δ phase hinted that more Re and Ru might solute into the δ phase when reaching thermodynamic equilibrium.

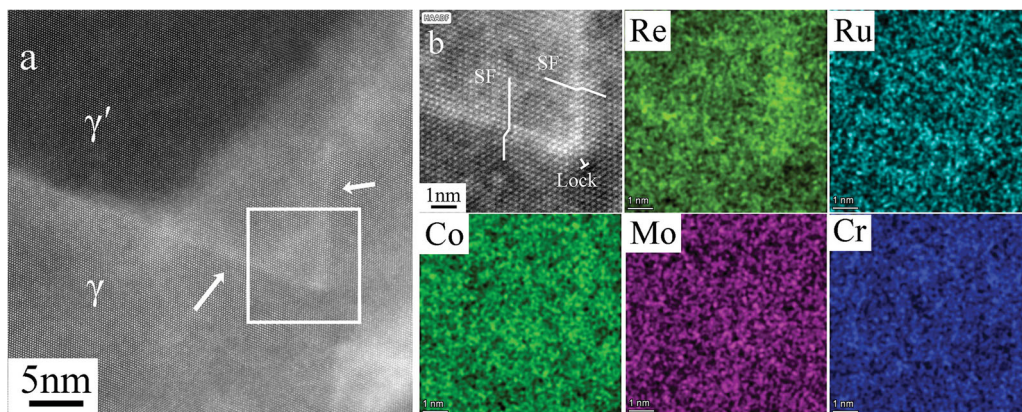


Figure 2. The elemental segregation behavior at SFs in γ phase. (a) The STEM image of SFs and lock in γ phase. The SF area marked by white frame was enlarged in (b). The EDS mapping results of SF were also exhibited in (b). White lines in (b) marked the atomic shifting in the SFs.

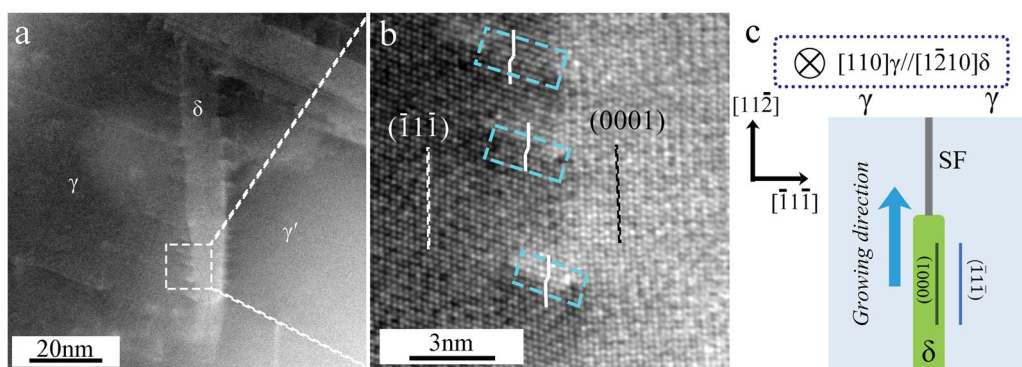


Figure 3. The high-resolution images of the δ phase and the interface structure in the ruptured samples. (a)(b) HRTEM images of δ phase and γ phase. The trails of SFs were marked by white lines and blue frames in (b). (c) The scheme of the growing direction of δ phase and the orientation relationship.

Cs-corrected scanning transmission electron microscopy (STEM) images in Figure 3(a and b) unveiled the atomic structure of the δ phase and the interface between the δ particle and the matrix. The interface was parallel to $(\bar{1}\bar{1}\bar{1})_\gamma$ and $(0001)_\delta$ and the morphology was relatively flat. The interface ledges rarely existed, indicating that the interface between $(\bar{1}\bar{1}\bar{1})_\gamma$ and $(0001)_\delta$ might possess relatively low interfacial energy [10,36]. Figure 3(c) illustrated the growing direction of the δ phase and the orientation relationship. The interface was parallel to the habit planes, indicating that the δ phase grew by expansion along $(\bar{1}\bar{1}\bar{1})_\gamma$ and $(0001)_\delta$. Interestingly, Figure 3(b) exhibited the intersection between the δ phases and SFs in the γ phase, hinting that the δ particle might impede the movement of SFs in the γ phase. Moreover, the bright contrast at the non-coplanar SFs indicated elemental segregation and the possibility that a new δ particle nucleated along the SF, based on the existing δ particle. The potential function of the δ phase on the creep properties requires further investigation.

From the results above, the structure and elemental segregation of SFs and δ phase were similar, hinting that the nucleation sites of these particles might be along the SFs in the γ phase. Due to the relatively low temperature, the diffusion rates of δ phase-forming elements were limited and the growth of δ particles was suppressed. Figure 4(a and b) compared the atomic structure of the SF and δ phase in detail. When observing along the direction of $[110]_\gamma$, the SF and the δ phase possessed a similar atomic configuration arranged in a zig-zag shape (marked by red dots). The model of the atomic arrangement of δ phase along $[1\bar{2}10]_\delta$ on the right side of Figure 4(b) further confirmed this structural similarity [30]. Furthermore, since the interface between the δ phase and the matrix was parallel to $(\bar{1}\bar{1}\bar{1})_\gamma$ and $(0001)_\delta$, the arrangement of atoms near $(\bar{1}\bar{1}\bar{1})_\gamma$ and $(0001)_\delta$ was also compared in Figure 4(c). The atomic configuration of $(\bar{1}\bar{1}\bar{1})_\gamma$ and $(0001)_\delta$ were almost identical, consisting of resembling triangular structural units with almost the same side length. Such similarity hinted that

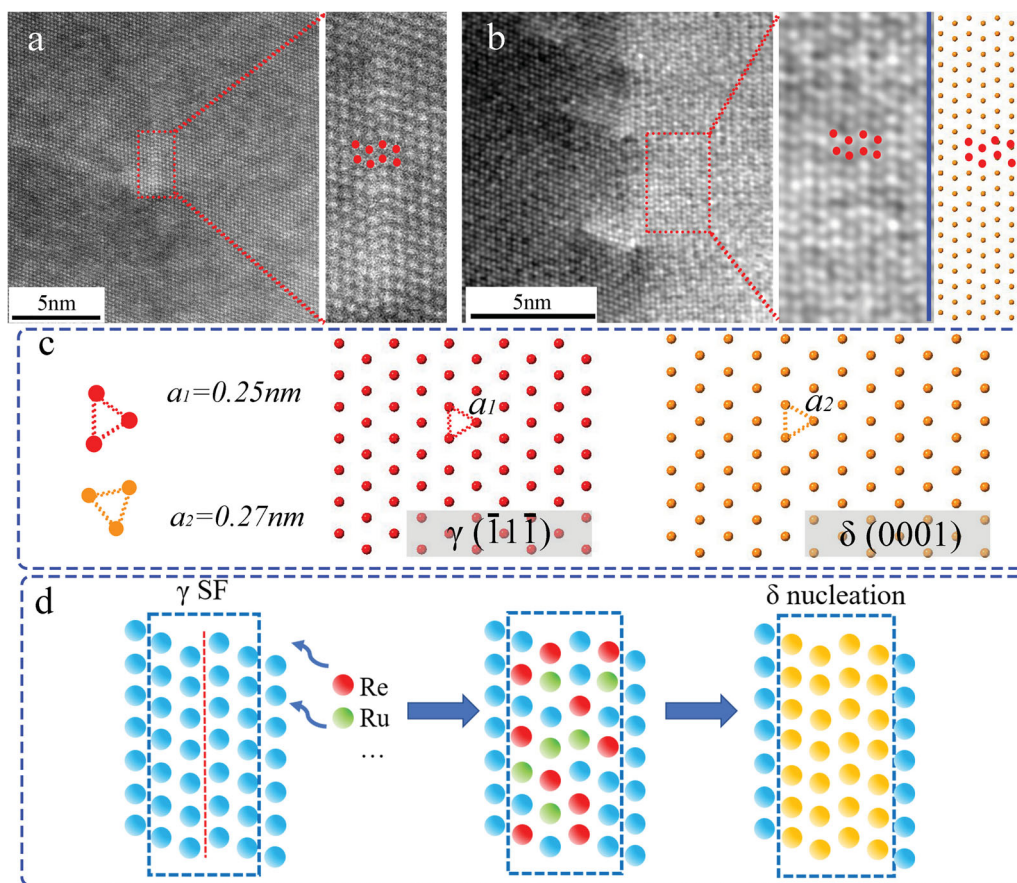


Figure 4. The comparison between the structure of (a) SF and (b) δ phase. (c) The atomic arrangement on the $(\bar{1}\bar{1}\bar{1})_\gamma$ and $(0001)_\delta$. (d) The scheme of the δ phase nucleation process.

during the phase transformation from the γ phase to δ phase, the change of atomic configuration from $(\bar{1}\bar{1}\bar{1})_\gamma$ to $(0001)_\delta$ might require little activation energy. The stacking faults in Ni-based SCs formed along the close-packed plane, i.e. the 111 planes, which had a quite similar atomic arrangement to $(0001)_\delta$. The SF structure and δ phase both possessed hexagonal characteristics. Therefore, the δ phase had the unique orientation relationship exhibited in Figure 1(e). The rod shape along $\langle 112 \rangle$ direction in Figure 1 was determined by the direction of SFs, which was mainly on the $\langle 112 \rangle$ 111 slip systems in Ni-based superalloys [24,37,38]. Combining Figures 1 and 2 and Figure 4, it could be concluded that the SF in the γ phase promoted the nucleation of the δ phase, as shown in Figure 4(d). The stacking faults formed in the γ phase when external stress was applied. Re and Ru atoms diffused to the regions of SFs to decrease the system energy. Therefore, the segregation of Re and Ru and the structural similarity between the SFs and δ phase promoted the precipitation of the δ phase. However, it was noteworthy that the δ particle had a width that was significantly wider than the SFs. The thickening process still requires

further investigation, which might be related to the formation of the interfacial steps [10] and the elemental diffusion process.

Figure 5 exhibited the relationship between the volume fraction of the δ phase and the content of Re and Ru in our alloys system calculated by ThermoCalc. The equilibrium volume fraction of the δ phase in the investigated alloy is about 4 vol.%. The addition of Re and Ru both benefit the precipitation of the δ phase, consistent with the observation results. With Re content increasing, the content of the δ phase slowly increases. However, Ru has a more facilitating effect on δ phase precipitation than Re, hinting that δ phase is more likely to precipitate in new generation Ni-based SCs with high Ru contents.

In summary, the effects of stacking faults in the γ phase on the precipitation of the δ phase were analyzed in detail. During the creep process at 800 °C and 735 MPa, the stacking faults appeared in the γ phase and refractory elements such as Re and Ru segregated in the SF regions. δ phase with a similar elemental segregation tendency emerged along the direction of SFs. The elemental segregation and the structural similarity between the SF

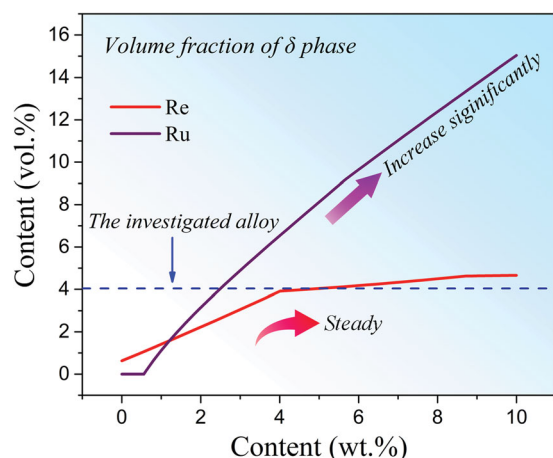


Figure 5. Calculation results of the relationship between the δ phase content and the content of Re and Ru.

regions and the δ phase provided the structural origins of the nucleation. The δ precipitates were short rods aligning along the $(\bar{1}1\bar{1})_{\gamma}$ with an orientation relationship of $[110]_{\gamma} // [1\bar{2}10]_{\delta}$ and $(\bar{1}1\bar{1})_{\gamma} // (0001)_{\delta}$. The structure of $(\bar{1}1\bar{1})_{\gamma}$ and $(0001)_{\delta}$ possessed similar structural units, facilitating the nucleation process. Furthermore, the influence of Re and Ru contents on the formation of the δ phase was calculated. Compared to Re, Ru possesses a more significant promoting effect on the precipitation of the δ phase, which provides a new perspective for the alloy design.

Data availability

The data are available from the corresponding author upon reasonable requests.

Disclosure statement

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

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