

Supplementary Materials for
**Topologically Inverse Microstructure in Single-crystal Superalloys:
Microstructural Stability and Properties at Ultrahigh Temperature**

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1. Detailed materials and methods

Selection of alloy system: On the one hand, Ni-Al-Mo series single crystal superalloy has great potential to service above 1200 °C. This alloy system is not newly invented, and was systematically studied for the first time at 1990s, by R.A. MacKAY [1]. Since the late 1990s, Y.F. Han has developed one Ni-Al-Mo series superalloy to an engineering-used designation, IC6 [2]. This alloy system has advantages of low density, low cost, high specific strength and good thermal strength. Recently, researchers are committed to improve high temperature properties of Ni-Al-Mo series alloys. H. Zhang [3] has enhanced the 1100 °C creep properties of one Ni-Al-Mo series alloys, and our previous work [4] has successfully raised the service temperature to 1200 °C. Meanwhile, this alloy system has been gradually conducted to engineering applications.

On the other, this alloy system exhibits both the microstructural evolution and dislocation behavior same to conventional superalloys. This means that the plastic deformation behavior and strengthening mechanism obtained from this work are able to extend to conventional superalloys.

Therefore, this work chooses Ni-Al-Mo series quinary alloys, and has proposed a 1200 °C-serviced, conventional alloy-applicable design strategy.

Materials: Alloy SMSC and TIMSC and other counterpart alloys are investigated in this work. The counterpart alloys include SMSC-0.3Al (reducing 0.3 w.t. % Al in Alloy SMSC), SMSC-1Ta (reducing 1 w.t. % Ta) and SMSC+1Ta (adding 1 w.t. % Ta). The master alloys were produced from pure metals (99.99 at %) by vacuum induction melting (VIM). The single-crystal rods were achieved by high rate solidification (HRS) using screw selecting method, with withdrawal rate of 3 mm/min. The standard heat treatments were carried out as follow:

- Alloy TIMSC: 1295 °C/ 2 h + 1315 °C/ 2 h + (1325-1345) °C/ 10 h, accelerated air cooling (a.a.c.) + 1200 °C/ 1 h, a.a.c. + 870 °C/ 32 h, a.a.c.
- Alloy SMSC: 1315 °C/ 4 h + (1325-1335) °C/ 10 h, a.a.c. + 1200 °C/ 1 h, a.a.c. + 870 °C/ 32 h, a.a.c.
- Other counterpart alloys: 1300 °C/ 4 h + (1325-1340) °C/ 10 h, a.a.c. + 1200 °C/ 1 h, a.a.c. + 870 °C/ 32 h, a.a.c.

The crystallographic orientations of these rods were determined by Laue diffraction analysis. Only the samples with the orientations within 8° deviating from the [001] orientation were chosen for our investigation.

Experimental: The samples were submitted to iso-thermal exposure test on the

condition of 1200 °C/ 100 hours and water quenched quickly. The samples for stress rupture tests were prepared with a cylindrical gage section in a length of 25 mm and a diameter of 5 mm, according to the National Standard (GB/ T 2039-2012). The 1200 °C/ 60 MPa stresses rupture test was carried out. The temperature deviation in the sample was within 3 °C by using a temperature controller and type S thermocouples. When the temperature comes up to the target 1200°C, the furnace temperature was held 30 min for homogenization. Each alloys was tested at least three times and the average of stress rupture properties was used.

Characterization: The heat-treated, exposed or ruptured specimen cut transversally or longitudinally and were prepared mechanically and chemically for characterization. The microstructure and dislocation configuration were observed using scanning electron microscopy (SEM, JEOL CamScan-3400) and transmission electron microscopy (TEM, JEM-2100F, operated at 200 kV), respectively. The interface density ρ_{int} , defined in this work equals to interface line length per area in SEM images. By using ImageJ software programs, the SEM image transformed to a black-and-white image, and the boundary between black and white regions was identified. The total length of the boundary divided by the image area was then calculated. At least 10 images were collected and analyzed for each specimens and the average of interface density data was obtained. The dissolution temperature of γ' -phase is determined by differential scanning calorimetry (DSC) at a heating rates of 10 K/min.

Filtered Fast Fourier transformation (fFFT) technology: This technology is specific for studying the periodicity. Epishin et al. [5] firstly introduced this technology to analysis periodical γ - γ' morphology during microstructural evolution. The Fast Fourier transformation (FFT) is applied via a fast algorithm to calculate amplitude spectra, as $F(\vec{g}) = \int f(\vec{r}) \exp(-i\vec{g}\vec{r}) d\vec{r}$, where $f(\vec{r})$ is SEM image intensity in real space with coordinates $\vec{r}$, and $F(\vec{g})$ is FFT amplitude in reciprocal space with coordinates $\vec{g}$. The “filtered” modification is a process to remove these low amplitudes, to clarify the geometric symmetry of FFT spots. For superalloys, the fFFT transformation of γ - γ' morphology image constructs a reciprocal space, and generates a spectrum consisting of discrete points. The angular position of one discrete point in a reciprocal space represents the direction normal to the corresponding γ/γ' interfaces in a real space, and the deviation of points from these principle orientations reflects interface distortion.

2. Straightness of γ/γ' -interface and its change

The smooth and straight interfaces that surround cuboidal γ' -phases have been regarded the optimal morphology of superalloys, and this is achieved mostly via alloying design optimization for controlling interface misfit [6]. However, this straightness of interfaces will lose during high-temperature service:

(i) The spheroidization of γ' -phases occurs in a coarsening process. The corners of cuboidal γ' -phases starts to dissolve initially, due to concentration of misfit stresses.

(ii) The interfaces become zigzag. Many crystal defects, such as vacancy and dislocations, react with the interfaces, which produces multi-scaled wavy interfacial structures including grooves [7], ledges [8], kinks [9], protrusions [10] and pores [11].

Above two ways break the straightness of interfaces, usually regarded as signs of structural evolution during high-temperature service. Thus, the straightness of interfaces in **Figure 1c** in the manuscript indicates that the microstructure in Alloy TIMSC is close to the state of standard heat treatment.

3. Specific connectivity number of γ -phase

In order to quantitatively describe levels of topological inversion completion, a specific connectivity number of γ , denoted as $N_A(\gamma)$, is introduced [12]. It is noteworthy that the main phase chosen to analysis connectivity is γ' phase in ref. [12], but γ phase in our work, owing to better research of topological inversion. $N_A(\gamma)$ is defined as the number of connected particles of γ phase per area unit, and can be calculated via

$$N_A(\gamma) = \frac{N_T - N_{TP}}{2S} \quad (1)$$

where N_T is the number of γ -terminations, N_{TP} is the number of triple points of γ -phase, and S is the area of analysis field.

Figure 1S shows the analysis for the evaluation of $N_A(\gamma)$. **Figure 1Sa** is a given SEM image. **Figure 1Sb** is obtained through transforming to a black-and-white image and marking the terminations and triple points of γ -phase, by the signs $\bullet$ and $\blacktriangle$ respectively. **Figure 1Sc** is another example for analysis. After counting N_T and N_{TP} and taking into Eq. (1), the $N_A(\gamma)$ is determined.

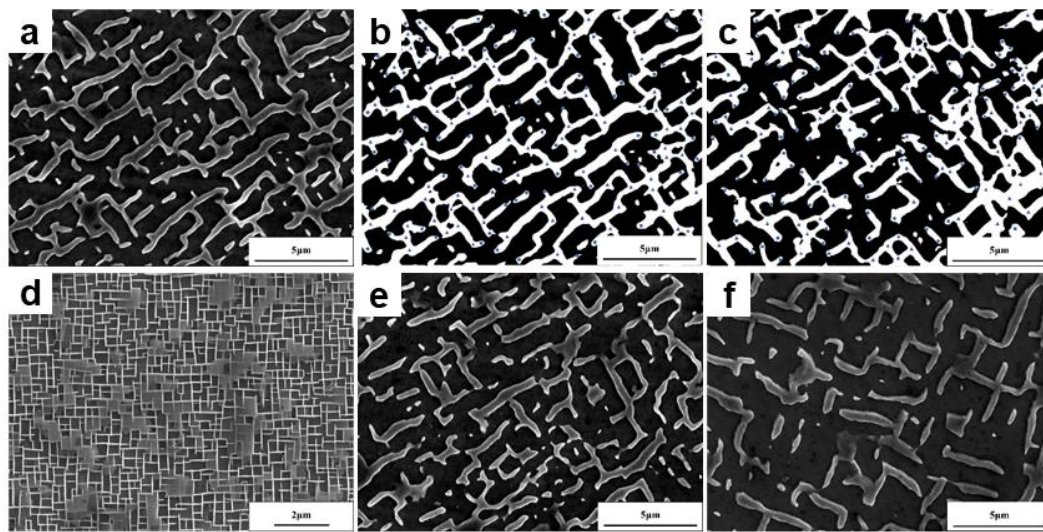


Figure 1S. a) One two-phase morphology of a SEM image; b) and c) image analysis for the evaluation of $N_A(\gamma)$, by counting of the terminations ($\bullet$) and triple points ($\blacktriangle$) of γ phases; typical two-phase morphology with different $N_A(\gamma)$ values: (d) $N_A(\gamma) < -0.02$, (e) $-0.02 < N_A(\gamma) < 0$ and (f) $N_A(\gamma) > 0$.

In this work, $N_A(\gamma)$ is negative when the γ phases are connected to matrix and changes to positive during topological inversing. **Figure 1Sd-e** shows the typical two-

phase morphology with different $N_A(\gamma)$ values. When $N_A(\gamma) > 0$, the isolated γ -phase is embedded in fully-connected γ' -matrix, as topological inversion occurs. When $N_A(\gamma)$ is less than a specific negative value, the microstructure consists of normal γ -matrix and γ' -precipitates. There is a transition state between above two conditions, where γ -phases remain low connectivity while γ' -phases have great connectivity. It is hard to find out the exact value of such specific negative value. After incomplete statistics of the relation between $N_A(\gamma)$ and two-phase morphology, this specific negative value that represents γ' -connectivity completion is assigned -0.02 in this work.

4. Doubled creep properties between adjacent generations superalloys

The current alloying design to upgrade superalloys to next generation is adding about 3 w.t. % Re or Ru, and the high temperature creep properties have been nearly doubled between superalloys [13, 14]. For instance, the 1100 °C/ 137 MPa creep lives of third-generation single-crystal superalloys CMSX-10 (being 309 hours) and Rene N6 (192 h) are approximately the double of the second-generation CMSX-4 (137 h) and Rene N5 (88 h) [15-17], respectively, and the creep life of fourth-generation TMS-138 (412 h) doubles than third-generation TMS-75 (227 h) as well [18].

5. Discussion of microstructural stability and evolution

5.1 Two-phase microstructural evolution

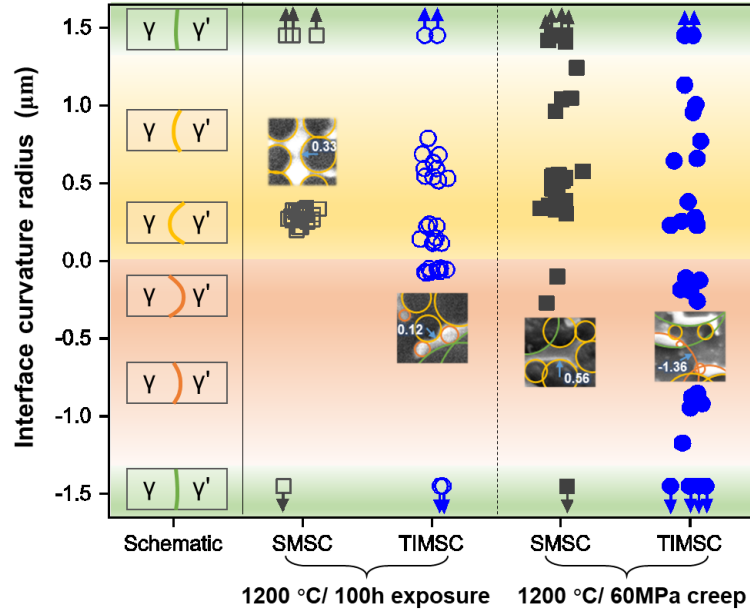


Figure 2S. Interface curvature radii in two alloys after exposure or stress rupture tests, providing an evidence of complicated coarsening behavior in the inverse structure.

Recalling **Figures 1d-f** in the manuscript, the different particle-coarsening processes in two alloys lead to the different features of interface distortion. Simply put, the spheroidizing and connecting of γ' -precipitates generate the distortion in Alloy SMSC, while the spheroidizing of γ -terminations in Alloy TIMSC exists, also contributing to interface distortion. In order to investigate such different behaviors, the interface curvature radius (r) is defined as a positive value when the interface protrudes to γ -phase and as a negative one when protruding to γ' -phase. **Figure 2S** shows the incomplete statistical data of r in two alloys. After exposure, almost all the interfaces in Alloy SMSC is r -positive, while nearly one-third of the interfaces in Alloy TIMSC is r -negative. When subjected to 1200 °C/ 60 MPa stress rupture test, the almost straight interfaces ($|r| > 1.5 \mu\text{m}$) are presented in two alloys, due to rafting or raft-like process. There still exists an approximately half-and-half combination of r -negative and -positive interfaces in Alloy TIMSC, in comparison with Alloy SMSC whose interface is of positive r .

According to Lifshitz-Slyozov-Wagner particle-coarsening theory [19, 20], for a matrix-precipitate system, the ripening precipitate is believed to be spheroidized and

the interface is to protrude to matrix phase. In our work, the positive or negative r -values theoretically reflect γ' - or γ -coarsening process. The r -positive interfaces are dominant in Alloy SMSC, which indicates that the main ripening phase is γ' -phase, being in line with classical particle-coarsening behavior of superalloys. However, both the positive and negative characters of r are presented in Alloy TIMSC, none character being dominant. It is suggested that the main ripening phase in topologically inverse alloys is not simply either γ' -phase or γ -phase, and the microstructural evolution maybe a result of comprehensive effects of single-phase coarsening or the competition between them. Further studies will be needed to elucidate the mechanism of microstructural evolution.

5.2 Precipitation behavior of Topologically Close-Packed phases

Except two-phase microstructural evolution, the microstructural stability includes Topologically Close-Packed (TCP) phase precipitation. TCP phases are abnormally-precipitated, Re, W, Cr-rich intermetallic phases, and causes performance degradation in the temperature range of 950-1100 °C [21, 22], in part due to (i) the brittleness of TCP, (ii) stress concentration and (iii) consuming a large number of solid-solution-strengthening elements. This effect is directly determined by the volume fraction of TCP. M.S.A. Karunaratne et al. [23] investigated the TCP precipitation behavior in Re-containing single-crystal superalloys and plotted the Time-Temperature-Transformation diagram for the onset of TCP precipitation. There is a strong temperature dependence: the temperature increasing from 800 to 1050 °C enhances precipitation activity, while further increased temperature causes the dissolution of TCP, in especial its complete dissolution at 1200 °C. This thermal instability above 1180 °C is common in single-crystal superalloys, including CMSX-4 [24, 25], MC2 [26-28] and MC-NG [29, 30]. In our investigated alloys, only a small amount of TCP phase is formed during 1100 °C/ 1000 hours long-term thermal exposure, with the volume fraction less than 1%, and no TCP phase is presented during the stress rupture test and thermal exposure at 1200 °C. This phenomenon is also found in other Ni-Al-Mo series superalloy IC6 [2].

Therefore, our investigated alloys have great microstructural stability of no TCP precipitation, and the harmful effect of TCP can be eliminated in this work.

6. Dislocation configuration in two phases

6.1 Dislocation configuration in γ phase

The contrast of γ -phases in **Figure 4a and 4b** is remarkably black, which demonstrates complex dislocation motion confined in there during testing. When submitted to ultrahigh temperature and low stress, such as creep, the FCC-disordered, Ni-based γ phase deforms plastically, via dislocation initiation, multiplication and motion. Some moving dislocations meet γ/γ' interfaces, and then wave to network to release the misfit-induced interfacial stress. Therefore, the deformed γ phase exhibits both the dense dislocations and the interfacial networked dislocations. It is important to notice that this two kinds of dislocations have different properties: those in γ phase keep significantly movability while networked ones lose the movability. During the cooling process after ruptured, the significant dynamic recovery of dislocations in γ phase operates, but it is hard to recover for stable networked dislocations. This finally results in the phenomenon that the misfit dislocation networks is more visible than dislocations in γ phase.

6.2 Dislocation configuration in γ' phase

The creep plastic deformation of single-crystal superalloys has been primarily confined in the γ -phase, rather than γ' -phase. The main driving force which prevents dislocations' penetration of γ' -phase, is antiphase boundary (APB) energy and stacking fault (SF) energy of $L1_2$ -ordered γ' -phases. The formation of two planar defects results from different dislocation behaviors in the ordered structure. Why pair-coupling of dislocations in γ' -phases is presented in our work is briefly discussed as follow.

When one $\frac{a}{2}\langle 110 \rangle$ dislocation shears γ' -phase via a $\{111\}$ plane, the area where the dislocation passes is the APB [31]. Two common ways are able to reduce and eliminate this APB energy: slipping via a $\{001\}$ plane and establishing pair-coupling structure [32]. The latter is presented in the **Figure 4** of this manuscript, which is the evidence of that APB energy can hinder dislocation penetration.

The formation mechanism of meso-scale SF in γ' -phase is firstly proposed by Kear et al. [33] as

$$\frac{1}{2}[011] + \frac{1}{2}[\bar{1}01] \rightarrow \frac{1}{3}[\bar{1}12] + SF + \frac{1}{6}[\bar{1}12] \quad (2)$$

and then modified [34] as

$$\frac{1}{2}[011] \rightarrow \frac{1}{3}[\bar{1}12] + SF + \frac{1}{6}[21\bar{1}] \quad (3)$$

Both two models occur in γ/γ' interfaces, and the resulting $\frac{1}{3}[\bar{1}12]$ and $\frac{1}{6}\langle\bar{1}12\rangle$ are penetrating the precipitates and left at the interface, respectively. The two driving forces of this reaction are *i*) relatively high stress applied, which generates massive reactive dislocations and operates slipping of the resulting dislocation, and *ii*) more important, low SF energy. Thus, low stress and high SF energy can reduce and eliminate the SF formation in mesoscopic scale (atomic-scale dislocation dissociation is always required to overcome SF energy, but this SF cannot be visible in bright or dark field images of TEM).

Such is quite common that meso-scale SF formation depends on creep conditions: it occurs at the conditions of low temperature (700-850 °C) and high stress (>500 MPa) [35-37], while disappears at high temperature (~1100 °C) and low stress (~137 MPa) [18, 38]. This is ascribed to the increase in SF energy with the temperatures rising [39]. Of course, the decreased stress applied in high-temperature creep maybe also contributes to SF absence. In order to clarify it, another work dealt with tensile behavior at different temperatures of single-crystal superalloys [40] is cited here. It is found that the SF indeed disappears at temperatures above 1000 °C, although the applied stress reaches the yield strengths (at least 3 times higher than creep stresses) within the final stage of rupture. This results strongly support that the increase of SF energy is the main reason for SF absence. Therefore, no meso-scale SF formation in γ' -phase of our alloys can be well explained by the far high SF energy at ultra-high temperature as 1200 °C.

7. Lattice misfit calculated by interfacial dislocation network

The interfacial lattice misfit δ determines the average spacing d of dislocation networks [18, 41]. One has $\delta = -|\vec{b}|/d$, where $|\vec{b}|$ is the magnitude of $1/2 a\langle 110 \rangle \{111\}$ dislocation [42, 43]. The spacings in the (001)-interfaces of Alloy SMSC, the (001)- and (010)-interfaces of Alloy TIMSC are 45.3 ± 3.2 nm, 41.8 ± 2.7 nm and 44.1 ± 4.9 nm, respectively. The difference of these misfit values is quite slight, just about relatively 8 % (from -6.07% to -5.61%).

8. Calculation of creep rate of Ni solid solution

Ernst Fleischmann et al. [44] determined the 980 °C/ 30–75 MPa creep rates in many single-phase single crystals with the compositions of the nickel solid-solution matrixes in superalloys. The solubility degree of γ -phase at 1200 °C in our investigated alloys is close to MSX W11 (as the γ -matrix of MC2), MSX W14 and MSX W5 in their work, whose creep rates at 980 °C/ 60 MPa were $2.04 \times 10^{-5} \text{ s}^{-1}$, $2.56 \times 10^{-5} \text{ s}^{-1}$ and $4.90 \times 10^{-5} \text{ s}^{-1}$, respectively. The 980 °C/ 60 MPa creep rates of γ -phases in our alloys are thus set in a possible range of $2.00 \sim 5.00 \times 10^{-5} \text{ s}^{-1}$. Referring to Larson-Miller relation, Larson-Miller parameter, which is a function of temperature and rupture life, is fixed when applied stress is unchanged. Based on this, 1200 °C creep properties can be predicted from 980 °C properties under same applied stress as 60 MPa. The calculated creep life of γ -phase in our alloys is in a possible range of $2.0 \sim 4.4 \times 10^{-3}$ hours.

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