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### Regular Article

## The effect of  $\text{Ni}_3(\text{Cr}_{0.2}\text{W}_{0.4}\text{Ti}_{0.4})$  particles with  $\text{DO}_{22}$  structure on the deformation mode and mechanical properties of the aged Ni-Cr-W-Ti alloy

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#### article info abstract

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Ni-Cr-W-Ti alloy Nanoscale DO<sub>22</sub> precipitates Deformation twinning Partial dislocations Transmission electron microscopy

It has been found that the  $DO_{22} Ni_3(Cr_{0.2}W_{0.4}Ti_{0.4})$  particles play a dominant role in improving the strength of the aged Ni-Cr-W-Ti superalloy. The precipitation of DO<sub>22</sub> phase also changed the preferred deformation mode from slip in the as-solutionized alloy into twinning in the aged alloy. Strengthening of the aged superalloy has been determined to be based on shearing mechanism to achieve high cutting stress which is required for partial dislocations to cut through coherent  $DO_{22}$  precipitates and thus produce plastic deformation, resulting in a dramatic increase in yield strength of the aged superalloy.

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Alloys based on Ni-Cr-W system constitute a very important class of superalloys that have great potential in the field of chemical and nuclear industry, due to their superior corrosion and creep resistance, and better tensile properties at high temperature [1[–](#page--1-0)4]. It has been proved that the nanoscale superlattice phases precipitate in some Ni-Cr-based and Ni-Mo-based superalloys through ordering reaction, and these ordered particles lead to an increase in yield strength by about 2-fold over that of the solid solution strengthened alloy [5[–](#page--1-0)8]. Nevertheless, there is a limited study pertaining to ordering reaction in Ni-Cr-W-based superalloy during prolonged exposure to proper temperature, which has been down by our research group [\[9\]](#page--1-0). And we revealed that the nanoscale  $DO<sub>22</sub>$  Ni<sub>3</sub>  $(Cr_{0.2}W_{0.4}Ti_{0.4})$  phase with high thermal stability can precipitate in an aged Ni-Cr-W-Ti alloy. However, the effect of the  $DO_{22}$  particles on the mechanical properties and deformation mode of the Ni-Cr-W-based superalloy has never been studied. In fact, the interaction between superlattice phase and dislocation during the plastic deformation has also rarely been studied in the well-studied Ni-Cr-based and Ni-Mo-based superalloys.

In the present paper, we demonstrated that  $Ni<sub>3</sub>(Cr<sub>0.2</sub>W<sub>0.4</sub>Ti<sub>0.4</sub>)$ phases formed in the aged Ni-Cr-W-Ti alloy lead to an increase in yield strength by about 4-fold over that of the solid solution strengthened alloy. The effect of the ordering transformation from fcc to  $DO<sub>22</sub>$ on the deformation mode in each structure was discussed in detail. We also discovered the superlattice particles were cut through during the plastic deformation, which has never been found in past few studies

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<https://doi.org/10.1016/j.scriptamat.2018.04.029> 1359-6462/© 2018 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved. [\[5](#page--1-0),[7,8\]](#page--1-0). The objectives of this present study were mainly focus on that how does the  $DO<sub>22</sub>$  phase effect the deformation mechanism and mechanical properties of the Ni-Cr-W-Ti superalloy.

The prepared alloy in this work had the composition Ni-26Cr-6W-2Ti (in at.%). The alloy ingots were prepared by arc-melting a mixture of pure metals (purity larger than 99.9%) under an argon atmosphere, and re-melted at least four times in order to ensure homogeneity. The alloy ingots were polished and cleaned by ethanol, and then cast into plates (50  $\times$  20  $\times$  3 mm<sup>3</sup>) through the vacuum suction casting. The plates were homogenized at 1200 °C for 24 h in a vacuum environment and furnace cooled to room temperature, and then annealed at 1250 °C for 1 h, followed by water quenching. Subsequently, the as-solutionized plates were aged at 700 °C for 240 h then air cooled (named as the aged alloy for simplicity in this letter).

Dog bone-shaped tensile samples with a gauge length of 14 mm, a width of 2 mm and a thickness of 1.5 mm were prepared from the assolutionized and aged plates. A MTS SANS-ACMT5105 testing machine was employed for tensile tests at room temperature with a nominal strain rate of  $1 \times 10^{-3}$  s<sup>-1</sup>. The tensile tests of the as-solutionized and aged specimens were performed for at least four times in order to ensure its precision. The deformation substructures of specimens were characterized by transmission electron microscopy (TEM).

For TEM, disks of 3 mm diameter were punched out from thin foils of 65 μm thickness. Electron transparent samples for TEM investigation were electropolished in a dual-jet electropolisher using a solution of 10 vol% perchloric acid in 90 vol% ethanol at −30 °C. TEM experiments were performed using an FEI Tecnai  $G^2$  F30 microscope operating at







300 kV. Standard TEM techniques such as Bright-field (BF) and Darkfield (DF) imaging, selected area electron diffraction (SAED) and high resolution transmission electron microscopy (HRTEM) were used to characterize the microstructure.

Fig. 1(a) provides the uniaxial tensile engineering stress-strain curves of the as-solutionized and aged specimens. The yield strength, ultimate tensile strength and elongation of as-solutionized alloy are about (274  $\pm$  7) MPa, (559  $\pm$  6) MPa and (44.6  $\pm$  1.7)%, respectively. For the aged alloy, yield and ultimate tensile strengths are even higher about (1021  $\pm$  6) and (1115  $\pm$  8) MPa, respectively, while the elongation is a about (11.6  $\pm$  1.2)%. The yield strength of over 1 GPa is particularly noteworthy since such high yield strength has not been reported in Ni-Cr-Mo-based superalloys before. Fig. 1(b) shows a BF image of the as-solutionized alloy and demonstrates absence of any precipitates. The corresponding SAED pattern along [001] zone axis of fcc matrix in Fig. 1 (c) reveals diffraction spots corresponding only to the Ni-based matrix. Electron diffraction pattern along [001] zone axis of the aged alloy, shown in Fig. 1(d), reveals the presence of superlattice reflections at the  $\{110\}, \{11/20\}, \{100\}$  positions, corresponding to tetragonal DO<sub>22</sub> structure. The DF micrograph in Fig. 1(e) imaged using superlattice reflection marked by white circle in Fig. 1(d), reveals the nanoscale  $DO<sub>22</sub>$  particles (bright contrast) with high number density are dispersed in the Ni-based matrix homogenously. The chemical formula of the  $DO<sub>22</sub>$  phase was revealed to be  $Ni<sub>3</sub>(Cr<sub>0.2</sub>W<sub>0.4</sub>Ti<sub>0.4</sub>)$  [\[9](#page--1-0)]. The low magnification optical microscopy (OM) images of the both as-solutionized and aged alloys showed that the grain morphology of them was very similar with each other. The temperature of 700 °C at which the Ni-Cr-W-Ti alloy was heat treated cannot make the microstructure of the aged superalloy have a significant change in contrast to that of the assolutionized alloy, but only make the matrix precipitate the nanoscale superlattice phases. Therefore, it can be concluded that the nanoscale  $DO_{22}$  precipitates lead to an increase in yield strength of the aged alloy by about 4-fold over that of the as-solutionized alloy. Strengthening of the aged superalloy is based on bypass and shearing mechanism to achieve a required stress [[10,11\]](#page--1-0), which is required for dislocations to bypass or cut through coherent  $DO<sub>22</sub>$  precipitates and thus produce plastic deformation. Moreover, it is also demonstrated that work hardening contributes to a much increase in the strength of the as-solutionized alloy as compared to the aged alloy. In order to determine the interaction mechanism between dislocations and  $DO_{22}$  precipitates definitely, investigations on deformation substructures of the aged superalloy have been carried out.

[Fig. 2\(](#page--1-0)a) and (b) show the characteristic features of the deformation substructure of the specimens after tensile test at room temperature. High density dislocations appear in the as-solutionized alloy shown in [Fig. 2\(](#page--1-0)a), while a large number of deformation twins are observed in the aged alloy shown in [Fig. 2](#page--1-0)(b), which is very similar to the results obtained by Tawancy et al. [\[5,7,8](#page--1-0)]. It was also revealed that the twinning deformation of the  $DO_{22}$  superlattice phases occurred according to the SAED pattern in the inset in [Fig. 2\(](#page--1-0)b).

The DF micrograph in [Fig. 3\(](#page--1-0)a) imaged using superlattice reflection marked by white circle in the inset in Fig.  $2(b)$ , reveals most  $DO<sub>22</sub>$  particles (marked by white arrows) seem to be cut through by deformation twins. [Fig. 3](#page--1-0)(b) shows the lattice image of deformation twin and fast Fourier transformation (FFT) diffractogram of the examined area indicated by marked square. From the SAED pattern in [Fig. 2](#page--1-0)(b) and FFT diffractogram in Fig.  $3(b)$ , it is demonstrated that twinning of  $DO<sub>22</sub>$ phase itself can occur when the precipitates are cut through, which is quite different from the results obtained by Tawancy et al. [[5](#page--1-0),[7,8\]](#page--1-0). All that mentioned above indicate that strengthening of this superalloy mainly follow the shearing mechanism.

The accumulation of dislocation in the sample during plastic deformation is responsible for the obvious work hardening in the tensile stress-strain curve [12–[14](#page--1-0)]. In the as-solutionized alloy, dislocation motion in the form of pile-ups on multiple slip systems results in the formation of Lomer-Cottrell type barriers created by intersecting dislocations from different slip systems [\[15](#page--1-0)]. However, most of slip systems are suppressed due to the precipitation of  $DO<sub>22</sub>$  superalttice phases, which lead to a slight strain hardening. All that mentioned above also further verify the deformation modes of the as-solutionized and aged alloys follow slip and twinning mechanisms, respectively. It has been reported that deformation twinning in fcc metals is rarely observed, especially in those with medium or high stacking fault energy, such as Cu and Ni [[16\]](#page--1-0). This is mainly due to the existence of many independent slip systems making slip a very sufficient deformation mode [\[8,17](#page--1-0)]. It is the precipitation of nano-scale  $DO_{22}$  superlattice phases with high number density that change slip systems of the aged alloy and make the deformation mode change from slip into twinning.

The change in deformation mode is correlated with the crystallography of the fcc to  $DO<sub>22</sub>$  transformation as illustrated in [Fig. 4](#page--1-0) (a) and (b). [Fig. 4\(](#page--1-0)a) shows arrangements of atoms on  $(1\overline{1}1)_{\text{fcc}}$ plane before ordering transformation, revealing three {111}〈110〉 slip systems and three {111}〈112〉 twinning systems. However, deformation by slip can occur more easily than by twinning in disordered Ni-based matrix due to the occurring of slip requiring lower stress [\[8,17\]](#page--1-0). After ordering transformation, the Cr/W/Ti atoms are arranged in periodic fashions in Ni-based matrix. As shown in [Fig. 4\(](#page--1-0)b), two  $\{111\}\langle 110 \rangle$  slip systems including  $(111)[011]$  and  $(111)[101]$  are



Fig. 1. (a) Engineering stress-strain curves of the as-solutionized and the aged alloys. (b) and (c) showing BF image and the corresponding SAED pattern of the as-solutionized alloy. (d) and (e) showing the SAED pattern and DF image of the aged alloy.

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