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Enhanced thermal stability and ductility in a nanostructured Ni-based alloy

Yanle Sun [a,b,c,d], Liming Fu [a,b,c], Zhiqiang Fu [d], Aidang Shan [a,b,c,*], Enrique J. Lavernia [d,**]

[a] School of Materials Science and Engineering, Shanghai Jiao Tong University, Shanghai 200240, People's Republic of China
[b] Collaborative Innovation Center for Advanced Ship and Deep-Sea Exploration(CISSE), Shanghai 200240, People's Republic of China
[c] Shanghai Key Laboratory of High Temperature Materials and Precision Forming, Shanghai Jiao Tong University, Shanghai 200240, People's Republic of China
[d] Department of Chemical Engineering and Materials Science, University of California, Irvine, Irvine, CA 92697, USA

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ABSTRACT

In this investigation we address the question of whether it is possible to simultaneously enhance the ductility and thermal stability of nanostructured (NS) Ni-based alloy by using an approach that involves the introduction of nano-precipitates. To address this question, both NS and ultra-fine grained Ni-based alloys with nanoscale γ'-precipitates were obtained via heat treatment of single-phase NS Ni-based alloy (~50 nm). Our results show that the thermally-stable γ'-precipitates contribute to both ductility and a remarkably high thermal stability close to 700 °C (0.62 T_m). The underlying mechanisms that are believed to be responsible for this behavior are discussed.

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The high density of barriers to dislocation motion (e.g., grain boundaries) that is characteristic of bulk nanostructured (NS) materials provide an opportunity to attain remarkable strengths, according to the Hall-Petch relationship [1,2]. Interestingly, however, the driving force for grain coarsening also increases with the high interfacial energy of NS architectures, and nanograins tend to have a low activation barrier for grain growth. This tendency for grain growth, which can occur even at room temperature for certain NS metals, limits their engineering application. Similarly, the low dislocation storage capacity of NS metals and alloys tends to limit their capacity to undergo plastic deformation, a topic that has been discussed at length by various authors [3–5].

Inspection of the scientific literature shows two basic approaches to hinder grain growth and improve the thermal stability of NS materials [6,7]. One is a thermodynamic approach in which the driving force for grain growth is lowered by reducing the grain boundary energy via solute segregation [7,8]. The other is a kinetic approach in which the grain boundaries are pinned in various ways to decrease grain boundary mobility, such as solute drag [9] and second phase Zener-pinning drag [10]. Using thermodynamic stabilization, Schuh et al. [8,11] fabricated highly thermally-stable W-20 at% Ti powders (~20 nm, stabilized up to 1100 °C) and Fe-10 at% Mg thin-film (grains coarsening from 18 to 26 nm after 30 min annealing at 763 °C). In related work, Rupert et al. [7,12] studied a series of binary model-alloys such as Cu-3 at% Zr powders and Ni-10 at% W coatings. The two common features of such binary materials are that they are single-phase and also the presence of solute with a high size mismatch and low solubility, and a prerequisite difference in crystal structure from the host metals. Using kinetic stabilization by introducing nano-precipitates, the thermal stability of NS alloys, such as Mg-alloys [13], Ti-alloys [14] and Al-alloys [15], has been reported to increase to 0.45–0.75 T_m which compares favorably to the stability of their precipitate-free counterparts below 0.4 T_m (T_m represents melting temperature in K) [16]. Koch et al. [6] compared the effectiveness of these approaches for various alloys, and concluded that Zener-pinning via nano-precipitates is perhaps the most effective strategy to retain nanograins at high homologous temperatures.

In precipitation-hardened Ni-based alloys, a higher threshold temperature for grain growth was reported in Inconel 718 (580 °C, 0.5 T_m) as compared to those corresponding to single-phase NS Ni-20%Cr (500 °C, 0.46 T_m), and this difference was attributed to Zener-pinning originating from nanoscale γ′-precipitates [17]. Similarly, Shankar et al. [10] reported a higher thermal stability for NS Inconel 718 at 600 °C. Zhao et al. [17,18] reported that the ductility of NS Al-alloys containing a high density of nano-precipitates was improved compared to their precipitate-free counterparts. These findings suggest that ductility of NS metals can be improved by introducing a dispersion of second-phase particles into the nanograins due to the fact that the dislocations are forced to accumulate while intersecting with second-phase particles, leading to enhanced strain-hardening [17–19].

* Correspondence to: A.D. Shan, School of Materials Science and Engineering, Shanghai Jiao Tong University, Shanghai 200240, People's Republic of China.
** Corresponding author.
E-mail addresses: adshan@sjtu.edu.cn (A. Shan), lavernia@uci.edu (E.J. Lavernia).

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Therefore, in the view of above discussion, this letter is motivated by the question whether it is possible to simultaneously enhance the thermal stability and ductility in bulk NS Ni-based alloys via introducing nano-precipitates. To accomplish this objective, a single-phase NS Ni-based alloy (~50 nm) was first produced by heavy cold-rolling at room temperature without any intermediate annealing [20]. Subsequently, NS and ultra-fine grained (UFG) Ni-based alloys with nanoscale γ'-precipitates (Ni_3(Al,Ti), $L1_2$ cubic) were obtained employing heat treatments on the cold-rolled (as-CRed) alloy. To provide insight into the underlying mechanisms, both microstructural evolution and mechanical behavior were systematically investigated.

A Ni-based alloy with a novel composition of $Al_{1.3-1.9}Ti_{1.2-1.6}Nb_{0.5}Mn_{0.6}W_{1.0}$ $Mo_{0.2}$ $Si_{0.1}Zr_{0.02}C_{0.03}B_{0.004}Fe_{20-30}Cr_{20-25}Ni_{bal}$ (wt%) was selected for study and was designed with higher amount of Al, Ti and a lower amount of Nb, relative to the standard Inconel 718 composition with the goal of precipitating more stable spherical γ'-precipitates instead of metastable γ″-precipitates [21]. Microstructural evolution and phase analysis of alloys were carried out on transmission electron microscope (TEM), X-ray energy-dispersive spectroscopy (EDS), X-ray diffraction (XRD) and scanning electron microscope (SEM). Mechanical properties were characterized by Vickers hardness and uniaxial-tensile test. Detailed experimental information can be found in our previous work [20].

As shown in Fig. 1, a high microhardness (HV480), almost three times of as-solution treated (as-STed) Ni-based alloy (HV169) before rolling, was obtained in the as-CRed alloy due to the presence of nanograins and a high density of defects [20]. The solid-line in Fig. 1 shows the microhardness variation as a function of annealing temperature for a 1 h exposure. The results show that a peak hardness of HV610 was achieved when annealed at 700 °C, and this can be rationalized on the basis of the precipitation-strengthening effect of γ'-precipitates. The evolution of microhardness of the as-CRed specimen during annealing at 700 °C is also shown in the figure via a dashed line. The microhardness decreased slightly with increased annealing temperature and a value of microhardness comparable to that of the as-CRed specimen was attained for 300 h, suggesting a high thermal stability at 700 °C.

Fig. 2 (a–c) show TEM images of different annealed alloys. As shown in Fig. 2(a), the average grain size (~90 nm) of the Ni-based alloy remains below 100 nm following annealing at 700 °C for 1 h. Compared to the severely distorted microstructure of as-CRed specimen [20], limited grain growth is observed and grain boundaries are still poorly defined due to the high level of elastic lattice distortions from the remaining high density of dislocations in boundary region. Note that [110], [211] and [221] superlattice reflections from Ni_3(Al,Ti) ordered phase are also observed in the diffraction patterns, suggesting the presence of γ'-precipitates. By contrast, a significant grain growth (~210 nm) is observed after annealing at 800 °C for 1 h (a_1-specimen) in Fig. 2(b). As the thermal stability of NS materials is defined as the threshold temperature during which grains remain nanosize after 1 h annealing [10], the as-CRed Ni-based alloy in this work is characterized by high thermal stability at temperatures close to 700 °C (0.62 T_m). Moreover, the average grain size remains ~180 nm after prolonged annealing at 700 °C for 300 h (a_0-specimen) as shown in Fig. 2(c) and (d), with the retention of high density of dislocations and sub-grain boundaries. This finding is consistent with published reports that thermally activated dislocation motion may be hindered by pinning from both solute atoms and precipitates [22]. Consequently, both the formation of γ'-precipitates via diffusion of atoms and the existence of γ'-precipitates will facilitate the retention of a high density of dislocations after annealing. These ultra-fine grains and the retained high density of sub-grain boundaries and dislocations, reveal the remarkly high thermal stability of the microstructure at 700 °C.

In related work, Chung et al. [23] reported an improved grain stability of crymilled Inconel 625 at 900 °C and attributed this finding to the grain-boundary pinning effect originating from the rapid formation of spherical NbC carbides and cylindrical-shaped Ni_3Nb intermetallic

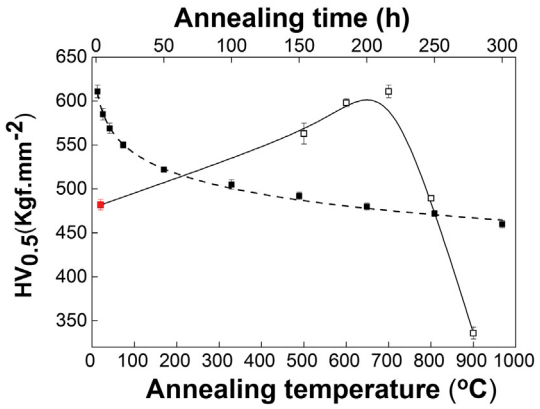

Fig. 1. Solid line: influence of annealing temperature (500 to 900 °C) on microhardness (annealed for 1 h). Dash line: microhardness evolution as a function of time at 700 °C. Red point represents microhardness of as-CRed alloy. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

precipitates at this temperature. Hence, one may conclude from these results that in order to attain a high thermal stability, rapid precipitation of γ′-precipitates should occur prior to grain growth at the onset of annealing. As shown in Fig. 3(a), rapid precipitation of γ′-precipitates is observed after annealing for only 2 min at 700 °C. This result is consistent with reports that the precipitation kinetics of NS alloys can be accelerated relative to those containing micrometer grains as a result of the shortened diffusion paths and high density of nucleation sites that characterize a nanocrystalline matrix [23]. Several mechanisms [6,7,11,23] have been formulated to rationalize the observed stability against grain growth. In the present case of NS Ni-based alloy, we need to first consider the pinning effect by second-phase particles, known as Zener-pinning, since the high volume fraction of nanoscale γ′-precipitates which formed during annealing represent the primary microstructural difference between NS Ni-based alloy and single-phase NS Ni-20%Cr (500 °C, 0.46 T_m) [24]. As shown in Fig. 2(d), spherical γ′-precipitates with an average size of ~50 nm have nucleated both along grain boundaries (pointed by yellow arrow) and in grain interiors (pointed by red arrow) in a_0-specimen. According to the STEM-EDS analysis, the main composition of matrix and precipitates of a_0-specimen were $Ni_{46.6}Fe_{26.02}Cr_{25.59}Ti_{0.3}Al_{1.5}$ (at%) and $Ni_{65.16}Fe_{4.20}Cr_{3.84}Ti_{9.18}Al_{16.54}Nb_{1.08}$ (at%), respectively. These high density of nanoscale γ′-precipitates can also be observed from the SEM image in Fig. 3(b).

The limited grain size D by immobile rigid particles evenly distributed can be estimated by the equation $D = \frac{4\alpha r}{3f}$, originally suggested by Smith [25]. In the case of a_0-specimen, the radius of precipitates r is ~50 nm, volume fraction of precipitates f is ~18 vol% calculated by JMatPro-v8 computer program [26]. α is geometrical constant related to grain boundary curvature [27] that ranging from 0.25 to 0.5 determined by experimental studies [23]. Furthermore, three-dimensional Monte Carlo simulations by Miodownik et al. [28] established an α value as approximately 0.5. Accordingly, we use a value of α that falls in the range of 0.25 to 0.5 and assume $f = 0.18$ pct and $r = 50$ nm in the a_0-specimen. The results show that the D stabilized by nanoscale γ′-precipitates is predicted to be in the range of 93 to 186 nm, which is consistent with the measured average grain size (~180 nm). Consequently, the grain-boundary pinning effect by γ′-precipitates is suggested to be principal mechanism suppressing the grain boundary movement and improving grain stability in NS Ni-based alloy. Thus, it is not surprising to observe the large grain size of a_1-specimen for its lower density of γ′-precipitates as shown in Fig. 3(c).

According to the above equation, fine precipitate size and large volume fraction of nano-precipitates are required to stabilize the nanograins. The coarsening behavior of γ′-precipitates at 700 °C,

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