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Optimization of B2/L2$_1$ hierarchical precipitate structure to improve creep resistance of a ferritic Fe-Ni-Al-Cr-Ti superalloy via thermal treatments

Gian Song [a,*], Soon Jik Hong [a], Jin Kyu Lee [a], Sung Ho Song [a], Sung Hwan Hong [b], Ki Buem Kim [b], Peter K. Liaw [c]

[a] Division of Advanced Materials Engineering, Kongju National University, Cheonan, Chungnam 330-717, Republic of Korea
[b] Faculty of Nanotechnology and Advanced Materials Engineering, Sejong University, Gwangjin-gu, Seoul 143-747, Republic of Korea
[c] Department of Materials Science and Engineering, The University of Tennessee, Knoxville, TN 37996-2200, USA

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ABSTRACT

The effect of precipitate structures of a B2-NiAl/L2$_1$-Ni$_2$TiAl hierarchical precipitate-strengthened ferritic alloy on the precipitate/matrix interface, elastic strain, and creep resistance has been investigated by systematic heat-treatments and creep experiments. The experimental results reveal that the highest creep strength was achieved in a heat-treated sample containing the largest coherent precipitate with fine internal networks, whereas the semi-coherent precipitate results in a sharp drop of the creep strength. It was found that the strong elastic strain field, affected by the internal structure within the precipitate, plays a crucial role in improving the creep resistance.

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For high-temperature applications, such as fossil-fuel-power plants, ferritic steels are more advantageous than austenitic steels and Ni-based superalloys, due to their low cost, high thermal conductivity, and low thermal expansion [1–3]. However, their industrial applications were limited to the relatively low-temperature environment (lower than 900 K), due to the microstructural instability, i.e., the coarsening of the incoherent strengthening carbides/nitrides during the long-term exposure to elevated temperatures, which degrades the creep resistance [4,5]. A new type of Fe-Cr-Ni-Al ferritic steels reinforced by a B2-NiAl precipitate has seen a recent increase of interest as a potential candidate for high-temperature applications because of their promising creep resistance [6,7]. These Fe-Cr-Ni-Al alloys consist of an ordered B2-NiAl precipitate (designated as β') distributed in a disordered body-centered-cubic (BCC) α-Fe matrix, and these two phases have a small difference in the lattice parameters, thus, coherent α/β' microstructure, which is analogous to that of γ/γ' Ni-based superalloys [8]. The coherent β' precipitate plays a crucial role in the creep properties of the α/β' Fe-Cr-Ni-Al alloys [9–11].

It has been reported that further improvement of the creep resistance of the α/β' Fe-Cr-Ni-Al alloys can be achieved by introducing a nano-scale L2$_1$-Ni$_2$TiAl phase into the B2-NiAl precipitate [12,13]. Such a two-phase precipitate consisting of a fine network of B2-NiAl and L2$_1$-Ni$_2$TiAl phases is referred to as a hierarchical structure [14]. The microstructure evolution of the hierarchically-structured B2-NiAl/L2$_1$-Ni$_2$TiAl precipitates-strengthened ferritic alloy has been investigated according to thermal treatment [15]. It was observed that the thermal treatment results in the morphological change from fine to coarse structures within the precipitate, which also affects the morphology and spatial distribution of the precipitates [16]. It was reported that the microstructural transition was associated with the elastic strain, which is influenced by the interface structure between the precipitate and matrix [16]. Thus, the morphological change is expected to affect the degree of the elastic strain, thus, creep behavior. However, limited studies have been conducted regarding the microstructure and creep resistance at temperatures above 973 K.

In the present research, we conducted the investigation on the microstructures and creep-deformation behavior of the two-phase NiAl/Ni$_2$TiAl precipitate-strengthened ferritic alloy, according to various thermal treatments. Systematic aging treatments at temperatures above 973 K were carried out to confirm the formation of the hierarchical structure and the morphological change of the precipitate. Moreover, creep tests were conducted at 1033 K in the stress range of 50–150 MPa, which allows us to understand the role of the precipitate structures (i.e., coherent/semi-coherent interface, elastic strain) on the creep resistance. Based on the modified version of the creep-power-law equation for precipitate-strengthened alloys, it was found that the threshold stress is dependent upon the precipitate interface structures.

The model alloy has a nominal composition of Fe-6.5Al-10Cr-10Ni-2Ti-3.4Mo-0.25Zr-0.005B in weight percent (wt%). An ingot of the

* Corresponding author.
E-mail address: gasong@kongju.ac.kr (G. Song).

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alloy with a dimension of 12.7 × 25.4 × 1.9 cm^3 was prepared by the Sophisticated Alloys, Inc., using the vacuum-induction-melting facility. Hot isostatic pressing (HIP) was applied to the ingot at 1473 K and 100 MPa for 4 h in order to reduce casting defects. The alloys were homogenized at 1473 K for 0.5 h. Four different aging treatments were carried out to introduce distinct precipitate sizes, specifically, (1) at 1053 K for 1 h, and at 1073 K for (2) 1 h, (3) 5 h, and (4) 50 h, respectively.

Scanning-electron microscopy (SEM) was conducted, using a Zeiss Auriga 40 equipped with an Everhart-Thornley secondary-electron detector. The SEM images were analyzed, using the ImageJ software to obtain the sizes of the precipitates [17], and the average values were estimated, employing more than 200 particles. Thin foils for conventional transmission-electron-microscopy (CTEM) observations were prepared by electropolishing, followed by ion milling at an ion energy of ~2.5 kV and an incident angle of ±~5°. The transmission-electron-microscopy (TEM) specimens were cooled by liquid N_2 during ion milling. The TEM observations were conducted with a Zeiss Libra 200 MC TEM/STEM. The TEM images were acquired at an acceleration voltage of 200 kV.

Tension-creep samples were machined with a gage diameter of 3.175 mm and a gage length of 28 mm. The samples were heated in a three-zone furnace, with the temperature controlled within 1 K by a thermocouple placed within 10 mm of the sample. Tension-creep tests were conducted at 1033 K under a constant load (at stresses ranging from 50 to 150 MPa). Extension in the specimens was measured, using a linear variable displacement transducer (LVDT) with a resolution of 10 μm. The applied load was increased when a well-defined minimum strain rate was observed. The cumulative creep strain per sample was kept well below the value where necking might occur. Strain rates at a given stress were estimated by measuring the slope of the strain vs. time in the steady-state creep regime.

Fig. 1 shows the micrographs of the Fe-Cr-Ni-Al-Ti alloy specimens aged at (a)–(b) 1053 K and (c)–(d) 1073 K for 1 h. The SEM image in Fig. 1(a) displays that the sample contains cuboidal precipitates with an average width of 81.8 nm and length of 108.6 nm. Note that the size of the precipitates was estimated, based on the rectangular or elliptical shape, and the averaged sizes are summarized in Table 1. Moreover, it can be seen that the precipitates are closely arranged to each other, and are preferentially aligned along certain orientations. The dark-field (DF) TEM image in Fig. 1(b) was obtained along the [100] zone axis, using (020) reflection, which shows the formation of narrow bright nano-zones within the precipitates, as denoted by white arrows. The microstructure of the current Fe-Cr-Ni-Al-Ti alloy upon aging at 973 K has been examined, using TEM and atom probe tomography (APT), which confirmed the formation of the nano-scale B2-type NiAl zones within the parent $L2_1$-type Ni_2TiAl precipitates [12,18]. Note that the super-lattice reflection employed to form the DF-TEM images in Fig. 1(b) is common to both the B2 and $L2_1$ structures [(001)-B2 and (002)-$L2_1$]. However, by considering the image intensity, and structure factor [15], the narrow bands inside the precipitates are the B2-NiAl phase, while the surrounding regions are the $L2_1$-Ni_2TiAl phase. It is worth noting that the matrix/precipitate interfaces are devoid of misfit dislocations, which implies the coherent interface between the precipitate and matrix.

It is well known that the morphological and spatial features of the precipitates are strongly affected by the interface and strain energies at the precipitate/matrix interface [19–21]. Specifically, when the interface energy dominates (equivalently, small sizes of precipitates), the stable shape of the precipitate becomes spherical, as observed in B2-NiAl strengthened ferritic alloys [22]. In contrast, when the strain energy becomes more influential than the interface energy (i.e., large

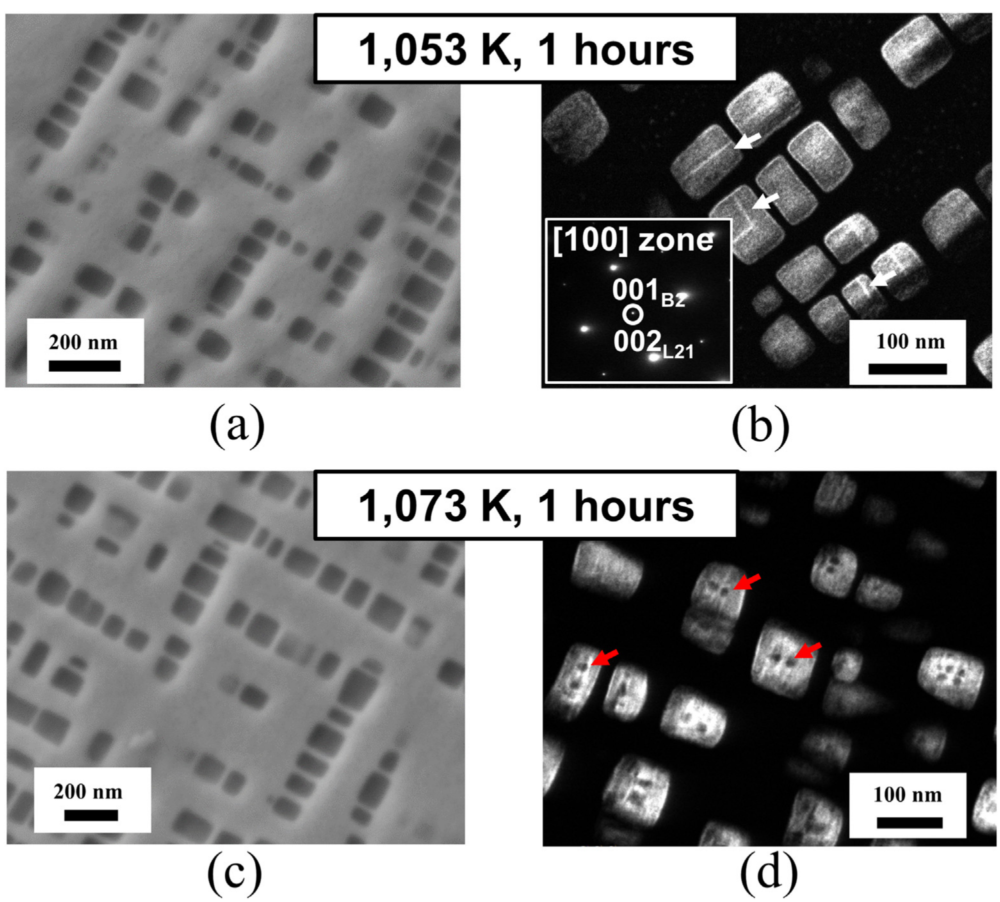

Fig. 1. Microstructures of the Fe-Cr-Ni-Al-Ti alloy specimens subjected to the homogenization treatment at 1473 K for 0.5 h, followed by the aging treatment [(a)–(b)] at 1053 K for 1 h, and [(c)–(d)] at 1073 K for 1 h, respectively. (a) and (c) SEM images, (b) and (d) dark-field (DF) TEM images. DF-TEM images are acquired along the [100] zone axis using the (020) reflection.

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