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Room temperature ductility of NiAl-strengthened ferritic steels: Effects of precipitate microstructure

Z.K. Teng[a,*], C.T. Liu[a,b,c], M.K. Miller[d], G. Ghosh[e], E.A. Kenik[d], S. Huang[a], P.K. Liaw[a,**]

[a] Department of Materials Science and Engineering, the University of Tennessee, Knoxville, TN 37996, USA
[b] Department of Mechanical Engineering City University of Hong Kong, Kowloon, Hong Kong
[c] Materials Engineering, Auburn University, Auburn, AL 36849-5341, USA
[d] Materials Science and Technology Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6136, USA
[e] Department of Materials Science and Engineering, Northwestern University, Evanston, IL 60208-3108, USA

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ABSTRACT

The effects of precipitate microstructure on the room temperature ductility of a series of carefully designed Fe–Al–Ni–Cr–Mo steels were investigated. Transmission electron microscopy (TEM), ultra small angle X-ray scattering (USAXS), and atom probe tomography (APT) were conducted to quantify the nano-scaled precipitates. The accuracy of the characterization results was verified by a numerical analysis. Three point bending tests results demonstrated that ductility was a function of the precipitate volume fraction and the Al and Ni concentrations in the Fe matrix, these relationships were discussed in terms of possible mechanisms. The ductility was also found to be independent of the precipitate size and inter-particle spacing in the studied range, which was validated by a theoretical model.

1. Introduction

NiAl-strengthened ferritic steels have been known as potential materials for ultra-supercritical steam turbine applications due to their low cost, promising creep and oxidation resistance [1–9]. However, recent development of these materials [5,9] revealed a critical issue restricting their application: poor ductility at room temperature. For example, Stallybrass and Sauthoff reported [5] that the Fe–Ni–Al–Cr alloy system with a high volume fraction (0.37) of β′ (NiAl type B2 precipitates) exhibited a bending ductility of near zero at room temperature. The steam turbine materials generally require a tensile ductility of ~10% at room temperature for sufficient workability in the as-cast condition and subsequent fabrication stages. The reasons for this kind of poor ductility have not been well investigated.

Since the ferritic alloys without β′ precipitates exhibit a tensile ductility of up to 25% at room temperature [10], the reduction of the ductility most likely comes from the formation of β′ precipitates and/or the compositional change associated with the partitioning of the alloying elements. The effects of precipitates microstructures (volume fraction, particle size, and inter-particle spacing) on the ductility of several precipitate-hardened alloys have been previously reported [11–19]. However, general agreement was not established among different materials. The results were strongly dependent on the properties of the matrix material [18], fracture mode after deformation [13], or some microstructural features of the precipitates [19]. How the β′ precipitate microstructure affects the ductility of NiAl-strengthened ferritic steels has not been investigated.

Another possible factor contributing to the poor ductility, the compositional change of both the Fe matrix (designated as α) and β′ precipitates by alloying, also needs to be carefully addressed. The influences of several alloying elements on the ductility of α-Fe have been investigated in previous studies [20–23], respectively. Among the alloying elements including in the alloys of current study, Ni was reported to have a softening effect on Fe and lower its cleavage transition temperature [22,23]. On the contrary, Al decreases the ductility of Fe with increasing its addition [21]. Alloying elements were also reported to affect the ductility of polycrystalline NiAl [24,25]. Small addition of Mo and Fe were found to increase the ductility of NiAl by promoting additional slip systems [26]. Although the effects of alloying on the ductility of α-Fe and NiAl have been investigated individually, the compositions of the β′ NiAl precipitates in the α-Fe matrix have not been determined due to its nano-scaled size. Therefore, the compositional effects on the ductility of the composite alloys have not been clearly identified.

The objective of this study is to investigate the effects of the β′ precipitate volume fraction, size and inter-particle spacing, along with the compositions of both α and β′, on the room temperature ductility. The base alloy composition in the current study is

* Corresponding author. Tel.: +1 865 455 0108; fax: +1 865 974 4115.
** Corresponding author. Tel.: +1 865 974 6356; fax: +1 865 974 4115.
E-mail addresses: zteng81@gmail.com (Z.K. Teng), pliaw@utk.edu (P.K. Liaw).

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Table 1
Nominal compositions of the model alloys (at.%).

Alloys #	Fe	Al	Cr	Ni	Mo
FBB-3	60.6	18.9	9.8	8.7	1.8
FBB-8 Nominal	66.1	12.7	10.2	9.0	1.9
FBB-8 Actual	66.3	12.6	10.1	9.0	1.8
FBB-12	70.2	8.0	10.4	9.2	1.9
FBB-13	71.9	6.1	10.5	9.3	1.9
FBB-18	56.1	18.9	9.8	13.0	1.8
FBB-19	51.6	19.0	9.9	17.4	1.8

one recently developed alloy FBB-8 [Fe-12.7, Al-9, Ni-10.2, Cr-1.9, Mo, Note that all the compositions are in atomic percent (at.%) throughout this paper, unless specified otherwise], which exhibited promising creep resistance but limited ductility [8,9]. This alloy was aged for different time periods to study the effects of the precipitate size and inter-particle spacing. Five other alloys with different β′ precipitate volume fractions (V_f) were carefully designed to study the effects of V_f and elemental partitioning. The results of these investigations are subsequently discussed to gain insights into the dominant microstructural features associated with the ductility.

2. Experimental

2.1. Fabrication

Alloy ingots with a weight of 120 g were prepared by arc-melting using commercially pure metals (99.97% in purity), and then drop cast into a Cu mold with the dimensions of 12.7 mm × 12.7 mm × 76.2 mm. The nominal compositions (at.%) are listed in Table 1. FBB-8 is the base alloy. In alloys FBB-3 through FBB-19, various amounts of Al and Ni were added to form different β′ volume fractions. The actual composition of the as-cast FBB-8 was determined by wet chemical analyses. The results summarized in Table 1 verified that the fabricated alloys exhibit the same composition as the nominal, indicating that the studied alloys can be fabricated accurately by the method described above.

2.2. Heat treatment

The ingots were subsequently cut into rods with a length of 20 mm by electron discharge machining (EDM). They were solution-treated at 1200 °C for 30 min in evacuated and sealed quartz tubes, followed by air cooling, and then aged at 700 °C for 100 h and air-cooled. In order to study the effects of different β′ precipitate sizes, samples of alloy FBB-8 were also aged at 700 °C for 500 and 1000 h, respectively.

2.3. Microstructure characterization

To characterize the microstructure of the precipitates, a combination of atom probe tomography (APT), transmission electron microscopy (TEM), and ultra small angle X-ray scattering (USAXS) techniques were utilized. The Imago Scientific Instruments Corporation local electrode atom probe (LEAP®) experiments were performed to quantify the compositions of both the α matrix and β′ precipitates. The phase volume fraction can be estimated by lever rule based on the obtained compositions. APT was conducted with a pulse repetition rate of 200 kHz, a voltage pulse fraction of 0.2, and a specimen temperature of 60 K. Due to the limitation of the analysis volume, it is not easy to determine the average size of β′ accurately by APT. TEM was conducted to quantify the β′ precipitate size in alloy FBB-8 aged for different time periods. A HITACHI-8100 TEM operating at 200 kV was used. Thin foils were prepared by a Fischione dual jet polisher. As a complementary technique for TEM, USAXS was utilized to measure the inter-particle spacing of selected bulk materials. USAXS was performed using the synchrotron source at the 32 ID beam line at the advanced photon source (APS), Argonne National Laboratory. X-rays of 16.85 keV energy and a beam size of 1.6 mm × 0.8 mm were used for these experiments.

Table 2
Elemental partitioning (at.%) and precipitate volume fractions (V_f) of the studied alloys. The standard error (σ) of the concentration estimated by APT is ~0.03%, given by $\sigma = \sqrt{C(1-C)N}$, where C is the concentration, N is the total number of atoms in the measured volume.

α Matrix	Fe	Al	Ni	Cr	Mo	100-V_f
FBB-3	69.0	14.5	2.2	11.7	2.5	80
FBB-8	75.1	7.2	3.0	11.9	3.1	82
FBB-12	79.3	3.0	3.1	12.1	2.4	86.4
FBB-13	80.6	2.6	3.0	12.0	1.8	92.7
FBB-18	73.5	9.3	1.7	12.4	3.0	72.5
FBB-19	77.0	4.9	1.5	14	2.5	62.5

β′ Precipitate	Fe	Al	Ni	Cr	Mo	V_f
FBB-3	12.2	45.2	40.3	1.5	0.8	20
FBB-8	12.7	43.6	41.2	0.8	1.4	18
FBB-12	12.0	38.5	48.2	0.6	0.2	13.6
FBB-13	18.1	36.5	43.1	1.7	0.3	7.3
FBB-18	10.6	43.6	43.6	1.2	0.6	27.5
FBB-19	10.4	43.2	44.1	0.8	1.1	37.5

2.4. Ductility behaviors

Three-point-bending tests were performed to assess the ductility of the model alloys. From the heat-treated rods, strips with a size of 19.05 mm × 3.17 mm × 0.76 mm for three-point-bending tests were cut by EDM, and then tested at a strain rate of $2 \times 10^{-4}\ s^{-1}$ at room temperature. The bending strain of a strip is calculated as [27]

$$\varepsilon_f = \frac{6Dt}{L^2} \quad (1)$$

where ε_f is the strain on the outer surface, D is the maximum deflection at the center of the strip (mm), t is the thickness (mm), and L is the support span (mm), which is equal to 10 mm in this study.

3. Results

3.1. Microstructure and ductility of the based alloy

Representative microstructures of the based material (FBB-8) in our study are shown in Fig. 1. The average grain size is ~100 μm (Fig. 1a). β′ Precipitates have an average diameter of ~130 nm (Fig. 1b) and a spherical morphology. Fig. 1c presents the USAXS intensity (I) versus scattering vector (Q) curves of the samples aged for different lengths of time. Each curve has a peak at $Q = 1–6 \times 10^{-3}\ A^{-1}$. Details regarding the quantitative analyses of the USAXS spectrum can be found in the literature [28]. Briefly, the establishment of this peak is due to the interaction of the precipitates developed during the heat treatment. The inter-particle spacing (λ) can be calculated by $\lambda = 2\pi/\text{peak } Q$ [28]. The value of λ is determined to be ~217 nm for the alloy aged for 100 h, which is consistent with the projected average distance between the precipitates in the TEM micrograph, as shown in Fig. 1b (~210 nm). The compositions of both phases can be determined by counting the ions obtained by APT (Fig. 1d). The results are listed in Table 2. Although the compositions of each phase in different alloys vary, the distributions of the elements exhibit similar characteristics, which have been presented in detail elsewhere [8]. Applying the lever rule to all the elements, the β′ precipitate volume fraction is estimated to be ~18%.