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Effects of Al on the microstructure and ductility of NiAl-strengthened ferritic steels at room temperature

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ABSTRACT

One of the major problems for the development of ferritic steels strengthened by NiAl-type (*B*2) precipitates is their poor ductility at room temperature. In conjunction with the computational alloy design, selected experiments are performed to investigate the effect of Al content on the ductility of prototype Fe–Ni–Cr–Al alloys. The microstructure and composition of the matrix (α-Fe type) and precipitate phases are characterized by transmission electron microscopy (TEM) and analytical electron microscopy (AEM). Three-point-bending experiments show that alloys containing more than 5 mass% Al exhibit poor ductility (<2%) at room temperature, and their fracture mode is predominantly cleavage type. Two major factors governing the poor ductility are (i) the volume fraction of NiAl-type precipitates, and (ii) the Al content in the α-Fe matrix. A bend ductility of more than 5% can be achieved by lowering the Al concentration to 3 mass% in the alloy. The alloy containing about 6.5 mass% Al is found to have an optimal combination of hardness and ductility.

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1. Introduction

NiAl-type phase with the *B*2 structure (designated as β') is known to form coherent–coplanar precipitates in a body-centered-cubic (BCC) α-Fe (designated as β) matrix. Thus, Fe-based β/β' alloys are microstructurally analogous to classical Ni-based γ/γ' superalloys. Furthermore, ferritic steels have higher thermal conductivity and lower thermal expansion, and are less expensive than Ni-based superalloys. These factors give them the advantages as potential candidates for steam turbine applications.

Creep resistance is one of the most important properties of materials for high-temperature structural applications. Consequently, several studies on β'-strengthened ferritic steels have focused on the creep behavior and related microstructural characterizations [1–7]. However, there is no systematic study of the ductility of these materials. Only recently, Stallybrass and Sauthoff [5] reported that the Fe–Ni–Al–Cr alloys with a high volume fraction (0.37) of β' exhibited a bending ductility less than 1.5% even at temperatures higher than 1000 °C. The reason for this low ductility of these materials has not been fully understood. Nevertheless, the steam turbine materials generally require a tensile ductility of about 10% at room temperature for sufficient workability in as-cast and subsequent fabrication stages.

In two-phase materials, such as Fe-based β/β' alloys, it is expected that many properties will be closely related to the intrinsic properties of these two phases. Therefore, we present a brief summary of the room temperature ductility of β and β', as reported in literature, particularly the influence of chemistry.

The effects of various alloying elements on the ductility and toughness of the β phase have been reported in literature [8–10]. Leslie ([8]; also references therein) has summarized the effect of many alloying elements on the strength, ductility, and toughness of the β solid solution. Specifically, it has been established that Co, Ni, Rh, Ir, and Pt increase toughness (decrease notch–impact transition temperature), while Si and P decrease toughness (increase notch–impact transition temperature) and also to a lesser extent by Cr, Mn, and Re. In this regard, Al behaves analogous to Si in the β solid solution. Recently, Herrmann et al. [10] reported the tensile strength and ductility of Fe-(4 to 18) Al (atomic percent) solid solution at room temperature. With increasing Al content, an increase in strength and a concomitant decrease in ductility were observed. Furthermore, α-Fe is known to exhibit cleavage fracture, particularly at low temperatures, due to the limited number of active slip systems.

Pugh [11] proposed that the ductility of pure metals is related to the ratio of bulk modulus to shear modulus (K/G). The rationale is that with decreasing K/G, more energy is stored as dilation rather

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than shear deformation. In fact, the measurement of polycrystalline elastic constants of β solid solutions show a marked increase in K/G when Ni and Pt are added to Fe, leading to an increase in fracture toughness. On the other hand, K/G slightly decreases when Cr and Mn are added, leading to decrease in fracture toughness. However, such a simplistic correlation fail in many cases [8]. A more fundamental hypothesis is that the elements which increase ductility, also change bonding so as to increase the cross-slip of screw dislocations in α-Fe [8].

Polycrystalline NiAl with the $B2$ structure exhibits no tensile ductility at room temperature due to the lack of five independent slip systems to satisfy the von Mises' criterion for polycrystalline plasticity [12]. Like many BCC metals and $B2$ aluminides, NiAl exhibits cleavage fracture. However, the cleavage plane is not well defined [13,14]. It has been observed that in $B2$-NiAl, fracture of {511} transient planes may proceed before cleavage on {110} planes [13,14]. Darolia et al. [15] reported that small additions of Fe, Ga, and Mo significantly improve the tensile ductility, but Fe is the most effective. For example, NiAl along <110> has a tensile ductility of only 0.8%, but with the addition of 0.25 at.% Fe (substituting for Al in NiAl), the tensile ductility increases to 5.9% [15]. In another study, Field et al. [16] reported that the addition of Cr promotes the activation of the <111> slip system. Law and Rachinger et al. [17,18] suggested that Cr, Mn, and Fe lower the ordering energy of NiAl and favor <100> slip, but this argument was disputed by Cotton [19]. Small additions of Mo were also reported to increase the ductility of polycrystalline NiAl at room temperature [20], while Hahn and Vedula [12] demonstrated that small deviations from the stoichiometric composition make NiAl even more brittle at low temperatures.

Based on the available literature, it is implicit that the dissolved Al in the β solid solution, and the volume fraction and stoichiometry of the NiAl precipitates are expected to be the major factors controlling the ductility of β/β' alloys. To investigate these factors, we have carried out a systematic study. Specifically, six β/β' (FBB) alloys with the nominal compositions of Fe–xAl–10Ni–10Cr–3.4Mo–0.25Zr–0.005B (x = 3–10 mass%) were fabricated to investigate the effects of Al on the phase transformation, microstructure, partitioning behavior of alloying elements, precipitate volume fraction, and bending ductility at room temperature [Note that all the compositions are in weight percent throughout this paper, unless specified otherwise]. An attempt was made to improve the ductility of the NiAl-strengthened ferritic steels by optimizing the Al concentration.

2. Experimental

Alloy ingots with a weight of 120 g were prepared by vacuum arc-remelting (VAR) using commercially-pure metals (99.97% in purity) and Fe–B master alloy (95.4%Fe + 4.6%B), then drop cast into a Cu mold with the dimensions of 12.7 × 12.7 × 76.2 mm. The nominal compositions are listed in Table 1. The FBB-3 is the base alloy composition. The additions of 10% Ni and 10% Al are used to form NiAl-type precipitates. On the other hand, Cr and Mo were added to impart oxidation resistance, solid solution strengthening, and control of lattice misfit by dilating the β matrix. Zr at a level of 0.25% is added to promote the slow diffusion in the matrix. 0.005% B is added to enhance the grain-boundary cohesion. From FBB-7 to FBB-13, various amounts of Al are added to study their effects on the microstructure and ductility at room temperature.

Table 1
Nominal compositions (in mass%) of prototype (FBB series) alloys.

Alloy#	Fe	Al	Ni	Cr	Mo	Zr	B
FBB-3	Bal.	10	10	10	3.4	0.25	0.005
FBB-7	Bal.	8	10	10	3.4	0.25	0.005
FBB-8	Bal.	6.5	10	10	3.4	0.25	0.005
FBB-9	Bal.	5	10	10	3.4	0.25	0.005
FBB-12	Bal.	4	10	10	3.4	0.25	0.005
FBB-13	Bal.	3	10	10	3.4	0.25	0.005

The ingots were subsequently cut into rods with a length of 20 mm. 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. From the heat-treated rods, strips with a size of 19.05 × 3.17 × 0.76 mm for three-point-bending tests were cut by electron discharge machining (EDM), and then tested at a strain rate of 2×10^{-4} s^{-1} at room temperature. The fracture surfaces were investigated by scanning electron microscopy (SEM) to identify the fracture mechanisms. A differential scanning calorimeter (DSC), NETZSCH 404C, was used to study the phase transformation in model alloys. A small piece of sample with a mass of about 50 mg was measured in Al_2O_3 crucible under an argon atmosphere at a constant heating rate of 40 K/min from 20 to 1500 °C.

To characterize the microstructure of prototype alloys, both transmission electron microscopy (TEM) and analytical electron microscopy (AEM) techniques were employed. A HITACHI-8100 TEM and HITACHI HF-2000 AEM, both operating at 200 kV, were used. Thin foils were prepared by a Fischione dual jet polisher. The AEM is equipped with an ultrathin window (ATW) Link energy dispersive X-ray (EDX) detector. The take-off angle for the X-ray detector was 68°. The X-ray collection time was between 300 and 500 s, and the electron probe size was about 10 nm. Care was taken to avoid a two-beam condition in order to minimize electron-channeling effects [21]. The background-subtracted integrated intensities of EDX spectra were converted to compositions. The details may be found elsewhere [22,23]. The elemental partitioning data of FBB-13 was determined by atom probe tomography (APT). The local electrode atom probe (LEAP®) was performed with a pulse repetition rate of 200 kHz and a voltage pulse fraction of 0.2, and a specimen temperature of 60 K.

3. Results

3.1. Phase transformation

Preliminary DSC studies on the as-cast materials revealed that there was no strong peak observed around the solvus temperature of NiAl due to the formation of limited amount of NiAl phase. Therefore, it was not able to determine the phase transition temperatures, especially the solvus temperature of NiAl precipitates. Consequently, aged materials were used for DSC studies to form sufficient amount of NiAl precipitates. The DSC heating curves of the alloys are compared in Fig. 1(a). All curves show that the transformation from β' to β starts at a temperature [$T_{\beta'(NiAl)}$] of about 750 °C and completes at a temperature (T_β) around 1050 °C. Therefore, the solution-treatment temperature (1200 °C) used in this study was adequate. There is another exothermic transition peak at a temperature higher than $T_{\beta'(NiAl)}$ for the alloys containing less Al (FBB-8 and FBB-12). This feature is most likely to be the FeAl phase separation (also a β′ phase) inside the NiAl particles, which was suggested by the investigation of an Al-rich Fe–23.2Al–4.1Ni alloy system by Liu et al. [24]. They revealed that two kinds of $B2$ phases (NiAl and FeAl) coexist in a Fe–Ni–Al system when the as-quenched alloy was aged at a temperature around 725 °C. In other words, this separation depends on the alloy composition, as this transition peak was not observed in the alloys with higher Al contents (FBB-3 and FBB-7). To determine whether the formation of possible FeAl phase occurred during the aging or the solution-treatment process, the DSC studies