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Microstructure evolution and mechanical behavior of as-cast, heat treated and directionally solidified Fe–15Al–10Nb alloys

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ABSTRACT

Fe–15Al–10Nb (at.%) alloys containing Laves phase fibers embedded in a disordered α-(Fe,Al) matrix were investigated in as-cast, heat treated and directionally solidified condition. Microstructure consisted of either duplex structure of primary dendrites and eutectic (as-cast and heat treated) or fully eutectic structure (directionally solidified). Nanoindentation on the Laves phase fibers revealed their anisotropic features as well as the onset of dislocation plasticity. Compression testing showed the yield strength anomaly, which occurred in the 500–650 °C range. Directional solidified alloy exhibited the lowest strength fracture toughness whereas the as-cast alloy had the highest strength and fracture toughness. The value of stress exponent obtained from the strain rate dependence of the flow stress indicated that the dislocation climb mechanism dominated the creep process. Deformation mechanisms were also discussed and related to the microstructure evolution.

1. Introduction

The eutectic architecture, a naturally occurring in-situ composite, can exhibit superior mechanical properties to those of either monolithic phase, owing to the strong constraining effects provided by the interlocking microstructure [1]. Adding refractory metals such as Cr, Mo, Nb, Ta, V, W and Zr to intermetallics is one approach to improve their brittleness and toughness at the room temperature [2,3]. Among all refractory metal elements, Nb is along with Ta assumed to be an ideal solute addition to Fe–Al intermetallics for the following reasons: (i) Nb with a limited but sufficient solid solubility in the Fe–Al alloys can form the stable fine Fe_2Nb Laves phase precipitates, able to increase the high temperature strength more pronouncedly in comparison with the phases containing V, Cr, and Mo [4–6]; (ii) due to the metallic radii, the efficiency in increasing the B2–A2 and $D0_3$–B2 transition temperatures, and the effect of decreasing T_{sol} and T_{liq} follows the sequence Nb > Mo > V > Cr [7]; (iii) the dissolution of Nb does not have impact on the vacancy concentration in some Fe–Al alloys, but it is inclined to trap carbon atoms from the solid solution, stabilizing the mechanical properties at high temperatures [8,9] and (iv) the relatively low diffusion rate of Nb leads to a slow structural coarsening at the temperature below 1000 °C [4].

Fe–Al–Nb alloys with a disordered structure (A2) have attracted attention in the recent years. For example, lamellae to fibrous structure transition was discussed in the Fe–23Al–10Nb eutectic alloys prepared by directionally solidification with different growth rates [2]; The cast-in continuous network of brittle Laves phase in the Fe–15Al–5Nb was converted into a relatively uniform dispersion of coarse particles by a high-strain thermo-mechanical forming process to improve its strength and to maintain the ductility [10]. Different from $D0_3$ and B2 Fe–Al alloys, the Fe–15Al–xNb series has special merits. On one hand, the low aluminum content could decelerate diffusivity at a fixed temperature, suggesting a considerable creep resistance [11]. On the other hand, the disordered structure implies a great potential as an engineering material with lowered susceptibility to environmental cracking [2,10–12].

Prymak and Stein [13] have investigated the exact position of eutectic valley on the ternary liquidus surface of the Fe–Al–Nb system. The established isothermal sections of Fe–Al–Nb system suggest that a eutectic trough runs nearly parallel to the Fe–Al side starting form 10 at.% Nb [14]. Fe–15Al–10Nb contains the disordered ferrite matrix and large amount of Nb, suggesting that it should exhibit high ductility and large amount of Laves phase. Therefore, this eutectic composition is chosen to further study the

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performance of alloys comprising α-(Fe,Al) (disordered A2) matrix and the hexagonal Laves phase (C14 type) fibers. Interfaces (grain boundaries and phase boundaries) play a fundamental role in mechanical properties of metals during plastic deformation, acting as sources of, storage sites for, barriers to, and sinks of defects via absorption and annihilation [15]. In this work, different micro and interface structures of Fe–15Al–Nb alloys were prepared by casting, heat treatment as well as directional solidification. A systematic study of mechanical properties of this material has been carried out and related to the microstructure evolution.

2. Materials and experiments

The as-cast alloys (AC) with a nominal composition of Fe–15Al–10Nb (all composition in at.%) were prepared by induction melting and gravity casting into copper moulds in the shape of cylindrical bar with a diameter of 30 mm and length of 200 mm. Heat treatment of some AC ingots was performed in a furnace at 1100 °C for 120 h. Then, the heat-treated ingots (HT) were furnace cooled to room temperature. Directionally solidified ingots (DS) with the same composition were grown in a Bridgman type crystal growing facility at 1400 °C at a growth rate of 10 mm/h.

DS samples parallel and perpendicular to the growth direction, AC and HT samples were cut and then embedded in cold cast resins, respectively. After grinding with SiC paper to 1200 grit finish and polishing with diamond pastes up to 1 μm, AC, HT and DS specimens were observed in back scattered electron contrast mode of a scanning electron microscope (SEM-EVO MA15, Zeiss) equipped with chemical microanalysis energy dispersive spectrometry (EDS-Oxford INCA 350). As for the metallographic observations by SEM in secondary electron mode and by optical microscope (Olympus BX-51), specimens were etched by reagent consisting of 5 ml HF, 15 ml of HNO_3 and 30 ml of lactic acid for 7 s. The corresponding image analysis software ANALYSIS in optical microscope was used for quantitative analysis of eutectic structures in DS specimens. For transmission electron microscopy (TEM) investigation, thin disks were sliced and ground to a thickness of approximate 200 μm. Twin-jet electropolishing at −45 °C and 7 V in a solution of 25% nitric acid in methanol mixture was employed to prepare the thin foil samples. TEM examinations were performed with a Philips-TECNAC F20 microscope operating at 200 kV.

Samples for the fracture toughness measurement were all cut in a radial–circumferential direction (R–C) from ingots according to the ASTM E399-90 standard. Each specimen with a precut notch (5.75×1.00 mm^2), whose radius tip was as cut without a pre-fatigue crack, was ground and polished to the final dimensions of $28 \times 6 \times 12$ mm^3. Three-point bending tests were performed with a loading span of 24 mm and a cross-speed of 10^{-4} mm/s. The fracture toughness K_Q in MPa m$^{1/2}$ was calculated according to:

$$K_Q = \left(P_Q S \Big/ BW^{3/2}\right) \cdot f(a/W) \tag{1}$$

$$f(a/W) = \frac{3(a/W)^{1/2}\left[1.99 - (a/W)(1 - a/W) \times \left(2.15 - 3.93a/W + 2.7a^2W^2\right)\right]}{2(1 + 2a/W)(1 - a/W)^{3/2}} \tag{2}$$

where P_Q is the load; B is the specimen thickness; S is the span; W is the specimen depth (width); a corresponds to the crack length.

For the compression and creep tests, specimens of $6 \times 6 \times 9$ mm^3 in dimensions were cut from each ingot. As for the DS material, specimens with solidification direction parallel to the loading axis were denoted DSL, and those with perpendicular, DST. The temperature-dependent stress-strain behavior was studied by compression testing in vacuum at a deformation rate of 10^{-4}/s in a Gleeble thermo-physical simulator (GLEEBLE 3800, DSI). Three specimens of each kind were tested in the 25–800 °C range at each testing temperature. Graphite foils and Ni lubricant were applied between the specimen and tungsten carbide compression heads to minimize friction. Tests in the air atmosphere were performed in a universal electromechanical testing machine (Instron 3384) until fractured with a load cell of 150 kN. Fracture mechanisms were investigated through the analysis of fractured and lateral surfaces of deformed samples by SEM. Since the Gleeble machine can speed up deformation during thermal cycling [16,17], it was employed for the accelerated creep tests at constant strain rates of 5×10^{-5} s^{-1}, 5×10^{-6} s^{-1} and 5×10^{-7} s^{-1} at both 700 °C and 800 °C, respectively. Flow stress at 0.2% plastic strain and creep stress at 1% strain were determined from true stress– true strain curves.

The Vickers hardness was measured by the HMV-2 microhardness tester (SHIMADZU) with a load of 100 gf. Nanoindentation testing at room temperature was conducted with a Nanoindenter™ II instrument (TI950, Hysitron) equipped with atomic force microscopy (AFM) to analyze the indented surfaces. A series of indents was made by a Berkovich indenter (tip radius ~280 nm) with loads ranging from 2 to 10 mN and a distance of 3 μm between each two indents.

3. Results and discussion

3.1. Microstructure evolution

Fig. 1(a) present the microstructures of the AC alloy, where the light phase corresponds to the Laves phase and the dark one to the α-(Fe,Al) phase. The flower-like morphology of primary Fe_2Nb dendrites indicated isotropic growth, characteristic for the non-faceted growth. The dendrites were embedded in the eutectic structure. Inside the eutectic colonies, eutectic grew as fine fibers with an average spacing of 760 nm. At the colony boundaries, the fibers curved toward the boundaries and looked like the lamellae. The composition of Laves phase presented in Table 1, shows that the Nb content of 28% is in good agreement with the [14], which suggests that the Fe-rich phase boundary of the Laves phase does not shift markedly with temperature, and the Nb content is about 27 at.%.

After heat treatment at 1100 °C for 120 h, the amount of dendrites decreased, whereas the dense lamellar-like structures coarsened and transformed to numerous rods and particles as shown in Fig. 1(b). Regarding the eutectic growth, it can be observed that around primary Laves phase dendrites, there is a thin film of the α-(Fe,Al) phase, from which eutectic is formed. When primary dendrites nucleate and start to grow at some undercooling below the equilibrium liquidus temperature, the primary phase can either promote or hinder the nucleation of the eutectic phases depending on its capacity to serve as a nucleating agent. Several