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Microstructure and room temperature fracture toughness of directionally solidified NiAl–Mo eutectic in situ composites

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ABSTRACT

The microstructure and room temperature fracture toughness of pseudo-binary NiAl–xMo (x = 7.8, 9, 13 and 16 at. %) in situ composites were investigated. For all four alloys examined, uniform and well-aligned Mo fibrous structures were directionally solidified by the liquid metal cooling process at the growth rate of V = 6 μm/s, respectively. With the increase of the Mo content, the spacing of the Mo fibers decreased and the volume fraction of Mo fibers increased. Significant influence of Mo addition on the toughness was observed for the directional solidification alloys, and the fracture toughness was found to increase with volume fraction of Mo rods. Scanning electron microscopy was used to characterize the fracture behavior. Based on the results of theoretical analysis, the composite structures of these alloys provided improvement in fracture toughness over binary NiAl primarily by crack trapping and crack bridging. Interface debonding, crack deflection and microcrack linkage provided further resistance to crack growth.

1. Introduction

The intermetallic compound NiAl has long been identified as a promising candidate for high-temperature structural applications in aggressive environments, because it possesses attractive combination of properties including high melting point (1638 °C), high thermal conductivity (~80 W/mK), lower density (~5.86 g/cm^3) and superior oxidation resistance up to 1300 °C [1–3]. However, like many other intermetallics, its intrinsic brittleness and insufficient room temperature fracture toughness restrict its utilization. There has been a systematic effort to overcome its shortcomings through grain refinement, single crystals, micro- and macro-alloying as well as incorporating second phase reinforcements, combustion synthesis and hot isostatic pressing [4–8]. An effective approach to improving room temperature fracture toughness of intrinsically brittle NiAl intermetallics is by in situ development of a composite structure through directional solidification (DS) of eutectic alloy, which would generate a composite structure formed by NiAl and another phase. The composite structure can effectively improve the mechanical properties. Typical examples were reported by Bei et al and Ferrandini et al. [9,10]. In their studies, the refractory metal Mo was incorporated into the NiAl, which greatly increased the mechanical properties compared to binary NiAl as the result of two phase microstructures of NiAl and Mo solid solutions. However, previous researches were all carried out at the eutectic composition (9Mo, all compositions in this paper are given in terms of at. % unless otherwise stated). The disadvantages of working with eutectic alloys are the limited composition ranges where couple growth may take place under a lower temperature gradient. The volume fraction of toughening phase (Mo) is within a narrow range. As is known that the mechanical properties of the eutectic in-situ composite largely depend upon the eutectic spacing and the volume fraction of the constituent phases. Whereas the volume fraction of the constituent phases is controlled to some extent by the composition of the alloy [11]. Therefore, efforts to promote the volume fraction of toughening phase (Mo) may further enhance the toughness of NiAl–Mo eutectics.

In the present investigation, a series of pseudo-binary NiAl–xMo (x = 7.8, 9, 13 and 16) alloys were directionally solidified (DS'ed) by liquid metal cooling (LMC) technique. Alloy compositions were varied to increase the volume fraction of toughening phase, which attempted to enhance the fracture toughness of NiAl–Mo alloys. The microstructure and the room temperature (RT) fracture toughness of DS NiAl–Mo eutectic alloys were evaluated.

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2. Experimental procedures

2.1. Alloy preparation and directional solidification

Four different stoichiometric NiAl alloys containing different Mo contents (7.8Mo, 9Mo, 13Mo and 16Mo) were prepared by vacuum induction melting furnace equipped with a pure argon injection system, and drop casting into cylindrical ingots measuring 80 mm in diameter and 100 mm in length. The compositions of all as-cast ingots were analyzed by using the inductively coupled plasma mass spectrometer (ICP-MS). It was observed that the compositions of all the alloys were close to the nominal values for the three elements with the deviations less than 0.48 at% for Ni, 0.75 at% for Al, and 0.1 at% for Mo, indicating negligible losses during processing. The purity of raw materials used in this study is Ni 99.99 (wt. %), Al 99.99% (wt. %) and Mo 99.95% (wt. %), respectively. In a previous study, it was learned that the addition of Mo to NiAl led to the occurrence of a eutectic transformation L→β NiAl+α Mo, which occurs in the composition of Ni–45.5Al–9Mo [12]. From this we can infer that the NiAl-7.8Mo is a hypoeutectic alloy, and NiAl–13Mo and NiAl–16Mo are hypereutectic alloys.

The as-cast rods 9.0 mm in diameter and 100 mm in length were cut from the master ingots by electro-discharge machining (EDM). The rod was put in a high purity alumna crucible (> 99.99% purity) which was 115 mm long and 9.1 mm in inner diameter with the thickness of 1 mm. DS experiments were carried out under an argon atmosphere in a Bridgman type crystal growing facility with LMC technique. In the directional solidification process, the sample was heated by a graphite heater at 1700 °C ± 10 °C and then kept isothermal for 20 min. After thermal equilibrium was reached, the sample was moved downwards into liquid Ga–In–Sn alloy at a fixed pulling velocity of 6 μm/s. The temperature gradient close to the solid/liquid (S/L) interface was measured to be approximately 334 °C/cm. When steady-state solidification was reached, the S/L interface was kept by quenching the solidifying specimen in liquid Ga–In–Sn pool. In this paper, the growth rate is substituted for the pulling velocity, because they are the same when the solidification process achieves the steady state after a short initial transient zone during DS process.

2.2. Fracture toughness tests

Three-point bending (3PB) specimens were machined by EDM from the DS'ed samples. Prior to toughness testing, all the flat faces of bend specimens were ground on emery paper to a finish of 1000 grit (nominal dimensions, 3 mm × 6 mm × 30 mm). A schematic illustration of the fracture toughness composite specimen is shown in Fig. 1. The length of each bend specimen was oriented parallel to the growth direction of the solidified sample. A narrow straight 3 mm-deep notch was machined, also by EDM. But a fatigue pre-crack was not initiated at the notch tip prior to 3PB testing.

Fracture toughness tests were performed in air at room temperature in an Instron 3382 universal test machine and at a cross-head speed of 0.05 mm/min. A peak load in a load-displacement curve was used to determine the F_Q (maximum load). In our study, four to five specimens were tested and the average value was employed for the RT fracture toughness of each specimen condition. We used the standard equation (1)–(3) to calculate fracture toughness value (K_Q) of samples.

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

$$S = 4W = 24\text{mm} \tag{2}$$

$$f(a/W) = 3(a/w)^{1/2} \times \frac{1.99 - (a/W)(1 - a/W)\left[2.15 - 3.93(a/W) + 2.70(a/W)^2\right]}{2(1 + 2a/W)(1 - a/W)^{3/2}} \tag{3}$$

Here, W is the width of specimen; B is the thickness of specimen; a is the length of the slit notch; S is the span of loading; F_Q is the maximum (fracture) load and f(a/w) is geometrical factor. In our study, some of the NiAl–Mo composite specimens were not broken into two separate halves after 3PB tests in order to observe the crack propagation path from the side surfaces.

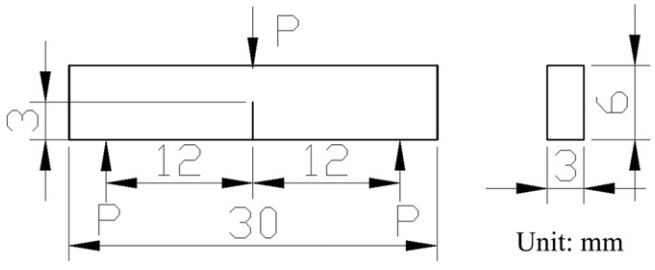

Fig. 1. Scheme of 3PB specimens.

2.3. Metallography and fractography

The DS'ed samples were sectioned both transversely and longitudinally by EDM. The metallographic process involved grinding, mechanical polishing and chemical etching with a solution 80%HCl–20%HNO_3 by volume. The quantitative image analysis was conducted by means of SISCIAS V8.0 metallographic image analysis software. In order to characterize the microstructure and fracture surface morphology, back-scattered (BSE) electron imaging was used to identify the phases in the DS'ed samples, fracture surfaces and the crack propagation path on the side surfaces of the bend samples.

3. Results

3.1. Solid/liquid interface morphologies and steady microstructures

The typical quenched S/L interfaces and microstructures of the DS alloys at V = 6 μm/s through longitudinal and transverse section are shown in Fig. 2. The eutectic microstructures formed by the NiAl (black matrix) and Mo phases (white fiber) present a fibrous morphology. It can be seen that all quenched S/L interfaces were maintained planar ones. As expected, approximately hexagonal arrangement of the Mo rods embedded within NiAl matrix in the transverse section. In the longitudinal section, the fibers were uniformly aligned along the growth direction, but the fibers appear as short rods, this is may be due to the various angles of cutting, i.e., the plane of cutting is not parallel to the growth direction. For the purpose of accurately determining whether any pro-eutectic