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Fracture toughness of a directionally solidified Al–Nb–Ni ternary eutectic

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ABSTRACT

Fracture toughness (K_{IC}), an important property of brittle materials, can be determined by indentation cracking tests. This paper reports on an investigation of the fracture toughness of a directionally solidified Al_3Nb–Nb_2Al–AlNbNi ternary eutectic, based on the Vickers indentation test applied to longitudinal and transverse sections of its microstructure. The measurements were taken using indentation loads varying from 2.45 to 24.5 N. Correlations between the resulting crack parameters and indentation load were evaluated using Palmqvist and half-penny cracks models. In the range of indentation loads studied, the results suggested that the Palmqvist model provided a better fit to the experimental data. Fracture toughness was calculated using equations developed for Palmqvist crack mode. The indentation fracture toughness values for longitudinal and transverse sections are in the range of 2.82–3.05 MPa $m^{1/2}$ and 2.98–3.59 MPa $m^{1/2}$, respectively. It was found that the addition of Ni and incorporation of a third phase to the Al_3Nb–Nb_2Al eutectic improved fracture toughness of this *in situ* composite material.

1. Introduction

In situ composite materials produced by directional solidification of high temperature eutectic alloys have been the subject of a number of investigations, usually aiming to replace Ni-based alloys in high temperatures applications [1]. This type of composite is composed of two or more phases, usually intermetallic phases, with the reinforcing phases produced during the manufacturing process [2]. Research has recently focused on the Al_3Nb–Nb_2Al eutectic alloy for the manufacture of eutectic-based structural composites. This eutectic material shows high strength at elevated temperatures, but poor fracture toughness at low temperatures [3,4]. Fracture toughness or the critical stress intensity factor, K_{IC}, is essential for predicting the mechanical behavior of brittle materials such as intermetallic compounds [5].

An interesting way to determine K_{IC} is by the Vickers hardness indentation technique [6–8]. This method was first examined as a tool to describe fracture toughness in the 1950s [7]. The application of certain indentation loads will nucleate cracks in brittle materials, which may be correlated to fracture toughness. In such cases, two types of indentation cracks occur, depending on their geometrical features: Palmqvist cracks and radial-median cracks [9,10].

Palmqvist cracks consist of four half-penny-shaped cracks that initiate only at the corners of the indentation, their extensions are restricted to small distances beneath the surface and they show a propensity to be just as deep as the indentation. On the other hand, radial-median cracks, also called half-penny cracks, consist of two fully developed semi-elliptical cracks that commence at the corners of the indentation [11–13]. They develop under and around the indentation, penetrating far below the surface and forming cracks normal to the indentation plane. Details of both these crack modes, including their dimensions, are illustrated in Fig. 1 [6–10].

It is well known that the phase arrangement in a eutectic microstructure controls its mechanical behavior. An earlier study found that adding Ni to the binary Al_3Nb–Nb_2Al eutectic led to a ternary eutectic transformation ($L \leftrightarrows Al_3Nb + Nb_2Al + AlNbNi$) at Al–40.4Nb–2.42Ni (at.%) [14] and application of directional solidification to this ternary eutectic resulted in a very regular microstructure composed of fiber-like phases [15]. It is possible that the incorporation of a third phase to the Al_3Nb–Nb_2Al eutectic may positively alter fracture toughness of this *in situ* composite material.

To address questions pertaining to the effect of an additional phase on mechanical behavior of the Al_3Nb–Nb_2Al eutectic, this paper presents the results of the Vickers indentation of directionally solidified $Al_3Nb + Nb_2Al + AlNbNi$ ternary eutectic and discusses correlations between the microstructure and fracture toughness.

2. Experimental procedure

The ternary eutectic alloy in the Al–Nb–Ni system was prepared with appropriate amounts of pure elements melted in an arc furnace equipped with a vacuum system combined with injection of

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high purity argon. Ternary eutectic samples were solidified directionally using the Bridgman technique under an argon atmosphere. The 30-mm-long, 6.0-mm-diameter ingots were placed in high purity Al_2O_3 crucibles and solidified by slowly moving the crucible from the upper hot region to the lower cold region, at a thermal gradient ranging from 80 to 100 °C/cm, with a growth rate of 1.0 cm/h.

Normal metallographic preparation procedures were utilized to investigate the microstructure. Chemical etching consisted of 10 vol.% HF, 30 vol.% HNO_3 and 60 vol.% lactic acid. The material was microanalyzed by optical microscopy (OM) (Olympus – BX60M) and scanning electron microscopy (SEM) (Jeol JXA 840A). Indentation tests were carried out on longitudinal and transverse sections of a directionally solidified sample, using a W-Testor hardness indenter under 2.45 N (250 gf), 4.9 N (500 gf), 7.35 N (750 gf), 9.8 N (1000 gf), 14.7 N (1500 gf) and 24.5 (2500 gf) applied for 15 s. A sequence of eight measurements was taken of each load.

Elastic behavior of directionally solidified ternary eutectic was evaluated by using pulse-echo ultrasonic echography technique. The diagram in Fig. 2 shows the experimental apparatus used for measuring the elastic constants. Piezoelectric transducers (5 MHz) in contact with the specimen were used to determine longitudinal V_L and transversal V_T velocities of sound waves in the material. The density (ρ) of the samples was determined by the Archimedes method. These velocities, density and geometrical features of the samples were used to determine Young's modulus (E), shear modulus (G) and Poisson's ratio (υ). They were determined according to the following equations [16,17]:

$$E = \frac{\rho V_T^2\left(3V_L^2 - 4V_T^2\right)}{V_L^2 - V_T^2} \quad (1)$$

$$G = \rho V_T^2 \quad (2)$$

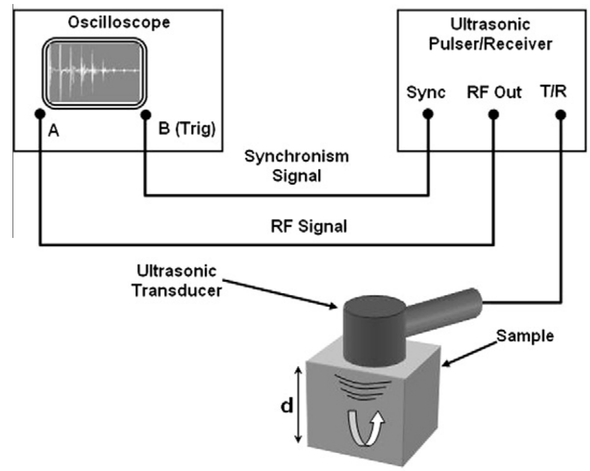

Fig. 2. Diagram of the experimental apparatus for measuring elastic constants.

$$\upsilon = \frac{E}{2G} - 1 \quad (3)$$

Crack mode evaluation and a 3D reconstruction of the eutectic microstructure were carried out by obtaining metallographic images at several planes in the 3D domain. This procedure is the so-called serial sectioning technique and involves removing a certain amount of materials by polishing samples containing Vickers hardness indentations, followed by recording of the metallographic plane. Repeating this procedure a number of times generates a significant amount of 3D information of the eutectic microstructure. The Vickers indentations were also utilized to align images of each plane with the images of the preceding metallographic plane. In this study, a sectioning depth of 4.0 μm was chosen [18].

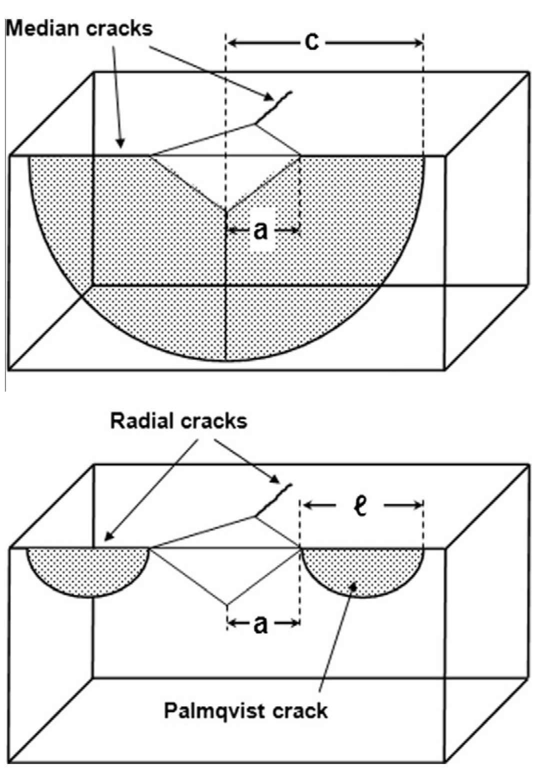

Fig. 1. Top and transverse views of the Palmqvist and Radial-median crack modes induced by Vickers indentation.

3. Results and discussion

Fig. 3a depicts a three-dimensional reconstruction of the phase arrangements of the Al_3Nb–Nb_2Al–AlNbNi ternary eutectic obtained by directional solidification using the serial sectioning technique. The reconstructed microstructure suggests that the phases are continuous in the growth direction and present some sinuosity. It is a complex task to make a complete identification of the constituent phases of the Al_3Nb–Nb_2Al–AlNbNi ternary eutectic microstructure under an optical microscope, since optical microscopy does not suffice to identify the AlNbNi phase [14]. On the other hand, the use of SEM (backscattered electron images)

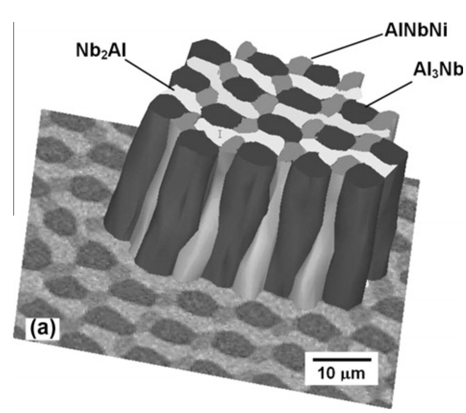

Fig. 3a. Three-dimensional reconstruction of the Al_3Nb–Nb_2Al–AlNbNi ternary eutectic structure.