



ISE and fracture toughness evaluation by Vickers hardness testing of an Al_3Nb – Nb_2Al – AlNbNi *in situ* composite

C.T. Rios, A.A. Coelho, W.W. Batista, M.C. Gonçalves, R. Caram*

State University of Campinas, C.P. 6122, Campinas, SP 13083-970, Brazil

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ABSTRACT

The aim of this work is to present correlations between Vickers indentation cracks and fracture toughness of an Al_3Nb + Nb_2Al + AlNbNi *in situ* composite. Correlations between the resulting crack parameters and indentation load suggested that the radial-median model resulted in a better fit to the experimental data. The hardness value was found to change according to the indentation load applied. Low indentation loads resulted in high hardness values, while the application of higher loads decreased the hardness. Experimental results indicate that Vickers hardness varied from 8.6 to 9.5 GPa, which is due to the indentation size effect (ISE). Fracture toughness was calculated based on several models and the results were found to vary in a broad range of values. The fracture toughness obtained from Vickers indentation was in the order of 1.65 – $2.26 \text{ MPa m}^{1/2}$.

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1. Introduction

Equipment and components designed to work at high temperatures are generally made of metallic materials that are able to preserve their mechanical and chemical properties quite well, particularly at temperatures exceeding 1000°C . Difficulties in preserving these properties can be overcome by employing eutectic alloys formed by intermetallic phases. *In situ* composites produced by means of solidification of eutectic alloys may have attractive properties that differ from those of their constituent phases [1,2]. A promising alloy for the production of high-temperature structural materials is the *in situ* composite consisting of the Al_3Nb , AlNb_2 and AlNbNi eutectic [3,4]. Aluminum-based intermetallic compounds usually present a high melting point, low density, and high strength at high temperatures, but also poor fracture toughness at low temperatures.

For brittle materials such as ceramic and intermetallic compounds, fracture toughness is defined as K_{IC} . In the event of fracture, if unstable crack propagation takes place, K_{IC} corresponds to the critical stress intensity factor [5,6]. This parameter is of paramount importance when an evaluation of mechanical behavior is required. A versatile and relatively simple method for establishing fracture toughness is obtained from Vickers hardness indentation technique [7,8]. As a result of indentation loads on a brittle material, cracks

may be nucleated at the indentation corners, which may be related to fracture toughness.

In this work, Vickers indentation technique was applied to evaluate correlations between the indentation cracks and fracture toughness of an Al_3Nb + Nb_2Al + AlNbNi ternary eutectic in the as-cast (A.C.) condition. In addition, hardness was also measured as a function of the indentation load.

2. Experimental procedure

Samples of the eutectic alloy of composition Al-40.4Nb-2.42Ni (at.%) were melted in an arc furnace equipped with a vacuum system combined with injection of high purity argon. The microstructural evaluation was carried out by means of regular metallographic procedures. The microstructure was revealed by applying a chemical solution of 10 vol.% HF, 30 vol.% HNO_3 and 60 vol.% lactic acid. The microstructure features were inspected by using optical microscopy (OM, Olympus BX60M) and field emission scanning electron microscopy (FESEM, JEOL JSM 6340F).

The Vickers indentation test was conducted on carefully finished and etched surfaces with a W-Testor Hardness, using indentation loads, P , of 250, 1000, 2000, 3000, 4000 and 5000 g applied for 15 s. Eight indentations were made for each load. Young's modulus (E) and Poisson's ratio were determined by an ultrasonic technique with 5 MHz piezoelectric transducers, which allowed for measurements of longitudinal and transverse sound velocities in the samples [9]. Density was determined by the Archimedes method.

3. Results and discussion

3.1. Indentation parameters and Vickers hardness

Fig. 1a and b presents identical-area FESEM images of the three-phase eutectic microstructure. Fig. 1a is a secondary electron image

* Corresponding author. Tel.: +55 19 35213314; fax: +55 19 35213314.
E-mail address: rcaram@fem.unicamp.br (R. Caram).

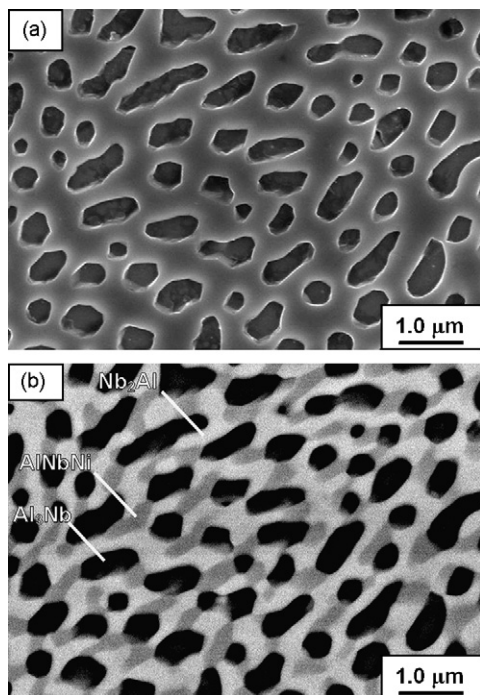


Fig. 1. FESEM images of the $\text{Al}_3\text{Nb} + \text{Nb}_2\text{Al} + \text{AlNbNi}$ ternary eutectic microstructure in the as-cast condition: (a) secondary electron image and (b) backscattered electron image.

and Fig. 1b is the corresponding backscattered electron image. Secondary electron image does not allow one to distinguish between AlNbNi phase and other phases. The same does not occur when this microstructure was analyzed by backscattered electron.

Fig. 2 depicts an optical micrograph of indentation and cracks on an Al-Nb-Ni ternary eutectic sample in the as-cast condition, produced by a 1000 g (9.8 N) load. The Vickers indentations were sampled always the same eutectic microstructure. Cracks due to indentation are well-formed and almost symmetrical. It is worth noting that only perfect indentation cracks were selected for the measurements and the dispersion of the values obtained is very limited. Table 1 shows indentation parameters as a function of the indentation load on the as-cast Al-Nb-Ni ternary eutectic sample. A description of these parameters is given in Fig. 3.

The accuracy of fracture toughness evaluations of brittle materials obtained by indentation methods may be affected by the way hardness is determined. The apparent hardness, H , of a given material is defined as the ratio of the indentation load to the projected

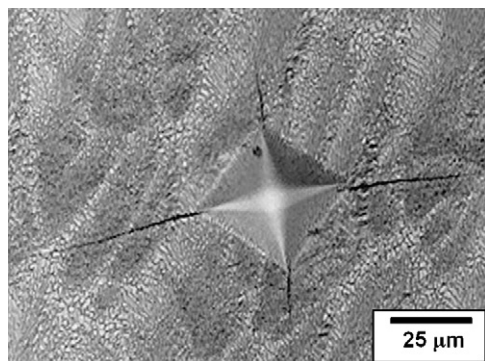


Fig. 2. Optical micrograph of Vickers indentation cracks on the surface of an $\text{Al}_3\text{Nb} + \text{Nb}_2\text{Al} + \text{AlNbNi}$ ternary eutectic sample in the as-cast condition (indentation load of 9.8 N).

Table 1

Indentation parameters as a function of indentation load, P , on the as-cast Al-Nb-Ni ternary eutectic sample.

Load, N (g)	Indentation parameters			
	$a = d/2$ (μm)	l (μm)	c (μm)	c/a
2.45 (250)	10.9 ± 0.2	10.8 ± 1.7	21.7 ± 1.7	2.0 ± 0.1
9.80 (1000)	22.2 ± 0.3	34.9 ± 4.9	57.3 ± 5.0	2.6 ± 0.2
19.6 (2000)	31.5 ± 0.2	59.8 ± 2.8	91.3 ± 2.9	2.9 ± 0.1
29.4 (3000)	39.0 ± 0.3	79.3 ± 2.6	118.2 ± 2.4	3.0 ± 0.1
39.2 (4000)	45.1 ± 0.5	86.9 ± 3.2	132.0 ± 3.2	2.9 ± 0.1
49.0 (5000)	50.7 ± 0.5	98.5 ± 4.7	149.2 ± 4.7	3.0 ± 0.1

area of the indentation, or $H = P/2a^2$. According to a number of studies [10,11], hardness is found to change according to the indentation load applied. Hardness values found under low indentation loads are generally high, while the application of higher loads results in a decrease in hardness. For Vickers hardness, $H_V = 0.9272H$. Fig. 4 depicts Vickers hardness as a function of the indentation load; these experimental results indicate that hardness varied from 8.6 to 9.5 GPa. The phenomenon associated with variations in hardness caused by the indentation load is known as the indentation size effect (ISE) [12–15]. If an indentation technique is employed to identify the characteristics of materials, such as fracture toughness behavior, and the ISE is found to be significant, the results obtained may lead to inconsistent conclusions. To date, no reasonable explanation has been put forward for the dependence of hardness on the applied load. This dependence may have to do with the presence of oxides on the surface layer, whose deformation behavior differs from that of the inner zones, or with work hardening due to indentation. Another explanation is based on the friction between indenter and sample surfaces [16,17]. At low indentation loads, the indentation size is reduced and the load fraction due to friction is relatively higher, so the measured hardness is also higher. The relationship between P and is given by [16,17]:

$$P = a_1 d + a_2 d^2 \quad (1)$$

where a_1 is associated with the surface between the indenter and the indentation, and a_2 is connected with the indentation volume due to deformation; hence, it is directly connected to the yield strength [16]. According to Gong [18] and Gong et al. [19], if the ISE should be applied over a wide range of indentation loads, Eq. (1) must be modified as follows:

$$P = a_0 + a_1 d + a_2 d^2 \quad (2)$$

where a_0 is associated with the residual surface stress due to machining of the sample [10], and a_1 and a_2 are the same constants of Eq. (1).

Fig. 5 presents the data on indentation load, P , and indentation size (indentation diagonal), d , obtained experimentally. A third-order polynomial fit of the data presented in Fig. 5 allows one to determine a_1 and a_2 as being $2.1 \times 10^{-2} \text{ N}/\mu\text{m}$ and $4.6 \times 10^{-3} \text{ N}/\mu\text{m}^2$ (4.6 GPa), respectively. The value of a_0 was found to be negligible, which could not be considered unexpected, since the eutectic samples of this study were subjected to careful surface finishing. Consequently, it was concluded that Eq. (1) provides the best fit for the experimental data on hardness variations due to indentation loads. While coefficient a_1 corresponds to the indentation size proportional resistance of the test sample, coefficient a_2 corresponds to the load-independent hardness [16].

3.2. Crack pattern

Depending on geometric features, cracks due to indentation may be classified as either Palmqvist or radial-median cracks;

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