

# Directional solidification, microstructure and properties of the $\text{Al}_3\text{Nb}$ – $\text{Nb}_2\text{Al}$ eutectic

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Available online 9 December 2004

## Abstract

The Al–Nb system exhibits a eutectic transformation at 1595.2 °C, which results in  $\text{Al}_3\text{Nb}$  (D0<sub>22</sub>) and  $\text{Nb}_2\text{Al}$  (D8<sub>b</sub>) phases. This paper is concerned with the processing of this eutectic by directional solidification. Alloys were prepared by arc melting and directionally solidified in Bridgman-type equipment. The resulting samples were utilized to evaluate the solidification microstructure and morphology regarding the growth conditions. Eutectic microstructures obtained were regular with lamellar morphology. Variations of the growth rate showed that an increase in this parameter causes a decrease in the lamellar spacing. With further increase in the growth rate, eutectic cells were observed as a result of constitutional undercooling. Oxidation tests of eutectic microstructures showed that this alloy suffers severe microstructure instability, with growth kinetics of the oxide scale of linear type. This indicates that the Al in the Nb–Al eutectic alloy is insufficient to form protective oxide films, such as,  $\alpha\text{-Al}_2\text{O}_3$ . Finally, the heat treatment in argon atmosphere showed that the eutectic alloy presents a high degree of microstructure stability at 1200 °C.

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PACS: 81.30.Bx; 81.30.Fb

Keywords: A1. Directional solidification; A1. Eutectics; A1. Phase diagrams; A2. Bridgman technique

## 1. Introduction

In the past few decades, considerable effort has been focused on developing advanced materials for high-temperature structural applications. Most monolithic materials that have acceptable high-

temperature properties are very brittle at room temperature. Therefore, the aim of many investigations has been to design polyphase materials, including in situ or natural composites [1], that exhibit a balance of properties and avoid many of the complex processing operations associated with the fabrication of synthetic composites. The in situ processing of many intermetallic alloys eliminates interface incompatibility between the reinforcing

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phase and the matrix, as well as allows growth near the thermodynamic equilibrium. Composite materials consisting of intermetallic compounds have properties such as high melting point, low density, high strength at high temperature and, in some cases, inadequate oxidation resistance, but also poor fracture toughness at low temperature. However, ductility can be improved by alloying or by adding reinforcements, resulting in substantial improvement in mechanical properties [2].

Solidification behaviour and structural characteristics of eutectic alloys in many systems continue to attract interest because of their influence on the properties and performance of materials containing eutectic constituents. The eutectic alloys processed by directional solidification allow one to obtain highly anisotropic structures of two or more phases, with regularly arranged microstructures. In addition, it is a promising technique for production of the so-called in situ composites. The directional solidification technique allows components to be produced from the melt in a single-stage process, and leads to a composite of phases that are thermodynamically stable, chemically compatible, well aligned and finely dispersed.

In the case of the eutectic alloy combining  $\text{Al}_3\text{Nb}$  and  $\text{Nb}_2\text{Al}$  intermetallic compounds, research efforts were directed to improvement of its mechanical behaviour and making it an attractive material for high-temperature structural applications [3]. Such studies have demonstrated that both phases exhibit high strength at elevated temperatures, but also poor fracture toughness at ambient temperature. Near  $900^\circ\text{C}$ , the  $\text{Al}_3\text{Nb}$  phase [4] and near  $1200^\circ\text{C}$  the  $\text{Nb}_2\text{Al}$  phase [5] undergo a brittle-to-ductile transition that allows material to relieve stress by slip mechanisms. Other studies on the  $\text{Al}_3\text{Nb}$  intermetallic (with a melting point of  $1680^\circ\text{C}$ , relative low density ( $4.54\text{ g/cm}^3$ ), with  $\text{D0}_{22}$  crystal structure, which might be suitable for use at temperatures up to  $1200^\circ\text{C}$ ) and on the  $\text{Nb}_2\text{Al}$  compound (with a melting point of  $1940^\circ\text{C}$  and  $\text{D8}_b$  crystal structure, attractive for applications requiring maximum operating temperatures of  $1400^\circ\text{C}$  [6]) revealed that the lack of adequate room temperature ductility and fracture toughness limit their applicability. The intrinsic

brittleness of the intermetallic compounds is due to the lack of active slip systems. In the case of  $\text{Al}_3\text{Nb}$ , only three slip systems were found and the density of dislocations in the  $\text{Nb}_2\text{Al}$  was very small [7,8]. In this paper, the microstructure evolution of directionally solidified  $\text{Al}_3\text{Nb}$ – $\text{Nb}_2\text{Al}$  eutectic alloy is characterized. In addition, the oxidation resistance and the microstructure stability at high temperatures of the eutectic composite were also studied.

## 2. Experimental procedure

The eutectic transformation of interest of this work occurs at a temperature of  $1595.2^\circ\text{C}$  and compositions close to  $\text{Al-42.2 at\% Nb}$ . These values were experimentally found by using differential thermal analysis and microstructure examination. Commercially pure niobium and aluminium were weighed to meet the eutectic composition, and then melted in an arc furnace equipped with a vacuum system combined with an injection system of high-purity argon.

After preparing the alloys, they were processed in a Bridgman-type solidification facility. Samples  $50.0\text{ mm}$  long and approximately  $5.0\text{ mm}$  in diameter were inserted in high-purity  $\text{Al}_2\text{O}_3$  crucibles and further within a tantalum tube used as susceptor. Next, the sample together with the alumina crucible and tantalum susceptor were coaxially positioned inside a  $50.0\text{ mm}$ -diameter quartz tube and heated by an induction coil properly connected to a radio-frequency generator. All the experiments were conducted at a temperature of  $1610 \pm 10^\circ\text{C}$ , a thermal gradient of approximately  $100^\circ\text{C/cm}$  and growth rates of 2.9, 2.0, 1.6 and  $1.0\text{ cm/h}$ .

The study of the microstructure stability was carried out on directionally solidified samples at a growth rate of  $2.0\text{ cm/h}$ . Thermo-gravimetric measurements of oxidation were conducted in oxygen at  $1200^\circ\text{C}$ , for 24 h, and the samples of  $3.0 \times 4.0\text{ mm}$  were mechanically polished. The weight change of samples was recorded continuously during exposure using a microbalance with an accuracy of  $1.0\text{ }\mu\text{g}$ . The thermal stability was studied in isothermal conditions, at temperatures

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