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Reinforcement of Al–Fe–Ni alloys with the in situ formation of composite materials

G. Vourlias*, N. Pistofidis, E. Pavlidou, G. Stergioudis

Department of Physics, Aristotle University of Thessaloniki, 54124 Thessaloniki, Greece

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ABSTRACT

One of the most effective methods for the improvement of the mechanical properties of metals is their reinforcement with non-metallic materials. In the present work powder of K_2TiF_6 and KBF_4 was added in an Al–Fe–Ni alloy while the alloy was in liquid form at 1060 °C with a 5 wt.% mixture of powders and with simultaneous stirring for 30 min. The liquid was squeeze-casted at 150 bar. The as-cast specimens were examined with electron microscopy and X-ray diffraction. SEM analysis revealed that the as-formed material is composed by needle-like crystallites along with a dentritic form and an interdendritic phase. The composition of the needle-like crystallites may presumably be expressed by the formula (Fe-Ni)Al_3. The rest of the matrix consists of almost pure Al grown dentritically, while the interdendritic phase contains Fe and Ni dissolved in Al. EDS analysis also proved the existence of spots with high Ti concentration, which probably refer to the Ti–B compounds. Finally TEM verified the presence of nanocrystals in the matrix.

1. Introduction

Metal matrix composite materials are ideal for structural applications where high strength-to-weight and stiffness-to-weight ratios are required [1,2]. Aluminum alloys are one of the preferable metallic matrices for this kind of materials. A usual method for their production is based on the dissolution of solid iron or ferrous alloys in liquid aluminum, which leads to the enrichment of aluminum in iron and/or other alloying elements. This process results in a subsequent growth of intermetallic and intermediate layers [3,4]. The simultaneous addition of inorganic salts (such as K_2TiF_6 and KBF_4), which react with each other in the liquid phase, results in the formation of ceramic particles in situ that are dispersed in the matrix and behave as reinforcement as they impede the dislocation movement [5–7].

Dissolution of metals in liquid aluminum mainly depends on thermodynamic conditions and experimental parameters such as temperature, stirring time, the degree of aluminum saturation and the chemical composition of the ferrous alloys in the reaction zone. The above-mentioned factors play also an important role to the formation of the different phases during dissolution and to the reaction of the salts that form the reinforcement material. Furthermore, it was established that the growth of the intermetallic phases is controlled by physicochemical reactions at the interfaces of the liquid and the solid phase and by interdiffusion through the different phases [3,4]. In any case, all these factors (matrix composition, dispersion and size of the reinforcement particles) affect the mechanical behavior of the as-formed material and the above phenomena are enhanced when the matrix is nanostructured (e.g. part of its crystals is nanosized), since in this case the amount of intergranular boundaries is increased. As a result increased mechanical resistance is achieved [8].

In any case the main objective of this study is the investigation of the structure of in situ composite materials in a Al–Fe–Ni matrix. After a brief review of the development of the diffusion structures of a Fe–Ni alloy dissolved in pure aluminum, the material was examined with electron microscopy in order to track nanocrystals in its mass and the results of the above research were connected with the improvement of the mechanical properties of the aluminum matrix.

2. Experimental

In the present work a Fe–Ni rod containing 97 wt.% Fe and 3 wt.% Ni was dissolved in liquid Al. The as-formed matrix was heated up to 1060 °C and a 5 wt.% mixture of K_2TiF_6 and KBF_4 was added with simultaneous stirring for 30 min. The liquid was squeeze-cast at 150 bar.

The initial observation of a polished surface of the as-cast specimens took place with a Karl Zeiss M8 low magnification binocular light microscope equipped with a CCD camera for image capture. Afterwards cross-sections have been cut from each sample, mounted in bakelite, polished down to 5 μm alumina emulsion and etched with Keller solution (1% HF, 1.5% HCl, 2.5% HNO_3, 95% H_2O). The as-prepared coupons were examined with scanning electron microscopy with a 20kVolt JEOL 840A SEM

* Corresponding author. Tel.: +30 2310 998066.
E-mail address: gvourlia@auth.gr (G. Vourlias).

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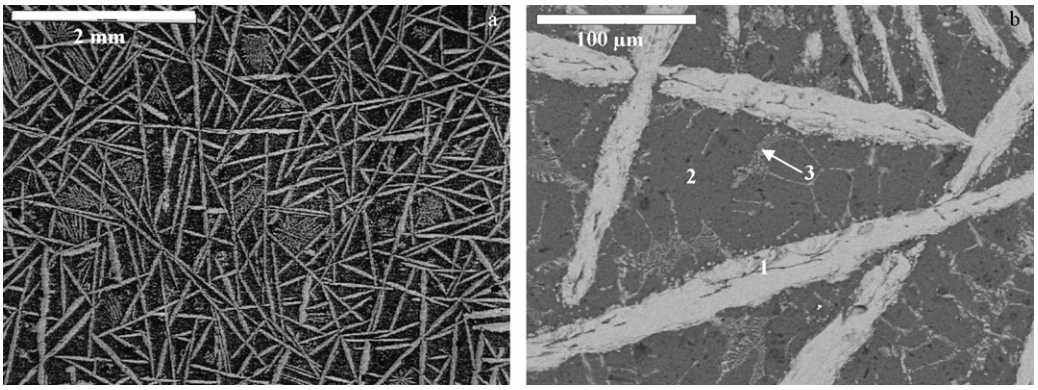

Fig. 1. SE micrographs of the composite (a) at low magnification, (b) at higher magnification). Point #1 refers to the needle-like crystallites, point #2 to the base phase and point #3 to the gray interdendritic phase.

equipped with an OXFORD ISIS 300 EDS analyzer and the necessary software to perform line scan and chemical mapping of the samples. X-ray diffraction was also used for their structural characterization. For this purpose a Bragg-Brentano Philips D5000 diffractometer was used, where CuKα radiation (λ = 1.54406 Å) was chosen. Selected specimens were also observed with conventional transmission electron microscopy (CTEM) and high resolution electron microscopy (HREM) with a 100 kV JEOL 100CX TEM and a 200 kV JEOL 2011 HREM respectively, after the suitable sample pretreatment [9].

TEM was used because it is characterized by higher mass resolution with regard to X-rays. Thus, one phase can be identified by electron diffraction even if it exists only in a small portion in the material. For this reason, in the present work TEM analysis was mainly used for the Al–Fe phase examination.

3. Results and discussion

Fig. 1a shows a SEM micrograph of a coupon of the material formed at low magnification. What is rather characteristic is the presence of needle-like formations that overrun its surface. The examination of the same area at higher magnification reveals more details (Fig. 1b). Three distinct areas are observed based on their relief, which consist of white needle-like crystallites randomly dispersed in the whole matrix, a gray area developed in an interdentritic form and a dark base phase.

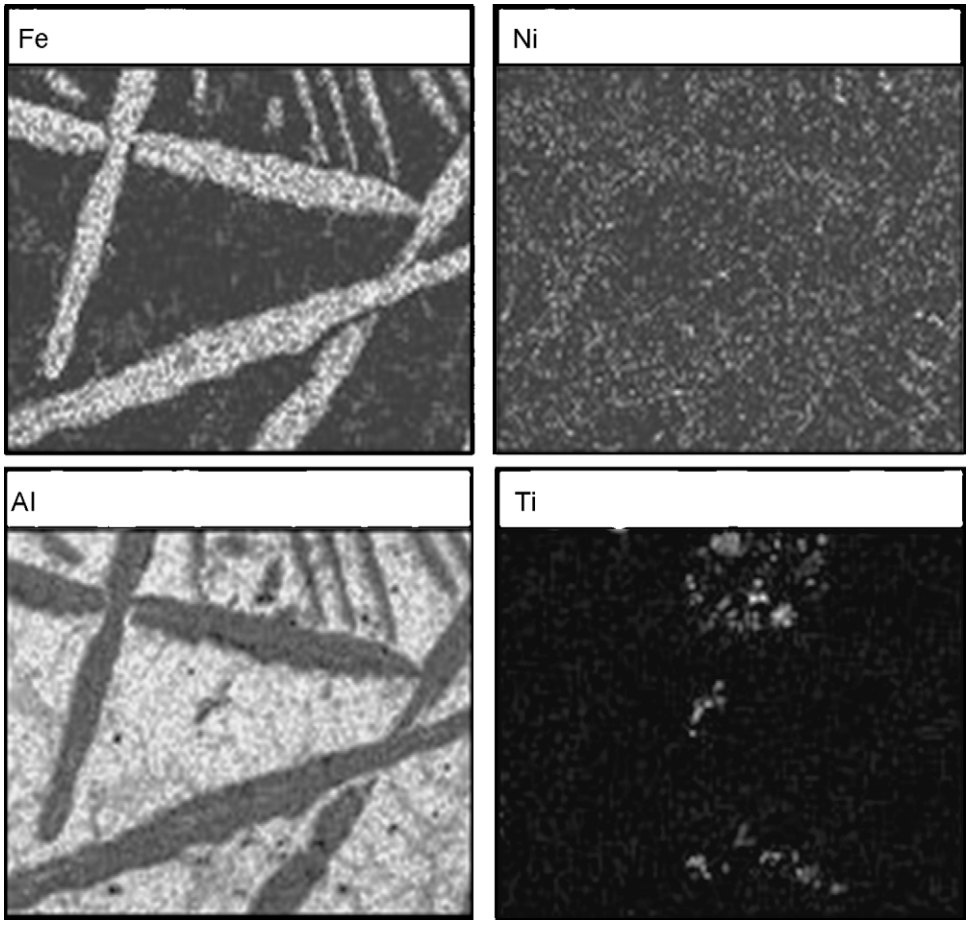

Fig. 2. Chemical mapping of Fig. 1(b).