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Microstructure of directionally solidified Ti–Fe eutectic alloy with low interstitial and high mechanical strength

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ABSTRACT

The performance of Ti alloys can be considerably enhanced by combining Ti and other elements, causing an eutectic transformation and thereby producing composites *in situ* from the liquid phase. This paper reports on the processing and characterization of a directionally solidified Ti–Fe eutectic alloy. Directional solidification at different growth rates was carried out in a setup that employs a water-cooled copper crucible combined with a voltaic electric arc moving through the sample. The results obtained show that a regular fiber-like eutectic structure was produced and the interphase spacing was found to be a function of the growth rate. Mechanical properties were measured using compression, microindentation and nanoindentation tests to determine the Vickers hardness, compressive strength and elastic modulus. Directionally solidified eutectic samples presented high values of compressive strength in the range of 1844–3000 MPa and ductility between 21.6 and 25.2%.

1. Introduction

Structural materials such as commercially pure (CP) titanium and its alloys are extremely important in a number of sectors, particularly in the transport, chemical, energy generation and biomaterial industries [1–5]. Although CP titanium possesses interesting properties, especially a good strength-to-weight ratio, high biocompatibility and enhanced corrosion resistance, its mechanical strength is relatively limited. However, its mechanical properties can be enhanced by alloying it with other elements. The mechanical behavior of the resulting alloys depends on the amount and type of alloying elements and processing routes applied. In this context, the performance of a structural material can be improved considerably by tailoring its microstructural morphology and the nature of its stabilized phases.

An approach to optimize structural materials involves combining Ti and other elements, which leads to an eutectic transformation and results in the *in situ* production of composites from the liquid phase [6]. *In situ* composites obtained by directional solidification processing of eutectic alloys have more attractive and special characteristics than their constituent phases. In this technique the eutectic liquid phase decomposition is used to produce two or more solid phases, which results in a refined microstructure whose phases are arranged side by side [7].

Eutectic transformation in the Ti–Fe system can be employed to produce *in situ* composite materials whose enhanced mechanical strength renders them potentially interesting structural materials. In composite materials obtained by eutectic solidification, the matrix is reinforced by means of eutectic precipitation. In these cases, the eutectic structure is composed of regularly dispersed reinforcing phase incorporated into the matrix. According to the Ti–Fe phase diagram shown in Fig. 1 [8] an eutectic phase transformation occurs at 1085 °C of a material with a composition of 32.5 wt% Fe whose liquid solidification gives rise to βTi solid solution phase and TiFe intermetallic compound. TiFe is stable at room temperature–1317 °C and presents a Pm-3m structure similar to that of CsCl, with a lattice parameter of a=0.2975 nm. The periodicity of the eutectic array depends on aspects of the growth of its solid phases and of solute fluxes across the solid/liquid interface [7].

Experimental results of the microstructure and mechanical properties of as-cast Ti–Fe revealed a mechanical strength of 2.2 GPa and ductility of 6.7%, with a microstructure composed of TiFe intermetallic compound and βTi phase [9]. Recent attempts to improve the mechanical behavior of Ti–Fe alloy have focused on the addition of alloying elements to this binary eutectic alloy. The effect of the addition of Sn to the Ti–Fe eutectic on its mechanical strength and ductility was investigated by Das et al.

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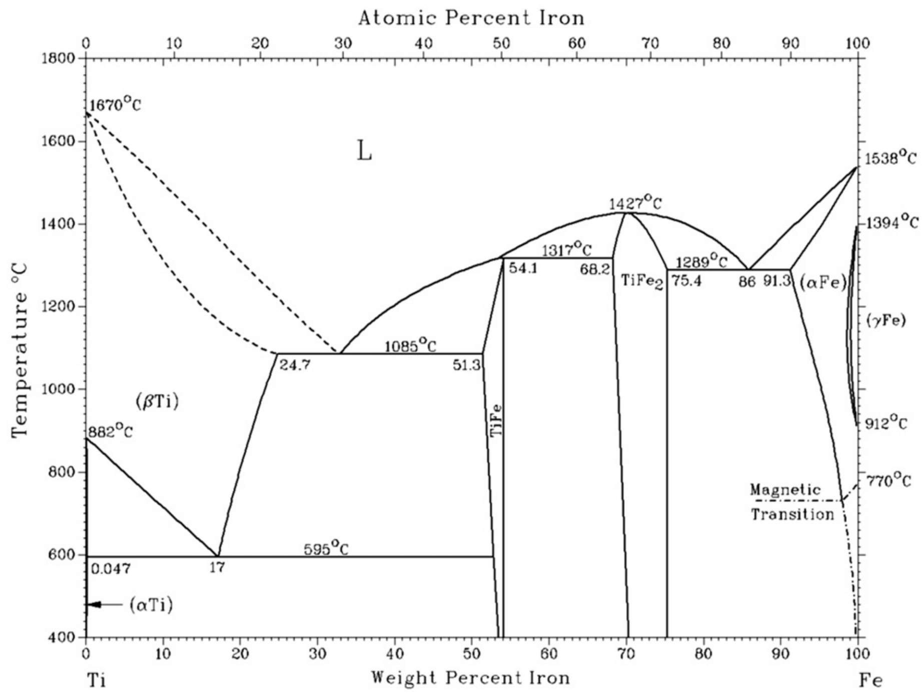

Fig. 1. Ti–Fe binary phase diagram [8].

[10]. It was found that adding Sn decreased the strength slightly but increased the ductility, while the changes in mechanical properties were attributed to changes in morphology and phase distribution in the eutectic microstructure [10]. In another study, the addition of Sn was found to result in ultrafine composites with fracture strength of 2350–2650 MPa and plasticity of 7.4–12.5% under compression [11]. Another study revealed that the addition of V leads to microstructural refinement and formation of metastable ω-phase. While a small amount of V added to the Ti–Fe eutectic alloy improved both its strength and plasticity a large amount of V resulted in the precipitation of ω-phase leading to the degradation of the alloy's mechanical properties, especially ductility [12]. The addition of Ga to Ti–Fe eutectic was recently evaluated by Misra et al. [13], whose experimental results suggested that the composites obtained *in situ* exhibit fracture strength of 2290–2766 MPa and plasticity of 4.8–6.5% under compression. The addition of In and Nb was also tested recently and the results suggested that alloys with a mechanical strength of about 2350 MPa and a ductility of 4.5% were obtained. An evaluation of the microstructure showed βTi dendrites as a primary phase and Ti (Fe, In and/or Nb)+βTi as the eutectic structure [14].

Very recently, Schlieter et al. [15] investigated the effect of solidification conditions on the microstructural and mechanical properties of Ti–Fe binary eutectic cast in different conditions. Samples were also solidified directionally by the Bridgman technique in Al_2O_3 crucibles coated with boron nitride to minimize oxygen contamination. X-ray diffraction results indicated precipitation of an oxygen solid solution phase (TiFeO solid solution), which may alter the properties of the resulting samples. Oxygen strongly affects the microstructure and mechanical behavior of Ti alloys; hence, its content should be strictly controlled [16]. Therefore, the accurate evaluation of the properties of a directionally solidified Ti–Fe eutectic sample is possible only in the absence of oxygen contamination.

Considering the potential use of Ti–Fe alloy as a structural material with enhanced mechanical properties, the purpose of this work was to prepare, process, and characterize directionally solidified Ti–Fe eutectics. In this work the directional solidification process was carried out in a novel setup that employs a water-cooled copper crucible combined with a moving voltaic electric arc under an inert atmosphere, thus preventing oxygen contamination.

2. Experimental procedure

The ingot samples of the Ti–Fe system studied here had 32.5 wt% of Fe content. They were prepared in an arc melting furnace using a nonconsumable tungsten electrode and water-cooled copper crucible in a high purity argon atmosphere. These ingots were remelted eight to ten times to ensure the homogeneity of their chemical composition and were produced from high purity Grade 2 Ti and high purity Fe (99.99%). The mass loss caused by melting the samples in the arc furnace was found to be less than 0.1 wt%. Samples 100 mm long and 15 mm in diameter were then solidified directionally in an apparatus built specifically for the directional solidification of titanium alloys, where samples were also electric arc-melted in a water-cooled copper crucible under argon atmosphere. As Fig. 2 shows, this directional solidification apparatus is equipped with a nonconsumable tungsten electrode that moves longitudinally along the ingot at different speeds. As the tungsten electrode and therefore the electric arc move, the sample remelts and subsequently solidifies under controlled conditions. After preliminary tests, the growth velocities chosen for the experiment were 10, 30 and 60 mm/h.

The metallographic preparation involved rubbing with SiC sandpaper up to 1200 grit, followed by polishing with a suspension of 6 and 1 μm diamond and ethyl alcohol as a lubricant and etching with a solution of 1 vol% HF, 2.5 vol% HNO_3, 1.5 vol% HCl and 95 vol% H_2O. The microstructural characterization involved optical microscopy—OM (Olympus BX60M), scanning electron microscopy—SEM (Hitachi TM-1000), transmission electron microscopy—TEM 200 kV (JEOL JEM 2100 HTP) and X-ray diffraction—XRD (PANalytical X'Pert diffractometer operating at 40 kV and 30 mA, Cu-K_α radiation, λ=0.15406 nm). Samples for