Materials Science and Engineering A 393 (2005) 366–373

MATERIALS SCIENCE & ENGINEERING A

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Studies on the formation and stability of nano-crystalline $Al_{50}Cu_{28}Fe_{22}$ alloy synthesized through high-energy ball milling

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Received 16 August 2004; received in revised form 26 October 2004; accepted 2 November 2004

Abstract

This work reports the formation of nano-crystalline $Al_{50}Cu_{28}Fe_{22}$ alloy by high-energy ball milling. The $Al_{50}Cu_{28}Fe_{22}$ alloy, synthesized through slow cooling of the molten alloy, was subjected to milling in an attritor mill at 400 rpm for 5 h, 10 h, 20 h, 40 h and 80 h with a ball to powder ratio of 40:1 in hexane medium. The X-ray diffraction and transmission electron microscopy observation of milled samples revealed that the milling duration of 5–40 h has led to the formation of nano-phase. The average crystallite size has been found to be of ∼17 nm. When the nano-crystalline alloy was vacuum annealed at a temperature of ∼500 °C for 5–20 h, new structural phases corresponding to the superstructures of crystallite size around 100–200 nm of the parent nano-crystalline phase were identified. The superstructures have been found to correspond to the simple cubic structure with $a_{sc} = \sqrt{2}a_p$ and the face centred cubic structure with $a_{fcc} = 2a_p$ (a_p: lattice parameter of parent nano-crystalline alloy). It has been proposed that the formation of two different types of superstructures resulting from ball milling followed by annealing is possibly governed by the minimization of free energy of phase constituents.

Keywords: Quasi-crystal; Nano-crystal; High-energy ball milling; Superstructure formation; B2 phase

1. Introduction

Recently there has been a considerable scientific and technological interest in the formation of nano-crystalline/quasi-crystalline phase in the Al–Cu–Fe alloys by mechanical milling [1]. Quasi-crystals have many properties which make them interesting for industrial applications like light weight, large strength to weight ratio and high hardness with a low frictional coefficient [2]. Nano-structured material, which can be defined as a material with the crystallite size less than 100 nm are synthesized by either "bottom-up" or "top-down" processes [3]. The bottom-up approach starts with atoms, ions or molecules as "building blocks" and assembles nano-scale clusters or bulk material from them. The "top-down" approach for processing of nano-structured materials starts with bulk solid and ends in obtaining a nano-structured phase through special processing routes e.g. mechanical milling, re-solidification through chemical methods etc. Nano-phase materials have significantly different behaviour from their macroscopic counterparts because their sizes are smaller than the characteristic length scales of physical phenomena occurring in bulk materials [4].

The nano-structure materials are produced by using various methods, among which high-energy ball milling (BM) which is also known as mechanical milling (MM) has attracted much attention [5]. The advantages of high-energy ball milling for the synthesis of nano-structured materials are the formation of a more homogeneous product and good reproducibility [6]. The mechanical milling technique has been used to obtain amorphous alloys [7], high coercively permanent magnetic metallic compounds [8] and quasi-crystals [9,10]. The formation of a quasi-crystalline phase by BM/MM has been reported in a number of Al and Ti based systems [11]. Recently Mukhopadhyay et al. [12]

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doi:10.1016/j.msea.2004.11.002

have studied the effect of mechanical milling on the stability of Al-rich, Al–Cu–Fe and Al–Cu–Co quasi-crystalline alloys. They have reported that the icosahedral quasi-crystalline phase in Al–Cu–Fe system undergoes transformation to a bcc (B2 type) crystalline phase as a result of ball milling [13]. In this case, B2 phase does not transform into any other crystalline/quasi-crystalline phase during isothermal annealing at 850 °C up to 20 h. It has been concluded that the B2 phase is more stable than the icosahedral quasi-crystalline phase at those compositions. It should be mentioned that Al-rich $Al_{65}Cu_{20}Fe_{15}$ system is of significant interest due to the high temperature structural stability of icosahedral quasi-crystalline phase. The available phase equilibria data indicate that the B2 phase is a major phase on the Al-deficient side of stoichiometry [14]. The B2 structure can be understood in term of ordering in a bcc lattice and converting it to be a simple cubic lattice. Therefore, unlike a bcc lattice, one type of atom occupies the body-centered position and another type occupies the cube corners in the ordered lattice. When the composition deviates from the stoichiometry, the compositional defects must be introduced to preserve the crystal structure. Its unit cell contains two different atoms located at the vertex and at the center of the cube, respectively. It is one of the basic simple structures that can transform into more complex structures via twinning at the atomic level, termed as chemical twinning [15]. The B2 type phase is often present together with the quasi-crystals and has fixed coherent orientation relationship with the latter [16,17]. The detailed investigation of the B2 phase is also important due to its practical applications [18,19]. Though quasi-crystals have many curious properties, they are also extremely brittle, porous and composition-sensitive. It is therefore interesting to substitute them by approximant materials, particularly B2 based ones, which are more easily prepared and have similar performance characteristics [20].

The purpose of the present study is to investigate the influence of high-energy ball milling on the phase stability, crystallite size, lattice strain and lattice parameter of B2 phase formed in the pre-alloyed $Al_{50}Cu_{28}Fe_{22}$ sample. Present investigation clearly shows the evolution of ordered simple cubic phase (a_{sc} = 4.12 Å) and as well as fcc (a_{fcc} = 5.8 Å) τ2 phase after milling followed by annealing. The evolution of the nano-structure at different stages of ball milling has also been investigated.

2. Experimental

An alloy of composition $Al_{50}Cu_{28}Fe_{22}$ was prepared by melting the high purity Al, Cu and Fe metals in an induction furnace, in the presence of dry argon atmosphere. The ingot formed was re-melted several times to ensure better homogeneity. The as-cast ingot was crushed to particles less than 0.5 mm in size and placed in an attritor ball mill (Szegvari attritor) with a ball to powder weight ratio of 40:1. The attritor has a cylindrical stainless steel tank of inner diameter 13 cm and the angular speed of mill was maintained at 400 rpm. The milling operation was conducted from 5 h to 80 h using hexane as a process control agent. The powder obtained after 10 h and 80 h of milling were annealed isothermally at 500 °C for 5–20 h in the evacuated quartz capsules (with vacuum of 10^{-6} torr). The milled and heat-treated powders were characterized by powder X-ray diffraction (XRD) using a Philips 1710 X-ray diffractometer with Cu Kα radiation. The effective crystallite size and relative strain of mechanically milled powders as well as heat-treated products were calculated based on line broadening of XRD peaks. The use of the Voigt function for the analysis of the integral breadths of broadened X-ray diffraction line profiles forms the basis of a rapid and powerful single line method of crystallite size and strain determination. In this case, the constituent Couchy and Gaussian components can be obtained from the ratio of full width at half maximum intensity (2ω) and integral breadth (β) [21]. In a single line analysis the apparent crystallite size 'D' and strain 'e' can be related to Couchy (β_c) and Gaussian (β_G) widths of the diffraction peak at the Bragg angle θ;

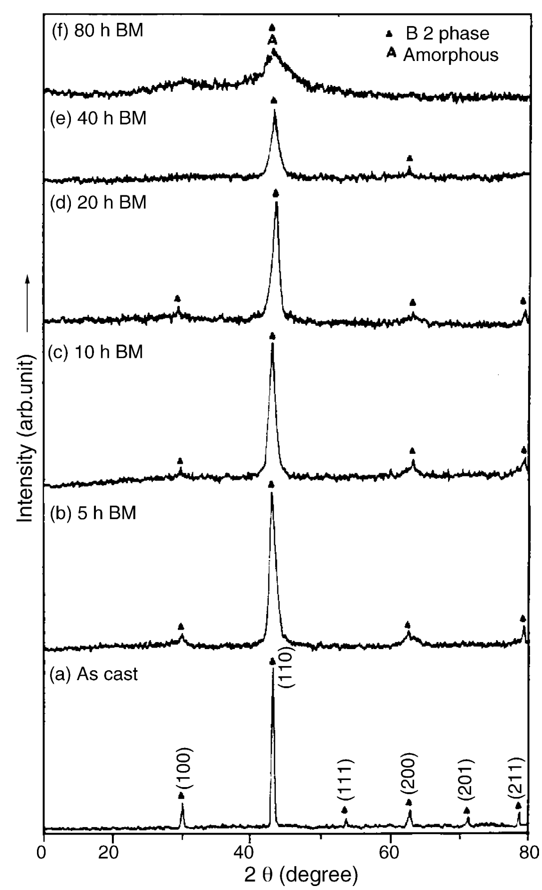

Fig. 1. X-ray diffraction patterns of as-cast powder (a), Ball-milled powders after various milling times 5 h (b), 10 h (c), 20 h (d), 40 h (e) and 80 h (f). From (a), the presence of micro B2 phase in the as-cast alloy was confirmed and (b–e) show the existence of nano B2 phase.