Acta Materialia 105 (2016) 273–283

Contents lists available at ScienceDirect

Acta Materialia

journal homepage: www.elsevier.com/locate/actamat

Full length article

Anisotropic dynamic compression response of a directionally-cast silver–copper eutectic alloy

O.T. Kingstedt [a], B.P. Eftink [b], I.M. Robertson [c], J. Lambros [d], *

[a] Division of Engineering and Applied Science, California Institute of Technology, Pasadena, CA 91125, USA
[b] Department of Materials Science and Engineering, University of Illinois at Urbana-Champaign, Urbana, IL 61801, USA
[c] Department of Materials Science and Engineering, University of Wisconsin-Madison, Madison, WI 53706, USA
[d] Department of Aerospace Engineering, University of Illinois at Urbana-Champaign, Urbana, IL 61801, USA

ARTICLE INFO

Article history:
Received 23 October 2015
Received in revised form
5 December 2015
Accepted 13 December 2015
Available online 9 January 2016

Keywords:
Eutectic
Ag–Cu
Dynamic plastic deformation
Interface structure

ABSTRACT

At the eutectic composition, the silver–copper bi-phase system can be produced such that the Cu phase forms as discontinuous reinforcements within a silver matrix. The reinforcements and matrix have a 101 growth direction along the axial direction of the produced material. This work examines the compressive deformation response of four such unidirectional Ag–Cu materials with nominal, bilayer thickness of 3.6 μm, 2.2 μm, 1.1 μm and 500 nm. From the cast material, specimens were machined to investigate the material response parallel (0°) to the ⟨101⟩ growth direction, 45° to the growth direction, and perpendicular (90°) to the growth direction. Specimens were loaded dynamically under compression using a split-Hopkinson pressure bar. The strength response of the unidirectional material was found to increase as the bilayer thickness decreased with the 0° orientation having the greatest strength followed by the 90° orientation then the 45° orientation. Deformation mechanisms were examined using postmortem microscopy. The orientation dependent strength and strain hardening are attributed to slip system orientations, deformation twinning, and the Ag–Cu interface structure.

1. Introduction

An attractive method to increase the mechanical properties of materials is by increasing the density of interfaces (e.g., grain boundaries, twin boundaries, phase interfaces) present throughout the material. Grain boundary density is commonly controlled through thermo-mechanical processing routes, such as rolling, equal channel angular pressing [1,2], high pressure torsion [3] and consolidation methods [4,5]. However, after microstructure refinement the grain boundaries are often thermally unstable resulting in relaxation of mechanical properties. Previous work on silver processed using high pressure torsion observed relaxation at room temperature over prolonged periods of time [6]. Bi-phase interfaces of low miscibility materials however have been shown to be stable at elevated temperatures at high interfacial densities. Bi-phase interface density can be controlled at the microscale in deposited multilayer systems and at the bulk scale through eutectic casting techniques or accumulative roll bonding [7].

Recent work has focused on the study of the mechanical properties of multilayered deposited metal–metal [8,9] and metal–ceramic [10] films under nano-indentation [11,12], tensile loading [13], rolling [14,15], and micro-pillar compression [16]. These multilayered thin film material systems are typically manufactured using physical vapor deposition (PVD) to produce layers that are single or polycrystalline [8]. An advantage of PVD is generating highly controlled geometries (e.g., uniform and controllable layer thicknesses), but PVD has the drawback of only being capable of producing films with total thicknesses of a few microns, limiting their structural applications. Many metal–metal and metal–ceramic multilayer systems have been seen to demonstrate a non-linear relationship of increasing hardness with decreasing layer thickness. In most cases, the hardness measure used to estimate yield stress is taken at a compressive strain value well above the traditional 0.2% offset in a uniaxial stress experiment. Therefore, the estimate of yield strength, σ_y, commonly determined through the Tabor [17] relation for hardness, $H\ (\sigma_y = H/3)$ will also be affected by the strain hardening response of the multilayer material system. The underlying mechanisms responsible for the non-linear strength increase have been studied previously – an excellent review is provided by Wang and Misra [18]. The proposed

---

* Corresponding author.
E-mail address: lambros@illinois.edu (J. Lambros).

http://dx.doi.org/10.1016/j.actamat.2015.12.024
1359-6454/© 2015 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

mechanisms include, the Hall-Petch model [19,20] for dislocation pileups which determines the logarithmic slope of the non-linear relation of yield strength when layer thicknesses are greater than 50 nm. For layer thicknesses below 50 nm modified versions of confined layer slip (CLS) [21] and single dislocations crossing the interface [21,22] have been proposed.

While previously conducted work on metallic multilayers has demonstrated that dislocation mechanisms are influenced by the spacing and structure of bi-phase interfaces, further experimental study and material characterization particularly of *bulk* materials with a high density of bi-phase interfaces is necessary for the following reasons: 1) bulk materials offer the ability to study loading orientations beyond predominantly normal to the interface (as in nano-indentation), and 2) the ability to study material response over the quasi-static and dynamic regimes. Through the examination of bulk materials with a high density of bi-phase interfaces additional insight can be gained on how the deformation mechanisms observed in previous thin film studies translate to bulk materials.

One method to produce a bulk bi-phase material with a high density of interfaces is the casting of bi-phase eutectics [23–26] with low miscibility, such as Ag and Cu, the bi-phase system of choice for this study. In flux melt casting a highly purified melt can achieve undercooling levels necessary for the solidification of submicron microstructure [20,24]. With high undercooling and rapid solidification the resulting Ag–Cu system microstructure consists of lamellar eutectic colonies. These colonies initiate at random points throughout the material, hence there is little control over their orientations. By subsequently growing these colonies directionally with the Bridgman technique [27], the Ag–Cu system forms a well-ordered directional microstructure [28–30] of Cu reinforcements aligned to the cast rod axial direction within a Ag matrix. By adjusting the rate at which the melt is removed from the furnace the length scale of the microstructure can be controlled.

Previous study of directionally cast Ag–Cu material has been limited. The quasi-static material response of a Ag–Cu eutectic with reinforcement spacing between 100 nm and 3.7 μm was studied by Cline and Stein [28] with loading parallel to and at 45° to the reinforcement direction, at room and at elevated temperatures. A linear Hall-Petch type relation was obtained between the reinforcement spacing and yield strength, pointing to dislocation pileups at the bi-phase interface. Similar linear relationships of lamellar spacing and yield strength have been observed in other directional cast eutectic systems [31,32]. A recent study by Eftink et al. [30] examined the dynamic compression response of directional Ag–Cu eutectic with microstructure features on the order of a micron. Transmission electron microscopy (TEM) found communication of deformation across the bi-phase interface in the form dislocation slip and deformation twinning. Under the loading conditions used (strain rates on the order of 10^3 s^{-1} at room temperature) deformation twinning would be expected in the Ag phase but not in Cu as Cu only exhibits deformation twinning at very high strain rates or cryogenic temperatures [33–38]. The deformation twinning observed in Cu within the $Ag_{60}Cu_{40}$ eutectic, resulted from the transmission of twinning partials from Ag across the cube-on-cube bi-phase interface into Cu [39].

The motivation for the present work is to study the response of a thermally stable directional material system with a high density of interfaces to ascertain if the deformation mechanisms and strength increases observed in deposited multilayer systems can be translated to bulk scale materials. The objectives of this work are to: 1) determine the effect of loading orientation with respect to the solidification direction on the mechanical response, 2) study the effect of decreasing microstructure feature size on yield strength and hardening, and 3) relate deformation mechanisms observed at the meso- and microscale to dislocation slip and twinning occurring at the nanoscale. The following sections of this manuscript present the processing, characterization, and dynamic mechanical response of the directional cast $Ag_{60}Cu_{40}$ eutectic material system. A discussion comparing the material response under three loading orientations, 0°, 45° and 90° to the growth, at four different microstructure bi-layer thickness, namely 500 nm, 1.1 μm, 2.2 μm and 3.6 μm, is presented.

2. Experimental methodology

2.1. Bulk material production

Following a method similar to that of Eftink et al. [31], directionally solidified $Ag_{60}Cu_{40}$ specimens were cast. Ag and Cu at the eutectic composition ($Ag_{60}Cu_{40}$) were heated to 1223 K within a vacuum sealed fused silica tube melting the components. The fused silica tube was removed from the furnace at a constant rate promoting directional solidification of the Ag and Cu phases. Three removal rates were used in this study, namely 0.46 mm/h, 7.3 mm/h and 73 mm/h, as a method to control the resulting size scale of the solidified microstructure. These low cooling rates resulted in a material with discontinuous Cu reinforcements (i.e., globular or lamellar platelets) elongated in the axial direction surrounded by a Ag matrix. Both the Ag and Cu phases grew with a common [101] direction parallel to the rod axis [29,31]. Following solidification, the cast rods were vacuum annealed (10^{-6} torr) at 673 K for 4 h. Annealing was conducted to reduce the solid solution solubility of the phases within each other, approaching solubilities on the order of 0.5 atomic percent [40].

2.2. Experiments: specimen preparation and loading

Using electrical-discharge machining (EDM), specimens were cut from cast rods such that different loading orientations could be studied, namely parallel to the growth direction (0°), 45° to the growth direction (45°) and perpendicular to the growth direction (90°). A schematic detailing the three selected orientations for dynamic experiments is presented in Fig. 1, with arrows indicating the compressive loading directions. Because of the somewhat variable size of the initial casting, the final dimensions of the 0°, 45° and 90° specimens vary but maintain length to diameter ratios of one-half across all orientations. The nominal dynamic specimen diameters used were 8 mm, 7 mm, and 6.8 mm for the 0°, 45° and 90° specimens, respectively. A quasi-static experiment was also conducted on the 500 nm bilayer system parallel to the growth direction (0°). The quasi-static specimens had dimensions of 8 mm diameter and 16 mm length. Prior to testing all EDM surface damage was removed the radial and flat surfaces by mechanical polishing with a final polish using a 1 μm diamond suspension. Specimens were prepared for scanning electron microscopy (JOEL 6060LV) by first sectioning the material using EDM, then casing in a conductive epoxy, followed by mechanical polishing with a final step using a 0.3 μm Al_2O_3 slurry.

Quasi-static compression experiments were conducted on a servo-hydraulic load frame at a strain-rate of 10^{-3} s^{-1}. Machine compliance was accounted for using the method outlined by Kalidindi et al. [44]. Dynamic high strain-rate testing was conducted on a split-Hopkinson (or Kolsky) pressure bar (SHPB) [41]. For this study, the diameter of the striker, incident and transmitted bar were identical at 12.7 mm. In the SHPB a cylindrical specimen is sandwiched between two long elastic bars, the incident bar and transmitted bar. The incident bar is impacted with a striker bar launched via a gas gun initiating a stress pulse that travels along the incident bar length. Strain gauges are mounted on the surfaces of