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# Nanoindentation creep of ultrafine-grained Al<sub>2</sub>O<sub>3</sub> particle reinforced copper composites

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### 1. Introduction

Ultrafine-grained (UFG) and nanocrystalline (NC) materials have potential applications as structural materials due to their excellent mechanical properties, including high yield stress and strain rate sensitivity of flow stress [1]. As a unique method to test the deformation behaviors, nanoindentation technique is suitable for studying the mechanical behaviors of UFG and NC materials [2], particularly the creep behaviors of materials (called nanoindentation creep). The advantages of nanoindentation creep over the traditional tensile/compressive creep include time and cost efficiencies, since a single test can obtain the stress exponent of the materials and the testing time is normally less than half an hour. Another advantage of nanoindentation creep is that it can be used for very small samples. However, it should be noted that some disadvantages and controversies still exist for nanoindentation creep. First, primary creep might have continuous effects on the steady state strain rate measurements due to the short duration of most nanoindenataion creep [3]. Second, stress exponent determined by nanoindentation creep is usually very high, compared to the traditional tensile/ compressive creep testing results [4]. The mechanism of high stress exponent determined by nanoindentation creep is still unclear. Third, the stress beneath the indenter is normally highly localized and much complicated than that in traditional tensile/compressive creep tests [5]. Nevertheless, as a novel and efficient testing method, nanoindentation technique can still be used to test the creep behaviors of the materials if the corresponding theory can be

## ABSTRACT

In this paper, the creep behaviors of two  $Al_2O_3$  particle reinforced copper composites were investigated using nanoindentation creep testing method. It has been shown that stress exponent determined by nanoindentation creep is much larger than that determined by traditional uniaxial tensile/compressive creep. A low externally applied loading results in a large scattering of the stress exponent due to limited grains joining the creep deformation and thus the different strain rates resulted from different grain orientations to the applied stress. The stress exponent of the composites increases with the externally applied loading for both composites, and the composite with higher reinforcement concentration shows larger stress exponents. © 2012 Elsevier B.V. All rights reserved.

established. In this work, the creep behaviors of two  $Al_2O_3$  particle reinforced copper composites were studied using nanoindentation creep, in order to understand the influence of loading stress and microstructures on the creep behaviors.

#### 2. Experimental

Cu-based composites with nominal compositions of Cu-0.3Al<sub>2</sub>O<sub>3</sub>% and Cu-0.03Al<sub>2</sub>O<sub>3</sub>% (wt%) were received in an as-extruded state, and machined to the dimension of  $10 \text{ mm} \times 10 \text{ mm} \times 50 \text{ mm}$ . Equal Channel Angular Pressing (ECAP) of the samples was conducted at room temperature with B-mode for 8 times. The total strain after ECAP is about 8. All the specimens were then polished using emery paper and fine alumina to meet the requirements for nanoindentation tests. A JEM2000 FX transmission electron microscope was used to determine the microstructures of two as-ECAPed composites. TEM thin foils were cut from the center of the as-ECAPed rods perpendicular to the longitudinal axis. For examining the grain boundary structures, electron backscattered diffractions (EBSD) were conducted on a scanning electron microscope equipped with a field emission gun and an HKL-Nordlys II EBSD detector. The creep behaviors of the composites were tested using nanoindentation creep method conducted on a MTS  $XP^{\mathbb{M}}$  indenter at room temperature, with a strain rate of 0.05  $s^{-1}$ . The maximum loads were ranging from 1 to 500 mN. After the maximum load was reached, it was fixed and held for 300 s to exam the creep behaviors. The displacement-time curves were correlated according to the following relationship [6]:

 $y = A(x - x_c)^p + y_0 = kx$  (1)

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(3)

Based on the load and displacement data, the effective strain rate and stress were calculated according to Ref. [7]:

$$\dot{\varepsilon} = \frac{1}{h} \frac{dh}{dt}$$
(2)

$$\sigma = \frac{1}{24.56h^2}$$

where h is the indentation depth, t is creep time. The stress exponent, n, which is an indicator of the creep behavior, was obtained from the slope of the fitted strain rate–stress line. As the value of n differs in different parts of the strain rate–stress relationship, only the one in the steady creep state was adopted for comparisons between different specimens. Every n value was averaged from 4 independent indents.



Fig. 1. Initial microstructures of two as-ECAPed copper based composites with the  $Al_2O_3$  content of (a) 0.03% and (b) 0.3%.



Fig. 2. EBSD profiles of (a) Cu–0.03%Al $_2O_3$  and (b) Cu–0.3%Al $_2O_3$  composites.

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