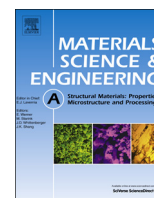




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## Creep behavior of two Cu-2 vol% TiC alloys obtained by reaction milling and extrusion



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## ABSTRACT

The creep behavior of two Cu-2 vol% TiC alloys prepared by reaction milling and extrusion are presented. Creep tests were performed at 773–1123 K. The activation energy values between 109 and 156 kJ/mol and the stress-exponent values between 3.1 and 6.3 indicated that no dispersion-strengthening effect occurred.

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## 1. Introduction

Alloys exhibiting high mechanical strength combined with high electrical and thermal conductivity at elevated temperatures are in increasing demand [1]. Because it has high electrical/thermal conductivity, copper is a promising metal for these types of applications if its high-temperature strength could be improved.

On the other hand, the creep properties of pure metals are often characterized through the dependence of secondary creep rate  $\dot{\epsilon}$  on stress ( $\sigma$ ) and temperature ( $T$ ) using power law equations as follows [2]:

$$\dot{\epsilon} = A\sigma^n \exp\left(\frac{-Q}{RT}\right) \quad (1)$$

where  $R$  is the gas constant. The parameter ( $A$ ), the stress exponent ( $n$ ), and the activation energy for creep ( $Q$ ) are also functions of stress and temperature. Also, for a given  $T$  value the above expression may be written as

$$\dot{\epsilon} = B\sigma^n \quad (2)$$

As reviewed by Langdon [3], over a wide range of high and intermediate stresses, the steady-state creep rate in pure metals and their solid solutions varies with stress raised to a reasonable high power of  $n$  ( $\sim 3$ – $5$ ). In this region the flow is dominated by the intragranular movement of dislocations: this behavior is generally termed power-law creep. At even higher stresses, the

creep rate varies exponentially with stress where this behavior marks a transition from diffusion-controlled flow at the lower stresses to a thermally-activated region analogous to flow at low temperatures. This transition is generally termed the power law breakdown (PLB). The flow mechanism is also characterized by the activation energy  $Q$  in relation to the controlling diffusive process. On the other hand, dispersion-strengthened materials, in comparison with pure metals and solid solutions, present higher  $n$  and  $Q$  apparent values [4].

The high-temperature strength of metallic alloys may be increased by adding a small fraction (e.g., between 2 and 5 vol%) of ceramic dispersoids. Several researchers have demonstrated that the addition of appropriate nanometric dispersoids may increase the creep resistance of copper based alloys [4–6]. Adding elements that form insoluble particles affects the electrical conductivity less than forming a solid solution. These dispersoids must be thermodynamically stable, nanometric in size, and homogeneously distributed within the metal matrix [1]. Additionally, when ceramic dispersoids are present, the strength of the material at high temperatures is primarily controlled by dislocation-particle [7] and grain boundary-particle interaction [8]. Reaction milling is a modern process that uses mechanical alloying to the in-situ development of nanometric dispersoids within a metal matrix [9]. Elemental powders are milled under a specific atmosphere combined with a liquid milling media to allow one or more metals to react with C, N, or O. Microcrystalline grains develop a high dislocation density because of attrition. This process also requires that the milled powders be consolidated using thermomechanical approaches, such as hot extrusion. In our previous studies [10,11], different dispersion-strengthened Cu alloys were prepared by

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reaction milling, in a small high-energy ball mill (Spex 8000D) and a medium-energy home-made attritor mill.

The aim of the present work is the study of the creep behavior of two Cu-2 vol% TiC alloys prepared by reaction milling in an attritor or Spex mill followed by hot extrusion. The effects of creep temperature, strain rate, and milling procedure were examined.

## 2. Experimental procedure

Two nominally composed Cu-2 vol% TiC alloys were produced by reaction milling from dendritic elemental powders of Cu (85 wt% under 40  $\mu\text{m}$ ) and Ti (< 45  $\mu\text{m}$ ). The milling process was performed in a Szegvari-type attritor or Spex mill, with rotational speeds of 500 rpm or 1.200 rpm, and milling times of 30 h or 10 h, respectively. All millings utilized a ball to powder weight ratio of 10:1, a nitrogen atmosphere, and toluene as the milling medium. The C necessary to form the nanodispersoids in-situ was provided by toluene. The resulting aggregates were encapsulated under low vacuum before being consolidated using hot extrusion at 1023 K and with a 10:1 extrusion ratio. These experimental conditions were based on previous results in our laboratory. Compression-test cylindrical specimens 6 mm in diameter and 10 mm in length were machined from the as-extruded bars such that the machined axis was parallel to the extrusion direction. Constant-stress compression creep tests were performed in a TT-DM Instron machine equipped with a high-temperature compression device, at 773,

973 and 1123 K; the utilized loads produced steady-state creep rates between  $1.1 \times 10^{-9}$  and  $2 \times 10^{-6} \text{ s}^{-1}$ . X-ray diffraction (XDR) was performed with a Siemens D5000 system. The density was determined using the Archimedes method.

## 3. Results and discussion

The chemical analyses of the powders treated in the Spex and in the attritor mills are presented in Table 1. With the Ti content and the excess C detected, vol% TiC values were calculated for each alloy, as indicated in Table 1; the vol% TiC values calculated for the Spex- and attritor- treated alloys, were 2.1 and 2.2, respectively. Additionally, O and Fe contamination was also detected; the former most likely originated from the  $\text{N}_2$  gas and surface Cu oxides, while the latter arose from the steel milling balls. The measured density of both extruded alloys was approximately 95% of the theoretical density.

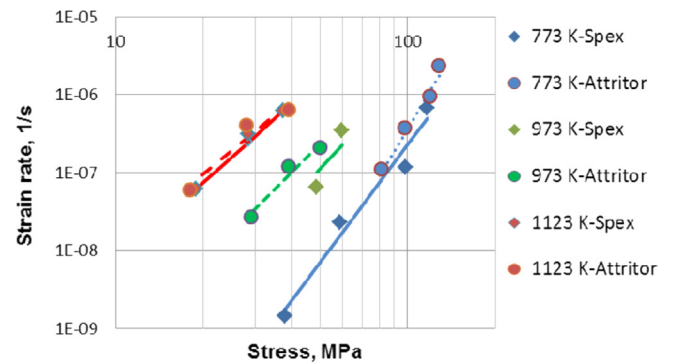
In Fig. 1, XRD diagrams of the Cu-Ti powders formed under different processing conditions are shown. Fig. 1(a) depicts the expected XRD data for powders immediately after blending; only peaks associated with Cu were detected. However, in Fig. 1(b) and (c), corresponding to the powders milled in the Spex and attritor facilities, respectively, a small maximum at a diffraction angle  $2\theta = 41.9^\circ$  appears in addition to the Cu peaks. A peak at this angle might be ascribed to the most intense TiC reflection at the {200}

**Table 1**  
Chemical composition of Cu-Ti-C powders.

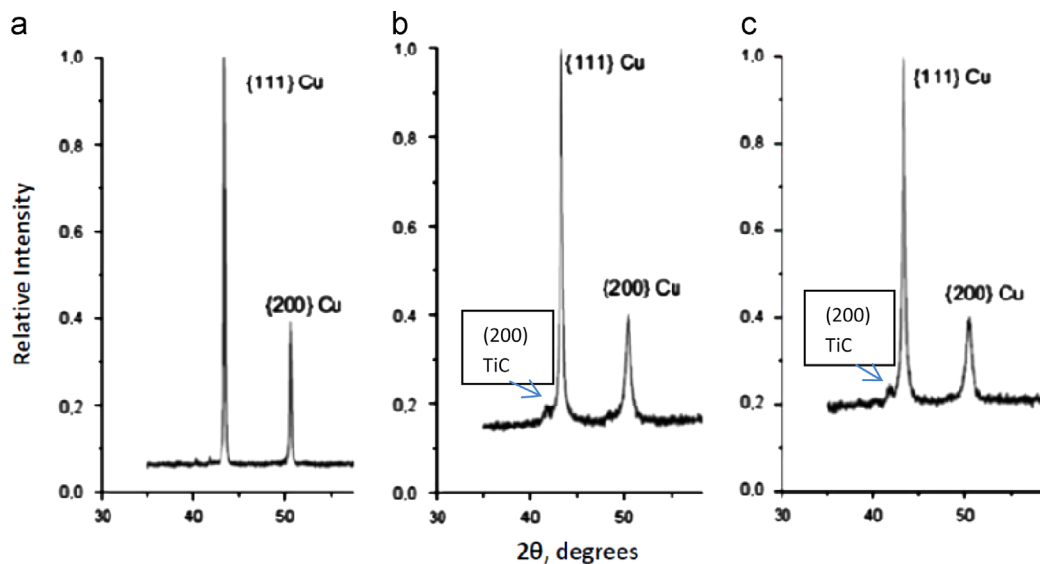
Element	Calculated element amounts in wt%, to obtain a Cu-2 vol%TiC alloy	Measured amounts, wt%	
		Spex milled powders	Attritor milled powders
Ti	2.25 <sup>a</sup>	0.93	1.03
C	0.56 <sup>a</sup>	1.21	0.69
O	–	0.37	0.59
Fe	–	0.10	0.17
TiC vol%		2.1 <sup>b</sup>	2.2 <sup>b</sup>

<sup>a</sup> Assuming a 50 wt% loss of each element, similar to previous results.

<sup>b</sup> Calculated for the measured Ti wt% with C in excess.



**Fig. 2.** Effects of compression stress on the strain rate at different temperatures for the alloys.



**Fig. 1.** XRD diagrams of Ti-C powders under different processing conditions: (a) immediately after blending; (b) after milling in the Spex facility; and (c) after milling in the attritor facility.

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