Mechanical properties and fracture study of alumina dispersion hardened copper-based nano-composite at elevated temperatures

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Abstract. Alumina dispersion hardened copper-base composite was fabricated by internal oxidation method. The high temperature tensile fracture of Cu-\textit{Al}_2\textit{O}_3 composite was studied and tensile strengths were determined at different temperatures of 600, 680 and 780 °C. Microstructure was investigated by means of optical microscope and field emission scanning electron microscope (FESEM) with energy dispersive spectroscopy (EDS). Results show that, ultimate tensile strength and yield strength of copper alumina nano-composite decrease slowly with increasing temperature. The yield strength reaches 119 MPa and ultimate tensile strength reaches 132 MPa at 780 °C. Surface fractography shows a dimple-type fracture on the fracture surface of the tensile tests where dimple size increases with increasing testing temperature and in some regions brittle fracture characteristics could be observed in the fracture surface.

Introduction
In recent years nanostructured materials have attracted much interest because of their considerable characteristics. It is known that by introducing fine dispersed particles into a metal matrix, significant reinforcing effects will achieve, which can be kept at elevated temperatures [1]. For this kind of composite materials, ultrafine particles of oxides are suitable reinforcement, because of their hardness, chemical stability and insolubility in the metal base. The Orowan mechanism is the best explanation for the strengthening mechanism of these composites, particularly when the oxide particle size is below 100 nm [2]. Copper alumina nano-composite can be fabricated by mechanical alloying [3, 14], thermo-chemical processing [3-5], electro-deposition [6] and internal oxidation [7-9]. Internal oxidation is proposed as the most suitable process for the synthesis of Cu-\textit{Al}_2\textit{O}_3 composite. The size of \textit{Al}_2\textit{O}_3 particles in the Cu based composite powder produced by means of this process is ranged from 10nm to 100nm [10]. Applications of Cu-\textit{Al}_2\textit{O}_3 nano-composite include electrode materials for spot welding [8], relay blades [11] and electrical and heat conductors where high temperature strength, high electrical conductivity and heat conductivity are required. Dispersion strengthened Cu-\textit{Al}_2\textit{O}_3 composites have shown significant thermal and mechanical stability at high temperatures. [12]. M. Entezarian and R.A. Drew[13] have suggested that the strong bonding at the interface of copper and alumina is the reason of high strength of Cu-\textit{Al}_2\textit{O}_3 composite. Also A. Simchi and H. Simchi [9] have shown that the tensile fracture mechanism revealed a ductile type fracture in the microscopic scale and shows micro-void formation and coalescence in fracture surface.

The purpose of this work is to study the high temperature fracture mechanism of Cu-based alumina dispersed nano-composite s. High temperature tensile testing of copper alumina nano-composite is also discussed.
Experimental

Cu-Al_{2}O_{3} nano-composite samples were prepared by internal oxidation technique. The preparation process includes the following stages: (i) producing Cu-0.5%Al pre-alloyed powder by water atomization method; (ii) Oxidation of atomized Cu-Al alloy powder at approximately 900°C for 1 hour in the air; (iii) Reduction in hydrogen atmosphere at 800°C for 1 hour; (iv) Pre compacting of powder at 500 Mpa in a copper can; and (v) Extruding of pre-compacted sample at 900°C in an argon atmosphere to a rod with 17 mm diameter followed by drawing to get a high density of dislocations. Tensile properties at ambient and elevated temperatures were determined in accordance to the standard test methods (ASTM E8M). The tension test specimens have a gauge length of 20 mm and a diameter of 4 mm. Tensile tests were performed at room temperature, 600, 680 and 780°C and the cross-head speed was chosen 10 mm/min (ASTM E8M). The Brinell hardness of the specimens was determined according to ASTM E10-01 standard test method with 250 kg load. The microstructure and fracture surface of specimens were observed using a field emission scanning electron microscope (FE-SEM) with energy dispersive spectrometer (EDS).

Results and Discussion

The results of high temperature tensile tests and hardness value of the Copper-Alumina nano-composite are shown in table 1. It can be seen that ultimate tensile strength and yield strength decrease slowly as the temperature increases (Fig.1 (a)). High strength of copper alumina nano-composite in comparison with pure copper may be related to the Orowan strengthening theory [2] or the strengthening caused by the increase of dislocation density. Orowan strengthening mechanism indicates the strengthening effect of dispersive particles illustrated by the equation of

\[ \Delta \sigma = \left( \frac{0.13Gb}{\lambda} \right) \ln \left( \frac{r}{b} \right) \]  

where G is the shear modulus of the matrix, b is the burgers vector of the matrix, \( \lambda \) is the interparticle spacing and r is the radius of the reinforced particles. Strengthening caused by the increase of dislocation density, results from different thermal expansion coefficient of the matrix and particles[2].

\[ \Delta \sigma = k G \rho^{-1} \]  

In this equation k is a constant and \( \rho \) is dislocation density. At elevated temperatures Al_{2}O_{3} particles block the formation and growth of recrystallization location in deformed matrix and the movement of the large angle boundaries. Dislocation slip mechanism at ambient temperatures changes to slip and climb mechanism with increasing temperature, thus work hardening competes with recovery and recrystallization. Moreover, at elevated temperature slip and rotation of grain boundaries could be resulted from the formation of micro-holes on the interface of Al_{2}O_{3} particles and copper matrix or on the crystal grain boundaries. These particles could act as recrystallization and grain growth barriers. As a result, the hardness decreases with a low slope at high temperatures. The reduction in area of the composite decreases rapidly with temperature increase up to 680°C and at these temperature range the elongation is about 4.3% (Fig.1 (b)). This results indicate that there is significant embrittlement appeared in composite at high temperature.

Table 1. Mechanical properties of Cu-Al_{2}O_{3} composites.

<table>
<thead>
<tr>
<th>sample</th>
<th>Test temperature</th>
<th>UTS [MPa]</th>
<th>Yield [Mpa]</th>
<th>%el</th>
<th>%r_{a}</th>
<th>Hardness [HBN]</th>
</tr>
</thead>
<tbody>
<tr>
<td>CA600</td>
<td>600°C</td>
<td>274</td>
<td>189</td>
<td>4.8</td>
<td>4.6</td>
<td>105</td>
</tr>
<tr>
<td>CA680</td>
<td>680°C</td>
<td>173</td>
<td>132</td>
<td>4.8</td>
<td>5.6</td>
<td>92</td>
</tr>
<tr>
<td>CA780</td>
<td>780°C</td>
<td>132</td>
<td>119</td>
<td>5.4</td>
<td>5.6</td>
<td>89</td>
</tr>
<tr>
<td>CA25</td>
<td>RT</td>
<td>547.5</td>
<td>510</td>
<td>14.11</td>
<td>38.3</td>
<td>160</td>
</tr>
<tr>
<td></td>
<td>((%e=0.2))</td>
<td></td>
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</table>
Fig. 1. The relation between strength (a) and plasticity (b) of Cu-Al$_2$O$_3$ composite with temperature.

Fig. 2 (a) shows a SEM fractograph of copper alumina nano-composite tested at 680°C with low magnification. The fracture surface of Cu-Al$_2$O$_3$ exhibits cup-and-cone morphology with shear type dimples at microscopic level and the fracture surfaces show some ridges and dimples presenting characteristics of ductile fracture. The excellent plasticity of copper resulted in the micro-voids and ridges in fracture surface. Also a layer of copper oxide is formed on the surface due to high temperature of the test. This oxide layer covered some features of fracture surface, especially fine micro-voids and dimples could not be observed even in higher magnifications. Nevertheless, the overall view could show a ductile mechanism for fracture. Also, soft matrix and high temperature of tensile test could result in this type of fracture, but micro-voids remained through fabrication process and some inclusions have changed the fracture surface in some regions. Thus, some sharp edges and shear planes are also observed in fractographs. Fig. 2 (b) shows some cracks formed during tensile testing. Interface of copper and alumina particle could be decohesion site of the particles from the matrix and causing formation of micro-cracks perpendicular to tension direction. These cracks contraction causes final fracture.

Fig. 2. The tensile fracture surface tested at 680°C (a) and cracks at surface fracture of Cu-Al$_2$O$_3$ composite (b)

Fig. 3 shows SEM fractograph of copper alumina nano-composite tested at the temperature of 600, 680 and 780°C, respectively. The tensile fractograph of testing at 600°C is covered by population of voids and dimples. Before the final fracture, plastic deformation caused equi-axial dimples which tend to elongate in tensile direction with the increase of the test temperature. These equiaxed dimples and voids are the main characteristics of the fracture surface of the samples. This could be a direct result of fine grain size and low cross slip occurrence in the matrix grains. Also the amount of dimples and ridges decrease with the increase of temperature (Fig. 3(a), (b) and (c)). Some inclusions were found in fracture surface (location "A" in Fig. 3(d)) which are considered as
the stable alumina or other inclusion such as Fe, Si and C, compounds remained from previous processes.

The fracture mechanism in Cu-Al$_2$O$_3$ composite is decohesion of the particles from the matrix and deformation of micro-voids (Fig. 3 (a)), which are expanded and coalesced to cause fracture. The region of stable crack growth for cracks meandered through the matrix and localized micro-plastic deformation (Fig. 3 (b)). Therefore, the fracture stages consist of micro-crack nucleation due to interfacial disconnection between matrix and reinforcement particles, growth of the cracks and final rupture. When the stress reaches the critical level, localized plastic deformation will start, pile-up of dislocations occurs at the interface of brittle alumina particles and ductile copper matrix and when dislocation pile-up reaches to some extent, microcrack nucleate and link each other and cause final rupture. Moreover, some flat areas were found in fracture surface, which demonstrate brittle fracture feature. The plastic deformation that is localized on the weak positions is the main reason of brittleness of Cu-Al$_2$O$_3$ nano-composite. Therefore Cu-Al$_2$O$_3$ nano-composite exhibits a hybrid ductile/brittle fracture mechanism. It is noticed that the alumina nanoparticles in the copper matrix have changed the micro-scaled deformation mechanism of the material. Some cleavage feature and cleavage facets (location "B" in Fig. 3(d)) are shown along with the ductile mechanisms including tearing ridges (location "C" in Fig. 3(d)). According to tensile tests results, the features of fracture surface morphology are in agreement with test data.

![Fig. 3 FE-SEM fractographs showing the fracture surface of Cu-Al$_2$O$_3$ composite at (a) 600°C, (b) 680°C, (c) 780°C and brittle fracture regions and inclusion in fracture surface of Cu-Al$_2$O$_3$ nano-composite.](image)

In addition, some alumina clusters were found on the Cu-Al$_2$O$_3$ composite rupture surface at elevated temperatures (Fig. 4 (a)). It could be considered that alumina particles are distributed non-uniformly in copper matrix (in agreement with the previously published data [15]), which have
negative effect on the mechanical behavior of the composite. For this reason, the aggregation of alumina particles should be avoided during the composite producing processes.

Fig. 4 SEM morphology and EDS spectra of cluster inclusion on the tensile fracture surface of Cu-Al$_2$O$_3$ composite at 600°C.

Conclusion
From the present work the following conclusions could be resulted:

1) By the use of nano-sized Al$_2$O$_3$ particle to fabricate copper based composites, high temperature strength was achieved. Ultimate tensile strength and yield strength of Alumina dispersion hardened copper-based composite decrease slowly by increasing temperature up to 600°C, but still remained 132 MPa and 119 MPa (UTS and Y.S., respectively) at 780°C.

2) Tensile fracture mechanism which is recognized as micro-void formation and coalescence, revealed a ductile nature of the nano-composite fracture in microscopic level. The size of voids and dimples increased with increase of testing temperature.

3) A combination of ductile and brittle fracture could be observed in the fracture surface of nanocomposites. Some Al$_2$O$_3$ particles agglomeration and other inclusion remained from pervious processes could decrease the strength of manufactured composites.

References