

ISSN (1897-3310) Volume 11 Special Issue 2/2011 71-74

FOUNDRY ENGINEERING

14/2

Published guarterly as the organ of the Foundry Commission of the Polish Academy of Sciences

Production and wear properties of copper based MMC strengthened with δ -alumina fibres

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Received 11.04.2011; Approved for print on: 26.04.2011

Abstract

The investigation was carried out on the production by pressure infiltration (squeeze casting) of metal matrix composite materials based on pure Cu-ETP copper containing δ -alumina SAFFIL fibres The microstructure of composite specimens and morphology was examined using SEM and optical microscopy. Physical properties: Brinell hardness HBW and density were characterized. Preforms with 10, 15 and 20 vol. % of fibres were preheated and infiltrated applying the infiltration pressure of 100 MPa. The strengthening of matrix with SAFFIL fibres resulted in significant increase of hardness. Metallographic examinations showed, that SAFFIL fibres are not destroyed in course of the infiltration process and are uniformly distributed in copper matrix. SEM observations confirm the poor wettability of fibres by liquid Cu-ETP. The wear of manufactured MMCs during dry sliding against cast iron applying a pin-on-disc tester were recorded after 1, 3.5 and 8.5 km of friction distance. Increasing content of SAFFIL fibres in the copper matrix results in the significant decreasing of wear.

Keywords: Metal matrix composite; copper, alumina fibres, squeeze casting, wear resistance

1. Introduction

Copper is characterized by high electrical and thermal conductivity as well as good corrosion resistance what places copper as a great prospective component of metal matrix composite materials [1-6]. However, poor adhesion to widely used reinforcements, chemical reaction at interfaces and quick oxidization of Cu can create serious problem in formation of the strong bonds at the interfaces between composite components. Molten Cu on solid alumina under protective atmosphere shows poor wettability and the contact angle is relatively high - 120-125° [7]. Taking this phenomenon into account after

solidification relatively weak joints are formed at the interface, and the relatively small transferring of loads from the matrix to the strengthening fibres takes place. As the result of discussed phenomena the medium mechanical and development properties of Cu based composite materials are reached. During typical infiltration process with molten copper on its exposed surface, Cu₂O forms, which next precipitates along interface fibresmatrix. Inhomogeneously distributed copper oxide adheres to alumina substrate with force dependent on oxygen partial pressure, temperature or lattice orientation [8]. At temperature above the eutectic temperature of 1065°C, CuAlO₂ oxide layer can be created according to the reaction:

$$Cu_2O + Al_2O_3 \rightarrow 2CuAlO_2 \tag{1}$$

This compound can be produced also be heat treatment and its formation can be controlled by applying Ar atmosphere [9]. According to the conditions oxide $CuAl_2O_4$ can be formed due to the intensive diffusion of aluminium into copper.

Alumina preforms used for infiltration with liquid alloys are frequently produced using inorganic binders, which significantly effect interface bonding and can promote some chemical reactions. Preforms used in presented study were covered with thin, nanometer silica layer which occurrence was confirmed by TEM investigations [10]. In this case wetting angle between Cu and SiO₂ is similar, 120-125°, but liquid penetration into silica and interdiffusion of Cu and Si, confirmed by [7] can improve bonding. The addition of Al into pure molten Cu can induce following exothermic reaction between silica and aluminium.

$$4Al + SiO_2 \rightarrow 2Al_2O_3 + Si + \Delta G \tag{2}$$

Thus, wetting in such reactive system depends on interfacial chemistry and can be tailored according to required composite properties.

Casting methods are the most economical way for manufacturing elements from copper and copper based composite materials. High pressure die casting has also numerous applications, but is followed by the formation of defects such as porosity or segregation failure of hot tears. Recently the most common production methods of MMC is infiltration of porous performs under pressure with molten alloys (squeeze casting). It is promising method for creating less defective cast components. Squeeze casting is divided into two types: direct squeeze casting, which is widely used for the production of parts characterized by the relative simple shapes and indirect squeeze casting applied to more complicated parts but it implies higher production cost.

In presented paper results of characterization of metal matrix composite: Cu + δ - alumina fibres are discussed. Microscopic observation allowed to determine proper parameters of casting process whereas wear examinations confirmed high reinforcing effect and improvement of resistance to wear.

2. Experiments

2.1. Materials

The composite materials were manufactured from the copper of 99.9 % purity. The basic physical properties of Cu - ETP are shown at Table 1.

Property	
Melting point	1083°C
Electrical conductivity	$57.0-58.0 \text{ m}/\Omega \cdot \text{mm}^2$
Thermal conductivity	385 W/m·K
Thermal expansion coefficient	17.7·10 ⁻⁶ /K
Young Modulus	130GPa
Hardness	81 HBW2.5/62.5

For infiltration the porous δ -alumina (SAFFIL) fibres (Table 2) preforms were used. The fibres contain an addition of 3-4% of silica, preventing from grain grown at high temperatures. Fibres were mixed in silica water solution, next drained out, formed and finally firedat 950°C to develop strong joints between fibres.

Table 2. Specification of SAFFIL alumina short fibres

Property	
Chemical composition	δ-Al ₂ O ₃ 96–97 %, SiO ₂ 3-4%
Tensile strength	2000 MPa
Density	3.3 g/cm^3
Length	100-300 μm
Diameter	2-4 μm

During the preparation of novel composites, preforms were preheated to 600°C and placed in preheated high-temperature steel die. The infiltration pressure of 100 MPa was applied and kept until solidification process was complete. In the course of squeeze casting process the manufacturing procedures were constantly monitored in order to control casing parameters. After squeeze casting the density was measured by Archimedes method and the results are shown at Table 3.

Table 3. Density of composite materials Cu-SAFFIL fibres

Materials	g/cm ³
Matrix	8.94
Composite with 10% Al ₂ O ₃ fibres	8.34
Composite with 15% Al ₂ O ₃ fibres	8.06
Composite with 20% Al_2O_3 fibres	7.79

2.2. Tribological examination

The investigations of wear were carried out on pin-on-disc tester without application of lubricant. Composite specimens were pressed against counterparts which was cut-out from the cast iron brake disk. The composite specimens of diameter 7,1mm acting on the wear track - radius 30mm were fastened in the immovable position and pressed down with the pressure of 1 MPa to the disc, which was rotating with a speed of 318 r.p.m (linear velocity 1m/s). The friction forces and mass loss were controlled after 1, 3.5 and 8.5 km of friction way.

3. Results and discussion

The structures of composite materials on the base of Cu-ETP containing 10, 15 and 20 vol.% of SAFFIL fibres were examined by optical microscope, whereas the tensile fractures were observed by scanning electron microscopy. Observations of microstructure exhibits relatively homogeneous distribution of fibres with planar random orientation in one direction (Fig.1), and semi-oriented in perpendicular cross section.



Fig. 1. Microstructure of Cu based composite material reinforced with 10 vol. % of SAFFIL fibres

The investigations revealed rather poor wetting of ceramic alumina fibres by liquid copper. After infiltration of porous skeleton metal crystallized heterogeneously on fibre surfaces. It results in decreasing of grain size from ca. $100-150\mu m$ in unreinforced Cu to $20-30\mu m$ in composite materials, see Fig.2.

Hardness HB examinations shown significant reinforcement effect, Table 4. Composite materials exhibit about 2 times higher hardness in relation to pure Cu casting. Values for all composites are comparable, what presumably results from refinement of structure and work hardening due to thermal mismatch between alumina fibres (thermal expansion coefficient for fibre $6-7\cdot10^{-6}$ /K) and Cu (Table 1).

Table 4. Hardness HBW of composite materials

Materials	HB
Matrix Cu-ETP	65
Composite with 10% Al ₂ O ₃ fibres	s 120
Composite with 15% Al ₂ O ₃ fibre	s 108
Composite with 20% Al ₂ O ₃ fibre	s 112



Fig. 2. Microstructure of the composite material: Cu with 15 vol. % of SAFFIL fibres (etched with the Mi18Cu reagent)

Scanning observations of fracture surface of composite materials after tensile tests shown relatively good bonding between reinforcement and the matrix, see Fig. 3. Fibres normal oriented to the fracture were not pull out, whereas parallel were bended and detached leaving grooves with characteristic pattern.



Fig.3. Fracture of Cu based composite materials reinforced with 10% of $Al_2O_3\ fibres$

Before fracture developed brittle fibres and interface cracked. At Figure 4, on the right side, thin gap between Cu and fibres was created. Presumably, without any oxide products connection was very weak and acted as a discontinuity of material.



Fig.4. Fracture of Cu-20 vol. % of Al_2O_3 composite material and magnified area with detached fibres

On the other hand, when infiltration reaction (1) proceeded and $CuAlO_2$ oxides were formed, as firmly attached particles to the fibre surface and they enhanced interface quality and the reinforcing effect. Fig.5 shows small oxide particles fixed to the fibres surface which after fracturing left corresponding traces of their shapes in the Cu matrix.

Tribological examination confirmed proper reinforcing effect and high wear resistance of composite materials, see Fig. 6. In relation to unreinforced Cu matrix composite material reinforced with 20% of fibres exhibits ten times lower wear loss (0.06g after 8.5 km distance). Reinforcing with 15% of fibres results in similar improvement, thus it could be ascertained that this is sufficient volume of fibres for the effective increasing of wear resistance.



Fig.5. Fractured bonding with reaction products on surface of alumina fibre



Fig.6. Mass loss during wear tests of copper based composite materials containing SAFFIL fibres

4. Conclusions

Cu- SAFFIL fibres composite materials are successfully produced by pressure infiltration and their microstructure, hardness and wear were studied in this paper. The key conclusions from our experiments are:

1. Alumina fibres are uniformly embedded in a copper matrix and during squeeze casting process no destruction of ceramic preform was observed.

2. SAFFIL fibres effect on the significant increase of the hardness of the composite materials. The composite material containing 10 vol.% of SAFFIL fibres was characterized by the maximum hardness of 120 HBW.

3. SEM photographs show medium bonding between alumina SAFFIL fibres and copper.

4. The wear rate decreases with increasing of Al_2O_3 . fibres content in the matrix.

Acknowledgments

Acknowledgements: The experiments were executed within the Research Project POIG.01.03.02-14-013/08-00 KomCerMet "Ceramic Metal Composites and Nanocomposites for the

Wytwarzanie oraz własności tribologiczne materiałów kompozytowych na osnowie miedzi umocnionej włóknami tlenku aluminium

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Streszczenie

Przeprowadzono obserwacje mikroskopowe, badania twardości tribologiczne materiałów kompozytowych na osnowie miedzi M1E umocnionej włóknami δ-Al₂O₃ SAFFIL. Porowate kształtki z włókien ceramicznych nasycano miedzią metodą infiltracji ciśnieniowej (squeeze casting) pod ciśnieniem 100 MPa. Przeprowadzone badania z wykorzystaniem mikroskopu skaningowego oraz optycznego wykazały równomierne rozmieszczenie włókien, jednakże z licznymi defektami i słabą zwilżalnością umocnienia przez ciekłą miedź. Zbadano twardość Brinella HBW oraz gęstość materiałów kompozytowych zawierających 10, 15 i 20 obj. % włókien Al₂O₃. Stosując urządzenie do badania zużycia typu "pin-on-disc" przeprowadzono badania odporności na zużycie ścierne w warunkach tarcia suchego i stwierdzono, że wraz ze wzrostem zawartości włókien zwiększa się twardość i odporność na zużycie ścierne. W stosunku do nieumocnionej osnowy odporność materiałów kompozytowych zawierających 20% włókien zwiększa się około 10-krotnie. W mniejszym stopniu ulega zmianie współczynnik tarcia, który przy tarciu w parze z materiałami kompozytowymi jest o około 10-20% mniejszy niż przy ścieraniu z nieumocnioną miedzią.

Slowa kluczowe: kompozyty o osnowie metalowej, włókna tlenku glinu, infiltracja ciśnieniowa, odporność na zużycie ścierne.

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