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Microstructural Study of Copper-Carbon Composite Interface

Graphite is a very attractive candidate for copper-metal matrix composites. However, copper does not wet graphite, which makes the production of copper-graphite composites difficult. Because of the strong possibilities of these combinations in forming highly desirable composites, copper-carbon composites prepared by hot uniaxial pressure were investigated. The copper-carbon interface is of the samples was examined microscopically using optical microscopy. The observed microstructure at copper interface is composed of copper-carbon interaction layer. The adhesion between copper and graphite was achieved. However, there is no intimate contact between copper and graphite film at interface. To determine the diffusion of carbon through copper matrix by X-ray diffraction was used.

Keywords: Cu–C matrix composite, Graphite composite, Wettability, Carbon diffusion

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1. Introduction

Composites are very interesting and important subset of advanced materials, because they allow a designer the ability to craft new materials with property compensations superior to those of non-reinforced metals, ceramics and polymers. Generally, a composite is considered to be any multiphase material that exhibits significant properties of both constituent phases such that better properties are achieved. The principal of combined action, an often valid supposition, states that a higher level of properties can be achieved by combining two or more dissimilar materials. Most composites are created to improve the following mechanical properties: stiffness, toughness, ambient and high temperature strength [1]. The ability to combine properties is especially crucial for aerospace, underwater, and transportation applications [2]. Metal matrix Composites (MMC) is a combination of matrix and reinforcement: matrix, is a ductile metal, usually super alloys or alloys of aluminum, magnesium, titanium, and copper. The reinforcements include particulates, continuous and discontinuous fibers, and whiskers. Some of the continuous fibers are carbon, silicon carbide, boron, alumina, and refractory metals [3]. These composites materials are usually used at elevated temperatures because they are often more resistant to creep, and they exhibit lower thermal conductivity than their bulk counterparts. Graphite copper composites have tailorable properties, are useful to high temperatures in air, and provide excellent mechanical characteristics, as well as high electrical and thermal conductivity [4]. They offer easier processing as compared with steel [5]. Although the various methods for joining graphite to other metals such as mechanical fastening, arc welding, or diffusion bonding have been examined, it was shown that it is difficult to produce a sound joint without leakage [6,7].

Previous studies were achieved to investigate the interfacial interaction for MMC and its interfacial structures [8-10]. Essentially, it is impossible to braze graphite with copper filler metal because no wetting occurs [6,11].

2. Experimental Work

Materials used in this research are: copper (99.95% purity) and graphite as base materials (graphite sheets were excluded due to fracture during the hot uniaxial pressing operation) to withstand the applied pressure [12,13]. Base materials in this research were copper and graphite and examined using X-Ray Diffraction, before and after the hot uniaxial pressing operation (see Fig. (1)).

Graphite samples were cut into squares with dimensions, 14x14x5mm$^3$ using a hydraulic cutting machine. One surface of the samples was wet ground using silicon carbide papers, polished with 1μm diamond paste, cleaned by alcohol, and finally cleaned ultrasonically for 10min. using acetone as a medium. Copper sheets interlayer were flattened by anvil, then subjected to pickling (10% HNO$_3$ and distilled water) to remove oxide film, cleaned with soap and water rinsed and finally ultrasonically cleaned for 10min. using acetone bath and then dried, etching solution for copper sheets was 50ml distilled water, 150ml hydrochloric acid (specific gravity 1.19 and 36% conc.) and 25g chromium (Vl) oxide for 20s. Prior to cleaning, each sample was then subjected to hot uniaxial pressing experiments.
Copper is highly susceptible to oxidation and oxide skin is very tenacious even at high temperatures, even though in the presence of argon as inert atmosphere. To minimize the oxidation problem, a special vacuum system of $(0.15-0.18 \times 10^{-2})$ N/m$^2$ was used. Most of copper oxidation problems associated with the heating experiments was eliminated.

In this study different heating temperature were used ranging from $(700-850)^\circ$C, holding time was $(30-120)$ min, and pressing pressure $(7.5-20 \times 10^6)$ N/m$^2$. Microstructural tests for
polished side of graphite were used using optical microscope, as shown in Fig. (2) and Fig. (3).

Fig. (2) Microstructure of graphite before attempting the hot uniaxial pressing

Fig. (3) Microstructure of copper before attempting the hot uniaxial pressing

3. Fractography and X-Ray Diffraction Analysis

After attempting the hot uniaxial pressing experiments for copper-graphite composites, fractography and XRD Analysis were used to examined the interfacial structure at the composites interface, the different features appeared could be classified as follows:

1- Diffusion of carbon at copper interface was observed and this was a function of changing heating temperatures (700-850)°C, were the diffusion is increased with increasing heating temperature. Figure (1c) shows Diffractograms from copper interface, indicating the effect of increasing heating temperature on diffusion of carbon through copper matrix. Figure (4) shows parallel lines of copper surface imprint at graphite interface and this was due to coarsening of graphite surface to increase the contact area. This procedure does not affect the newly formed contact region. Increase in heating time (30-120min), increases the recrystallization of copper (fine grains) but on the other hand does not affect the diffusion of carbon through interface markedly and this is clearly observed in Fig. (5).

2- Increasing the pressing load (7.5-20MPa) does not affect the diffusion of carbon through copper matrix markedly, and this was due to induced cracks during the pressing operation and this inhibit diffusion as well [14]. Figure (6) is a fractograph of copper, the dark area on the top of the micrograph represent the graphite adhered material, and the white area at the bottom of the micrograph represent copper contact interface, there is no diffusion of carbon in this region.

3- The inert atmosphere had a significant effect on the reliability of the hot- pressing operation. The high oxidation of copper had a tailorable effect at high temperature and this
induces an interfacial corrosion at interface, this inhibits the diffusion of carbon through copper matrix. As a result the oxide film became a barrier to form contact or bond, as shown in Fig. (7). Vacuum atmosphere (0.15-0.18x10^{-2}) N/m^2 is more reliable than argon atmosphere to achieve adhesion, and this agrees well with previous study of joining copper to ceramic [15].

4- The adhesion between copper ad graphite was achieved, and this was due to mechanical or interlocking theory. However, there is no intimate contact between the copper and graphite film [16], as shown in Fig. (8) and Fig. (9).

5- The increase in heating temperature enlarges the contact area during the hot uniaxial pressure experiments at interface and this is clearly observed in Fig. (10), (large dark area, is graphite bonded to copper interface), even though, the failure was observed during cooling to room temperature at graphite bulk material near joint interface for adhesive joint. On the other hand, failure occurs at copper-graphite interface as shown in Fig. (11), (grey regions represent graphite films bonded to copper and black spots represent graphite partitions) were weak boundary layered is formed, this is in reasonable agreement with the result reported [11,17].

4. Conclusions
The experiments with the copper-graphite composite showed a dramatically different behavior. Hot uniaxial pressing parameters had a noticeable effect on diffusion of carbon and the resultant adhesion of graphite through copper matrix at the interfacial region. Obviously carbon does not wet copper, so the proposed mechanism for producing copper-carbon composite in solid state is, mechanical interlocking. The main effective parameter in improving composite reliability is, temperature. The diffusion of carbon at copper interfaces increased with increasing heating temperature and has it
maximum values at 850°C. Heating time has no considerable effect on diffusion through interfacial interface.

Increasing the pressing load during the hot uniaxial pressing operation induce cracks through graphite and inhibit diffusion of carbon through copper matrix. The effect of inert atmosphere appeared to be very effective at elevated temperature and implies that vacuum atmosphere (0.15-0.18x10⁻²) N/m² is more reliable than argon atmosphere for overcoming the problem of copper corrosion at high temperature. The adhesion between copper and graphite is achieved. However, there is no intimate contact between the copper and graphite film at interface. For all specimens the failure was observed through cooling to ambient temperature. This failure has two features. Firstly one at graphite bulk material near joint interface for adhesive joint, secondly at copper- graphite interface, where weak boundary layered is formed.

References

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