Copper-Carbon and Aluminum-Carbon Composites 
Fabricated by Powder Metallurgy Processes

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Abstract. The increase in both power and packing densities in power electronic devices has led to an increase in the market demand for effective heat-dissipating materials, with high thermal conductivity and thermal expansion coefficient compatible with chip materials still ensuring the reliability of the power modules. In this context, metal matrix composites: carbon fibers and diamond-reinforced copper and aluminum matrix composites among them are considered very promising as a next generation of thermal-management materials in power electronic packages. These composites exhibit enhanced thermal properties compared to pure copper combined with lower density. This article presents the fabrication techniques of copper/carbon fibers and copper/diamond and aluminum/carbon fibers composite films by powder metallurgy and hot pressing. The thermal analyses clearly indicate that interfacial treatments are required in these composites to achieve high thermomechanical properties. Interfaces (through novel chemical and processing methods), when selected carefully and processed properly will form the right chemical/mechanical link between metal and carbon, enhancing all the desired thermal properties while minimizing the deleterious effect.

1. Introduction
Electronic packaging refers to the protective features and safety devices that enable continued and efficient operation of electronic components, and does not refer to shipping containers. Electronic components typically refer to assemblage of resistors, capacitors, transistors, diodes, etc., in a complex well designed group as integrated circuits usually soldered to a printed circuit board. Since these components are very sensitive in all respects, the packaging is designed to prevent damage, avoid exposure to dirt and other adverse environment, dissipate heat very efficiently, enhance reliable service life and sustain efficient working conditions [1-3].

Composite materials show the highest potential for application in heat sinks for electronic applications. They can combine metal, polymer and ceramics in a broad range of different products and can be tailored to the final desired properties. It is critically important to note that final properties of composites depend not only on the basic constituent materials but also on the methods of manufacture. Among the large number of different composites, the most efficient and the most commonly developed for heat dissipation purposes are metal matrix composites (MMCs). More specifically MMCs based on copper and aluminum matrices with a broad range of different reinforcements show great promise. Undoubtedly, the most efficient composites for heat sinks are those based on Cu and Al with a number of different reinforcements [4-9]. In general, MMCs show several improvements in comparison with currently used materials in electronic packaging, including:
Lower and tailorable CTE (the higher the volume fraction of reinforcement used, the lower the CTE),
Higher heat dissipation capability,
Light-weight, suitable for space applications,
Higher stiffness at elevated temperatures (for dimensional stabilities at elevated temperatures).

In this paper, we mainly focus on understanding the thermal management requirements of electronic packaging, the key scientific and technological issues, advances in terms of materials, design and processing, and future prospects of the field. In specific, we will address how the competing physical and thermal requirements are optimized in heat sinks. Many of the issues addressed can be equally applicable to the design of any component with similar thermal and mechanical requirements including that of heat exchanger tubes. Though in this paper, we focus our attention on copper-carbon and aluminum-carbon system, concepts presented here can be easily extended to other material systems.

2. Experimental procedure: materials and processing

2.1 Copper matrix
Many techniques are used to manufacture powders. In the case of copper, generally either chemical or atomization methods are employed. Each technique plays a major part on final material properties, microstructure (grain size, distribution, morphology, and particle content), chemistry and finally, the cost of the powders. Chemical methods can be used to prepare metallic powders; the difference between each is linked to the initial components and the chemical reaction. It is possible to use oxides, sulphates or carbonyl compounds which are reduced, precipitated or decomposed in order to obtain copper powders. Among these chemical methods, electrolysis which is a method using an electric current to drive the metallic ion reduction is promising [10].

2.2 Heat sink fabrication
MMC fabrication processes are numerous; therefore, we will just discuss the process relevant to heat sink fabrication. It is necessary to sinter materials with a high density (porosity < 4%) in order to obtain high thermal conductivity. Indeed, the gas (air, argon…) in the porosities is a thermal insulator and can strongly hinder the thermal conductivity of the material. Fabrication processes of heat sinks can be classified in two groups: liquid route and solid route. The solid route process is a powder metallurgy process, i.e., the powder of metal matrix is mixed to the particle or short fiber reinforcement. This mix is then sintered in solid state. There are various powder metallurgy processes that can be distinguished by the method of use to produce the stress. The conventional powder metallurgy process consists in 1) shaping at ambient temperature and 2) strengthening the powder using at high temperature near the melting point. This last step is called sintering. This process does not allow obtaining composites with high density, which is why a pressure is usually applied during the sintering, referred to as hot sintering processes [11]. In the uniaxial hot pressing, a uniaxial pressure is applied at a temperature close to the melting point, which increases the density of the final composite (porosity < 5 vol.%). This technique allows producing composite materials with a high volume fraction of reinforcement (up to 50 vol. %). Nevertheless, the mechanical resistance of the tool limits the maximum stress that can be applied. This process is widely used to fabricate MMC with aluminum or copper matrices because these metals are ductile even at low temperature.

3. Results and discussions

3.1 Copper/Carbon fibers composites

3.1.1 Process
Copper and carbon fibers pitch type, named K6371T and K223HG, usually form the starting powders (Table 1). Typical Scanning Electron Microscopy (SEM) observations show that copper particles are present with dendrite shapes with sizes ranging from 25 to 30 µm. Chopped carbon fibers have diameters ranging from 9 to 10 µm and lengths between 100 to 300 µm. In the first step, copper
powders and carbon fibers are mixed together. The mixed powder is then compacted in graphitic mould, which is heated by an induction system. The samples are pressed at 650 °C under 50 MPa during 20 minutes; the heating rate is 25 °C/min. The temperature is controlled with a thermocouple inserted inside the mould. A vacuum in the chamber of 0.66 Pa prevents the oxidation of copper powder during the heating cycle. The final density of the composite material is close to 97% and no specific degradation of the carbon fiber is observed. The optical micrographs of the samples show a strong anisotropic structure (Fig. 1), with an orientation of the carbon fiber perpendicular to the compaction direction.

![Figure 1: Optical micrographs of the composite material in (a) cross and (c) parallel section after densification and (b) schematic of the Carbon fiber orientation.](image)

This anisotropy is due to the strains and stresses imposed by the processing-induced deformations and geometry, and will be reflected in the anisotropic thermal (k) and thermo-mechanical (CTE) properties of the composite.

### 3.1.2 Thermal properties

Thermal conductivity (k) and coefficient of thermal expansion (CTE) have been measured using a flash laser method and a horizontal dilatometer respectively. Due to the anisotropic structure of the composite, these properties are determined in two perpendicular directions namely parallel to the pressing direction ($k_{//}$ and $\text{CTE}_{//}$) and perpendicular to the pressing direction ($k_{\perp}$ and $\text{CTE}_{\perp}$). The CTE$_{//}$ of the composite are close to $17 \times 10^{-6} \text{K}^{-1}$, whatever the type and the volume fraction of carbon fibers used. In contrast, the CTE$_{\perp}$ decreases when the carbon fibers volume fraction increases whatever the fiber type used (Table 2). These anisotropic properties are due to the strong anisotropy of carbon fiber properties (Table 1) and their orientation perpendicular to the pressing direction in the composite.

| Material   | Density | $k$ (W.m$^{-1}$K$^{-1}$) | CTE ($10^{-6}$ K$^{-1}$) |
|------------|---------|--------------------------|--------------------------|
| Matrices   | Cu      | 8.96                     | 400                      | 17                       |
| K223HG     | 2.2     | $540 / 10$               | -1 / 12                  |
| XN100      | 2.2     | $900 / 10$               | -1 / 12                  |
| Diamond    | 3.5     | $800 - 2000$             | 1                        |

Same anisotropy can be observed for the thermal conductivity measured on the various composites. Table 2 shows the evolution of $k_{//}$ and $k_{\perp}$ for two types of carbon fibers (CF) with a volume fraction ranging from 30 to 40 vol. %. This table shows that $k_{//}$ decreases when the carbon fiber volume fraction increases; $k_{//}$ is just link to the CF volume fraction but not to the fiber type. However, the variation of $k_{\perp}$ with the volume fraction is different; $k_{\perp}$ is obviously link to the carbon fiber volume fraction but also to their intrinsic thermal conductivity along their axis. Indeed, when the fibers used have a thermal conductivity along their axis greater than that of copper, $k_{\perp}$ increase with the volume fraction (example of K223HG carbon fiber type). On the other hand, when the carbon fibers used have a thermal conductivity along their axis lower than that of copper, $k_{\perp}$ decrease with the volume fraction (example of K6371T carbon fiber type).
Table 2: Thermal conductivity parallel (//) and perpendicular (⊥) to the pressing direction and CTE perpendicular (⊥) to the pressing direction for copper/carbon fibers composites.

| Cu/CF   | CF vol. % | Density | $K$ (W.m$^{-1}$.K$^{-1}$) | CTE ($10^{-6}$ K$^{-1}$) |
|---------|-----------|---------|----------------------------|---------------------------|
|         |           |         |                            |                           |
| Cu / K6371T | 30        | 6.8     | 300 ⊥, 180 //              | 14                        |
|         | 35        | 6.5     | 330 ⊥, 160 //              | 12                        |
|         | 40        | 6.2     | 360 ⊥, 140 //              | 10                        |
| Cu / K223HG | 30        | 6.8     | 240 ⊥, 180 //              | 14                        |
|         | 35        | 6.5     | 210 ⊥, 160 //              | 12                        |
|         | 40        | 6.2     | 180 ⊥, 140 //              | 10                        |

3.2 Copper/Diamond composites

3.2.1 Material and process

Diamond has exceptionally high thermal properties with the highest thermal conductivity at room temperature of all known materials ($\lambda = [800 - 2000]$ W.m$^{-1}$.K$^{-1}$). However, its very low CTE ($\alpha = 1.0 \times 10^{-6} \degree$C$^{-1}$) remains a problem. Thus, it is interesting to consider the use of diamond as reinforcements embedded in a thermal package substrate, such as a copper matrix (Cu). In this study, Cu/D composite films were fabricated by tape casting and hot pressing. Diamond powders from Henan Zhongxin Co., China, and dendritic copper powder from Ecka Poudmet, Germany, were used to process the Cu/D composites. After tape casting, the Cu/D composite tapes are first heat treated at 400°C under air during 2 hours (debinding treatment), then heat treated 60 minutes at 400°C under Ar/H$_2$ (reducing treatment). Finally, after stacking of 10 to 15 tapes, the Cu/D composites were hot pressed during 20 minutes at 650°C under 50 MPa to obtain dense materials.

3.2.2 Copper particles deposition onto diamond powders

Cu/D composites fabricated by powder metallurgy have shown weak interfacial bonding because pure liquid copper does not wet diamond particles. The non-reactive Cu/D interface is critically detrimental to the thermal properties of the Cu/D composites. Carbide forming materials, namely chromium or boron, are usually employed to bond diamond reinforcements to the copper matrix, either by being alloyed to the matrix [12, 13] or by being directly coated onto diamonds [14]. However, the carbide interphase also acts as a thermal barrier and affects the heat transfer between electronic conduction in copper and phonon conduction in diamond. We used here an innovative process that consists in depositing copper nanoparticles onto diamond reinforcements through C-O-P-O-Cu and/or C-P-O-Cu bonds prior to sintering. This new process creates strong chemical bonding between the diamonds and the copper matrix [15]. This process leads to dense and cohesive Cu/D composites without using carbide forming elements, thereby allowing high thermal performances with minimum diamond content making these Cu/D composites highly cost-effective.

Figure 2 shows diamond particle covered by Cu submicronic particles. One can see that the coverage of the diamonds surface by the copper particles is remarkably uniform. In addition, the size of the surface Cu particles can be adjusted depending on the reduction time and temperature, as shown on figure 8a and Figure 2b with Cu particle sizes ranging from 400 nm to 1 µm (after 60 minutes at 400°C under Ar/H$_2$), on Figure 2c with Cu particles sizes ranging from 300 to 800 nm (after 1 minute at 400°C under Ar/H$_2$) and Figure 2d with Cu particle sizes ranging from 100 nm to 400 nm (after 1 minute at 350°C under Ar/H$_2$). However, the influence of the size of the Cu nanoparticles coating on the final thermal properties of the composites is not yet clear. Regarding the chemical process responsible for the Cu particles deposition, XPS analysis has demonstrated that the mixing of the dispersant agent (phosphate ester) with the powders during formulation leads to the creation of phosphorus-functionalized nucleation sites on the diamond powders surface. During debinding, copper powders are oxidized and wire-shaped CuO nanostructures are observed to grow on the copper dendrites. Such CuO nanowires growth process has been reported in previous studies [25]. During the reduction treatment, copper sublimed from CuO nanowires and settles again onto the chemically active sites on the diamond particle surface through C-O-P-O-Cu and/or C-P-O-Cu chemical bridges.
During the hot pressing step, Cu-Cu metallic bonds are formed between the Cu nanoparticles onto the diamonds and the copper particles that will form the Cu matrix after densification. The Cu nanoparticles thus enable the formation of a cohesive interface between the copper matrix and the diamond reinforcements through strong chemical bonding without requiring any carbide forming materials.

![Figure 2: SEM micrographs of diamond reinforcements coated with copper particles: (a,b) after 60 minutes under Ar/H$_2$ at 400°C (c) after 1 minute under Ar/H$_2$ at 400°C (d) after 1 minute under Ar/H$_2$ at 350°C](image)

3.2.3 Thermal properties

As shown on Figure 3, the average thermal conductivity of multilayer composites materials, fabricated by tape casting process, increases from 320 to 460 W.m$^{-1}$.K$^{-1}$ with respect to diamond volume fractions ranging from 0 to 40 %. This confirms that the copper particles deposited onto the diamond reinforcements prior to sintering effectively act as bonding agents between diamond and the copper matrix, enabling efficient interfacial heat transfer between electronic and phonon conductions. However, we observe a drop of thermal conductivity to 313 W.m$^{-1}$.K$^{-1}$ with a diamond volume fraction of 50 % due to the low compaction of the Cu$_{50}$D$_{50}$ composite, which is detrimental to the thermal conductivity. This decrease in thermal conductivity means that the percolation threshold (i.e. the reinforcement volume fraction at which a continuous path between reinforcements exists in the composite) has been reached.

![Figure 3: Experimental thermal conductivity of the diamond reinforced copper matrix composites as a function of diamond volume fraction](image)
The percolation threshold occurs between 40 and 50% in diamond volume fraction. We reach a maximum thermal conductivity of 460 W.m\(^{-1}\).K\(^{-1}\) with a diamond volume fraction of 40%, which is a significant improvement compared to pure copper (\(\lambda_{\text{Cu}} = 400 \text{ W.m}^{-1}.\text{K}^{-1}\)). We expect to improve this result in our ongoing effort using single tape systems. As shown on Figure 4, the averages measured CTEs of composites decrease linearly from 18.9 \times 10^{-6} °C\(^{-1}\) to 11.8 \times 10^{-6} °C\(^{-1}\) with diamond volume fractions ranging from 0 to 50%, which is a strong enhancement relative to the CTE of pure copper (CTE\(_{\text{Cu}} = 17 \times 10^{-6} \text{ °C}^{-1}\)). These results demonstrate that the copper deposition process leads to a strong interfacial bonding enabling an efficient thermal expansion load transfer at the matrix/reinforcement interface.

### 3.3 Aluminum based composite

#### 3.3.1 Materials and fabrication process

Spherical aluminum powder (F3731, Poudres Hermillon) with 0.88J/K of heat capacity and discontinuous pitch-based carbon fiber (Raheama® R-A301, Teijin Limited.) with 8mm of average diameter, 200mm of average length, 0.70J/K of heat capacity and 600 W/m.K of thermal conductivity in longitudinal fiber direction, were employed. AlSi11.3at% alloy powder (F2071, Poudres Hermillon) was also prepared. And differential scanning calorimeter (DSC) revealed the melting point of this Al-Si alloy was 584.6°C. Discontinuous pitch-based carbon fibers was mixed with aluminum matrix powder, which was added 5vol% of Al-Si alloy powder, for 1 hour in air. Then, volume fractions of carbon fibers were controlled in 10, 20, 30, 40 or 50vol%. Mixed composite powder was consolidated by hot pressing with 60 MPa of uniaxial pressure, for 30 minutes of holding time at 600°C (i.e. between the melting points of aluminum and Al-Si alloy) of holding temperature. Temperature was monitored by a K-type thermocouple positioned in the hole spaced at the centre of carbon mold.

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![Figure 4: Experimental CTEs of the diamond reinforced copper matrix composites as a function of diamond volume fraction](image)

#### 3.3.2 Results

And as shown in Figure 5, carbon fibers tended to be aligned in plane due to uniaxial pressure in hot press process, while this alignment was less than perfect due to the high aspect ratio of carbon fiber.
Figure 5: SEM micrographs of Al-CF50vol% composite with 5vol% of Al-Si alloy to (a) parallel and (b) vertical directions of applied pressure axis.

Figure 6(a) shows the thermal conductivity of Al-CF composite to transverse direction of carbon fibers. Thermal conductivities were decreased with carbon fiber addition, regardless of Al-Si alloy addition. To in-plane direction of carbon fiber, thermal conductivity of Al-CF composite, which was added 5vol% of Al-Si alloy in aluminum matrix, were increased with carbon fiber addition, and achieved to 258W/m.K in Al-CF50vol% composite as shown in Figure 6(b), although that was decreased without Al-Si alloy. Figure 7 shows the CTE of Al-CF composite to in-plane direction of carbon fibers. CTE of aluminum, 24.0×10^{-6}/K was slightly degraded to 25.3×10^{-6}/K whether Al-Si alloy was added or not. And CTEs of Al-CF composite, with/without 5vol% of Al-Si alloy, were improved with less than 20vol% of carbon fiber. However, CTE of Al-CF30vol% without Al-Si alloy was slightly higher than that of Al-CF20vol%, and not stable. Furthermore, CTE of Al-CF composite without Al-Si alloy was immeasurable with more than 40vol% of carbon fiber due to the deterioration of the composite attributed to low relative density. On the other hand, CTE of Al-CF composite with 5vol% of Al-Si alloy was further improved and achieved to 7.0×10^{-6}/K in Al-CF50vol% composite.
Figure 7: CTE of Al-CF composite to in-plane direction of carbon fibers.

Figure 8(a) shows TEM micrographs of Al/CF interface in Al-CF50vol% composite. It was observed needle like aluminum carbide (Al₄C₃) crystals, and these crystals were oriented to same direction. And the fringe indicated the strain in aluminum matrix. Figure 8(b) shows TEM micrographs of tip of carbon fiber in Al-CF50vol% composite. Al₄C₃ crystals were observed as clusters, carbon fiber tip was covered with Al₄C₃.

3.3.3 Discussion

In conventional hot pressing at 600°C (i.e. between the melting points of Al (660°C) and Al-Si (584.6°C)), Al-Si alloy changes to liquid phase. And this liquid phase of Al-Si alloy infiltrates into slight spaces such as aluminum particle boundaries, between carbon fibers and Al/CF interface. Consequently few quantity of Al-Si alloy allows to fabricate fully-dense composite without particular or complicate process, although thermal conductivity and CTE of aluminum are slightly decreased due to 5vol% of Al-Si alloy addition. Not only the pitch-based carbon fiber which we have employed, but also other several pitch-based carbon fibers, which have excellent thermal conductivity to longitudinal direction have low thermal conductivity to transverse direction. Therefore, thermal conductivity of Al-CF composite is decreased by carbon fiber addition, to transverse direction of carbon fiber. On the other hand, the thermal conductivity of Al-CF composite is improved to in-plane direction of carbon fibers although that is decreased in Al-CF composite, which is fabricated without Al-Si alloy, due to low relative density. Thermal conductivity of Al-CF50vol%, 258W/m.K is higher than the value of pure aluminum, 240W/m.K.
The assemblies for computer are generally constructed by silicon (4.1×10⁻⁶/K) or alumina (6.7×10⁻⁶/K). To avoid the fracture by thermal stress, the heat sink material also should have a similar CTE to them, around 4.0–7.0×10⁻⁶/K to parallel direction to assembly/heat sink contact surface. Here, it seems that our Al-CF50vol% composite with 7.0×10⁻⁶/K of CTE is suitable as a heat sink material. As shown in Figure 7, it is revealed CTEs of our Al-CF composites tend to be amenable with the values estimated by Schneider’s equation while it should be taken account this equation is for continuous fiber reinforced composite. Here, it required a strong interfacial link to bring out remarkable CTE of carbon fiber in aluminum matrix because it is assumed that matrix/fiber interface is perfectly linked. It has been reported interfacial Al₄C₃ is valid for effective load transfer due to strong chemical bond at Al/Carbon interface. In our Al-CF composite, it seems Al/CF interface is not delaminated due to reinforce by interfacial Al₄C₃ formation. Indeed, the thermal strain around Al/CF interface is shown as the fringe in TEM micrograph (see Figure 8). Therefore, it seems aluminum carbide at Al/CF interface (i.e. Al-C chemical bond) contributes to effective CTE improvement.

4. Conclusions

In the case of copper/carbon fibers composites, the orientation of the carbon fibers in the matrix induces anisotropic properties of the composite. Due to the processing method, we have measured the thermal conductivity parallel to the loading direction, which shows lower thermal conductivity of the composite. The coefficient of thermal expansion (CTE) has been measured perpendicular to the loading direction. The optimized interfaces obtained with this method give a higher thermal conductivity of the final composites compare to the Cu-X/D₃₀. Indeed, this technique allows to create a chemical link between the copper and the diamond through C-O-P-O-Cu and/or C-P-O-Cu bridges which have a thermal conductivity nearer to copper than carbides. The thermal conductivities of composites prepared with an interfacial treatment, Cu/D₃₀-Cu dots are greater than pure copper (400 W.m⁻¹.K⁻¹) and twice that of the Cu/D₃₀ reference composite.

Al-CF composite was fabricated by conventional powder metallurgy process, hot press with 5vol% of Al-Si alloy between the melting points of Al and Al-Si alloy. Liquid phase of Al-Si alloy allows to fabricate fully-dense composite without particular or complicate process even if thermal conductivity and CTE of aluminum are slightly decreased due to 5vol% of Al-Si alloy addition. With 50vol% of carbon fiber, thermal conductivity and CTE of Al-CF composite were achieved to 258W/m.K and 7.0×10⁻⁶/K to in-plane direction of carbon fiber. 258W/m.K is slightly higher than pure aluminum (240W/m.K), thermal conductivity was improved by carbon fiber addition. And 7.0×10⁻⁶/K of CTE satisfies the value as the heat sink materials for the computer assemblies, around 4.0–7.0×10⁻⁶/K to parallel direction to assembly/heat sink contact surface. Then, it seems interfacial Al₄C₃ formation, i.e. Al-C chemical link can effectively contribute to heat transfer and Al/CF interface strength.

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