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Thermal conductivity improvement of copper–carbon fiber composite by addition of an insulator: calcium hydroxide

S. Couillaud · Y. F. Lu · J.-F. Silvain

Abstract The effects of adding calcium hydroxide ($\text{Ca}(\text{OH})_2$) to a copper–CF (30 %) composite (Cu–CF(30 %)) were studied. After sintering at 700 °C, precipitates of calcium oxide (CaO) were included in the copper matrix. When less than 10 % of $\text{Ca}(\text{OH})_2$ was added, the thermal conductivity was similar to or higher than the reference composite Cu–CF(30 %). A thermal conductivity of $322 \text{ W m}^{-1} \text{ K}^{-1}$ was measured for the Cu– $\text{Ca}(\text{OH})_2$ (3 %)-CF(30 %) composite. The effects of heat treatment (400, 600, and 1000 °C during 24 h) on the composite Cu– $\text{Ca}(\text{OH})_2$ (3 %)-CF(30 %) were studied. At the lower annealing temperature, CaO inside the matrix migrated to the interface of the copper matrix and the CF. At 1000 °C, the formation of the interphase calcium carbide (CaC_2) at the interface of the copper and CFs was highlighted by TEM observations. Carbide formation at the interface led to a decrease in both thermal conductivity (around $270 \text{ W m}^{-1} \text{ K}^{-1}$) and the coefficient of thermal expansion (CTE ($10.1 \times 10^{-6} \text{ K}^{-1}$)).

Introduction

Power density growth in integrated circuits becomes faster every day. Thus, heat dissipation is critical; consequently,

the cooling efficiency of the substrate must be improved. Heat-spreading materials for ICs must have both high thermal conductivity (TC) and low coefficient of thermal expansion (CTE) [1]. The high TC prevents electronic components from overheating while the low CTE prevents mechanical failure after the heat cycle, between the ceramic substrate (with low CTE) and the heat sink [2].

Dissipating heat is one of the major problems occurring inside of power electronic devices. During the last 50 years, several studies have been conducted on this subject. Copper and aluminum are the elements most often studied because of their good TC (i.e., 400 and $230 \text{ W m}^{-1} \text{ K}^{-1}$, respectively). However, their CTE is too high (i.e., 17 and $23 \times 10^{-6} \text{ K}^{-1}$, respectively) compared to the other elements used in power electronic devices. In order to maintain the high TC of the elements and decrease the CTE, the formation of composites reinforced with silicon carbide (SiC), CF, carbon nanotubes, and diamond was proposed [3–7]. For example, using CF with copper can provide an inexpensive material that is easily machinable with good TC (i.e., around $300 \text{ W m}^{-1} \text{ K}^{-1}$ with 30 vol.% of CF) and a lower CTE (i.e., $12 \times 10^{-6} \text{ K}^{-1}$ with 30 vol.% of CF).

Moreover, the properties of a composite are linked to the properties of the matrix, the reinforcement, and the interphase. Consequently, improving the interphase is an important challenge. In a copper–carbon fiber (Cu–CF) composite, the copper and carbon are totally inert so the interface is only mechanical (i.e., connection with the porosity and the defects and the fretting induced by the difference between the CTE of the matrix and the reinforcement). After several thermal cycles, the interphase is degraded, leading to a decrease in the TC and an increase in the CTE.

In order to improve the interface between copper and carbon, the use of carbide-former elements was proposed

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[6–10]. Carbide formation induces a chemical bond between a matrix and reinforcements leading to an improvement in the properties. For example, Schubert et al. [6] have shown an improvement by a factor of two in the TC of a composite of chromium copper alloy 0.8 (CuCr_{0.8}) and diamond. Numerous carbide-former elements, such as boron, chromium, titanium, aluminum, and zirconium, have been studied. Three important points highlight the efficiency of a carbide-former element. First, the standard enthalpy of the carbide formation must be negative and around -60 to -20 kJ mol⁻¹. Second, the carbide-former elements must have a low solubility with the matrix. Third, the carbide layers formed during the sintering must be controlled and kept thin in order to maintain a maximum thermal interface conductance.

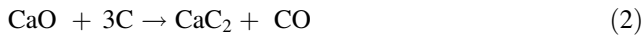
Based on these criteria, calcium, with an enthalpy formation of -62 kJ mol⁻¹, appears to be a good candidate. In this paper, the effects of adding calcium into the Cu–CF(30 vol.%) composite were studied. Because calcium is not stable under air (i.e., the rapid formation of calcium hydroxide), two solutions are proposed:

- (i) Using commercial Ca(OH)₂.

According to the literature, the formation of calcium carbide (CaC₂) from calcium hydroxide Ca(OH)₂ is possible. Ca(OH)₂ is decomposed into calcium oxide (CaO), above 550 °C, according to Reaction (1) [11]



Then, CaO reacts with carbon to form CaC₂, according to Reaction (2). This reaction starts around 1000 °C [12].



- (ii) Using a copper–calcium (CuCa) binary compound.

In this way, the calcium stays metallic and can be formed more easily (CaC₂). Moreover, the low melting point of a CuCa compound (i.e., 567 °C) could induce a semisolid sintering improving the diffusion of Ca and reducing the porosity [13].

In this paper, how adding Ca(OH)₂ affects the TC and CTE of a Cu–CF(30 vol.%) composite is discussed. Microstructure analyses were conducted in order to explain the effects on the TC values of adding calcium (Ca). The effects of adding CuCa are not discussed in this paper.

Experimental details

Dendritic copper (Eckart Granules Poudmet, 30 μm), CFs ($l \geq 100$ μm, $\Phi = 5$ μm), and calcium hydroxide powder (Aldrich, 1 μm) were used in the preparation of the Cu–

Ca(OH)₂(*X* vol.%)–CF(30 vol.%) composites. The CFs are PITCH fibers which exhibit a longitudinal TC of 600 W m⁻¹ K⁻¹ and a perpendicular TC of 10–15 W m⁻¹ K⁻¹.

Copper, CF, and Ca(OH)₂ were then mixed in the appropriate amounts using a 3D Turbula for 2 h. Then the mixture was hot pressed in a steel mold ($\Phi = 40$ mm) at 700 °C/80 MPa for 30 min. The pellets were then trepanned in order to obtain pellets 6 mm in diameter, with the CFs oriented. The orientation of the fibers is due to the pressure applied during sintering. CFs are randomly dispersed in a plane, perpendicular to the axis pressure direction. In this study, the pressure direction was the reference for the orientation of the fibers.

The densities of the composites were measured using both the Archimedes and geometric methods and compared to the theoretical densities calculated using the rule of mixture:

$$\rho_c = \rho_m V_m + \rho_{CF} V_{CF} + \rho_x V_x$$

where, ρ_m , ρ_{CF} , and ρ_x are the densities of the copper matrix, the CFs, and the calcium compounds, respectively, given in g cm⁻³. V_m , V_{CF} , and V_x are the volume fractions of the copper matrix, the CFs, and the calcium compounds, respectively.

All samples were characterized by X-ray powder diffraction using a Philips PW 1050 diffractometer with CuK α radiation ($\lambda = 0.15405$ nm). These patterns were scanned by steps of 0.02° (2θ) from 5° to 80° with a constant counting time of 30 s.

The bulk samples were investigated by electron probe microanalyses (EPMA) with metallic Ca (K α), Cu (K α), and C (K α) as standards.

Scanning electron microscopy (SEM) micrographs were acquired using a Tescan VEGA II-SBH working at 10 and 20 kV with a current beam of 90 μA.

Transmission electron microscopy (TEM) micrographs were done using a Joel 2100 microscope working at 200 kV and equipped with energy-dispersive X-ray spectroscopy (EDS).

The thermal diffusivity D (m² s⁻¹) was measured using a Flash laser technique (Netzsch LFA 457) on 6-mm diameter samples with a thickness between 2 and 5 mm. Thermal diffusivity was measured at 70 °C for reasons of measurement stability. Each measurement was performed at least 30 times, and an average value was calculated.

The CTE was measured with a differential dilatometer (Netzsch DIL 402 C). The heating rate was 2 °C/min and ranged from room temperature to 250 °C under argon atmosphere. The CTE value is an average of at least six cycles measured where only the linear part was used (i.e., between 80 and 200 °C).

Results and discussion

Composite Cu–Ca(OH)₂ (X %)–CF(30 %) after sintering

Numerous composites with the composition Cu–Ca(OH)₂(X %)-CF(30 %) were fabricated where X = 0, 0.5, 1, 3, 5, 10, and 30 %.

The relative density ($d_{\text{exp}}/d_{\text{theoric}}$) of each composite is given in Table 1. For each sample, the sintering conditions led to the required density, above 98 %. However, when the content of Ca(OH)₂ increased, the density increased, up to 106 %. These too-high values can be explained by the transformation of Ca(OH)₂ in CaO (Reaction 1) that can occur during the sintering. Indeed, if the density of CaO (i.e., 3300 kg m⁻³) was used instead of Ca(OH)₂ (i.e., 2240 kg m⁻³), the relative density would decrease at 100 % (Table 1, Lines 2 and 4). Thus, the fact that Reaction 1 can occur during sintering at 700 °C was considered and was in agreement with the literature [11].

In Fig. 1, the X-ray patterns are given for X = 0, 10, and 30 % vol. The X-ray pattern of the sample Cu–Ca(OH)₂(30 %)-CF(30 %) exhibited some new peaks, which can be indexed by CaO. Therefore, the transformation of Ca(OH)₂ into CaO (Reaction 1), during the process, was confirmed. Moreover, for each composition, the peaks of copper were fixed. There was no variation in the lattice parameters. Consequently, the calcium did not make a solid solution with the copper, which agreed with the binary diagram.

Table 1 Archimedes and geometric relative densities of samples Cu–Ca(OH)₂(X %)-CF(30 %) (W = 0, 0.5, 1, 3, 5, 10, 30) considering the density of Ca(OH)₂ (gray) and the density of CaO (white)

Ca(OH) ₂ content	0	0.5	1	3	5	10	30
Geometric density (%) considering Ca(OH) ₂ density	98.9	98.3	99.4	101	101	102.7	106.8
Geometric density (%) considering CaO density	98.9	98.2	99.3	100.6	100.4	101.5	102.6
Archimedes density (%) considering Ca(OH) ₂ density	100.4	99.8	100.6	100.2	101.4	101.6	105.4
Archimedes density (%) considering CaO density	100.4	99.8	100.5	99.8	100.8	100.5	101.2

According to the SEM micrographs (back scattering electrons, Fig. 2), the three elements can be easily detected. No porosity is visible at the mechanical interface between the copper and the CFs. These observations are in good agreement with the relative density measured for each sample. The Ca(OH)₂ decomposed in CaO was mainly embedded in the copper matrix. The size of the CaO precipitates was around 10–15 μm. The size of the precipitates remains the same regardless of the amount of CaO, but their number increases when this amount increases. The precipitates near the CFs seemed to interact with them (Fig. 3). However, the nature of the interactions is unknown. Indeed, according to the literature, the formation of CaC₂ from CaO seems possible above 1000 °C. Thus, at a 700 °C sintering temperature, carbide should not be formed during the process. However, higher temperatures, inside the bulk material, can be locally generated due to the decomposition of Ca(OH)₂.

The sample with 3 % of CaO was investigated by TEM analysis. The micrographs and the electron diffraction pattern are given in Fig. 4. The EDS analysis confirmed the presence of calcium (gray phase on the micrograph) at the interface between the copper and the CF. This interphase, where the calcium is located, seems granulous and composed of nanometric particles (Fig. 4b). This nanostructure is probably induced by the decomposition of Ca(OH)₂. Ghosh-Dastidar et al. [14] reported similar behaviors. They explain that, during sintering, one grain of Ca(OH)₂ was progressively decomposed in small particles of CaO from the edge to the core.

The electron diffraction pattern (Fig. 4c) can be indexed using the crystallographic planes of copper, carbon, and calcium oxide. This observation confirms the transformation of Ca(OH)₂/CaO. No carbide was found in the numerous electron diffraction patterns studied.

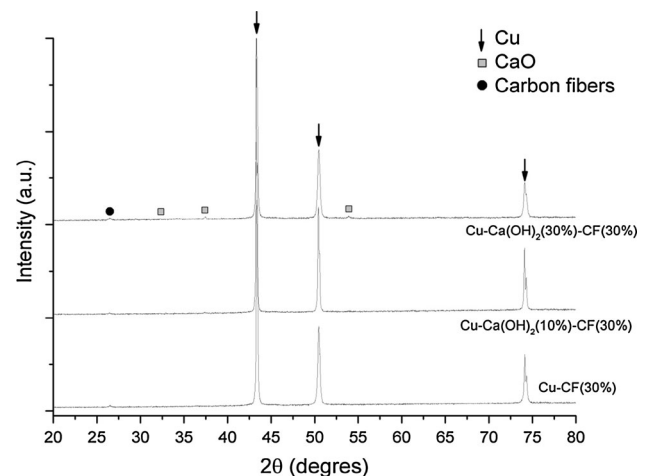


Fig. 1 X-ray patterns of Cu–CF(30 %), Cu(OH)₂(10 %)-CF(30 %), and Cu(OH)₂(30 %)-CF(30 %) composites

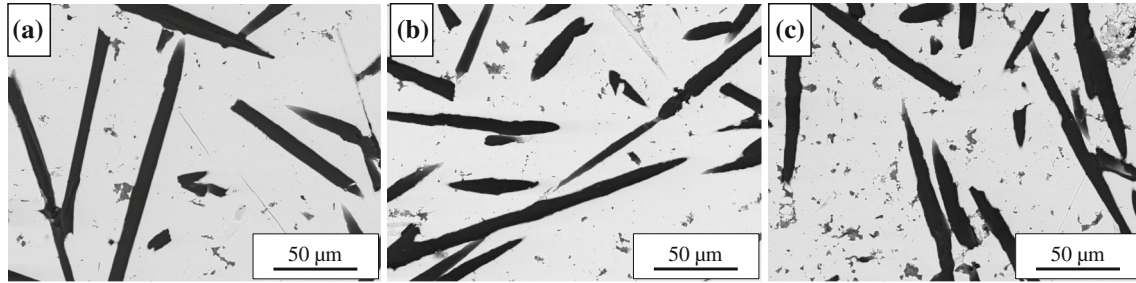


Fig. 2 SEM micrographs (Back Scattering Electrons) of the composites **a** Cu–Ca(OH)₂(3 %)-CF(30 %), **b** Cu–Ca(OH)₂(5 %)-CF(30 %), **c** Cu–Ca(OH)₂(10 %)-CF(30 %)

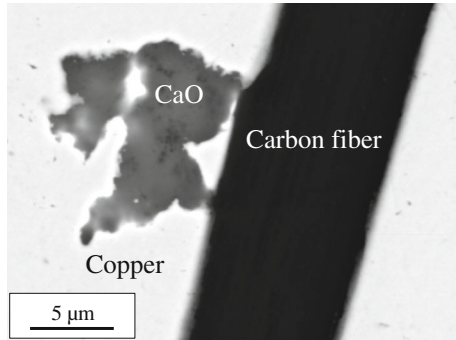


Fig. 3 SEM micrograph (BSE) of the composite Cu–Ca(OH)₂(3 %)-CF(30 %)

The TC properties and the CTE were measured for each sample. The results are given in Fig. 5. The composite without Ca(OH)₂, sintered with the same conditions as the composite with Ca(OH)₂, exhibited a parallel TC of 303 W m⁻¹ K⁻¹, a perpendicular TC of 202 W m⁻¹ K⁻¹, and a parallel CTE of 11.6 × 10⁻⁶ K⁻¹. These values are slightly higher than the ones from the literature and will be used as reference values in this paper [15–17]. For the samples with a low content of Ca(OH)₂ (i.e., ≤ 1 %), the parallel TC did not change; but the perpendicular TC decreased about 5 % for $x = 1$ % of Ca(OH)₂. For $x = 3$ %, an improvement of around 7 % of the parallel TC was measured. In the same way, a higher or similar TC was measured for $x = 5$ and 10 %. This improvement remains unexplained. Indeed, CaO material exhibited a low TC value (i.e., 20 W m⁻¹ K⁻¹). The reasons why the TC increased while a fraction of CaO increased are still unknown. The nanostructuration of CaO from the decomposition of Ca(OH)₂ could be one of the reasons. However, when the content of Ca(OH)₂ increased, the perpendicular TC decreased. The CTE was almost the same for all the composite materials. Indeed, the CTE range is between 11.5 and 12.2 × 10⁻⁶ K⁻¹. Thus, we can suppose that the addition of hydroxide plays no role in thermal expansion. A variation in the CTE can be directly correlated to a change

of the interface [18]. Consequently, the calcium hydroxide (or calcium oxide) did not change the interface properties between the metallic matrix and the CF.

To conclude, the addition of 3 % of Ca(OH)₂ led to an improvement in the parallel TC of 7 % without affecting the interface between the matrix and fibers. We can suppose that chemical interactions at the interface could both decrease the CTE and maintain a good thermal conductivity. For these reasons, few heat treatments have been done on the composite Cu–Ca(OH)₂(3 %)-CF(30 %) in order to enhance the chemical interaction between the matrix and fibers through calcium carbide formation.

Effect of the heat treatment on the Cu–Ca(OH)₂(3 %)-CF(30 %) composite

The Cu–Ca(OH)₂(3 %)-CF(30 %) composite was chosen to analyze the effect of different heat treatment temperatures on interfacial Cu/C and composite properties. The compound was heat treated for 24 h. under nitrogen flux at 400, 600, and 1,000 °C. No evolution of the composite density was analyzed after the heat treatments.

Each sample was studied by EPMA analysis. The maps of the repartition of the carbon, copper, and calcium elements are given in Fig. 6. Before heat treatment, the composite was described as a copper matrix with precipitates of calcium inside. After the heat treatment at 400 °C, the size of the precipitates decreased, and the calcium seemed to be more distributed around the CFs. Nevertheless, some precipitates with a size around 10 microns were still visible in the copper matrix. At 600 °C, the size of the calcium precipitates inside the copper matrix decreased. Moreover, more calcium was observed around the CFs. At 1000 °C, the observations were the same. A higher calcium content was observed along the CFs forming layers as well as precipitates with a size around 10 microns or less. The heat treatment increased the migration behavior of the calcium inside the matrix to the matrix-fiber interface. When the temperature increased, the concentration of calcium at the interface increased.

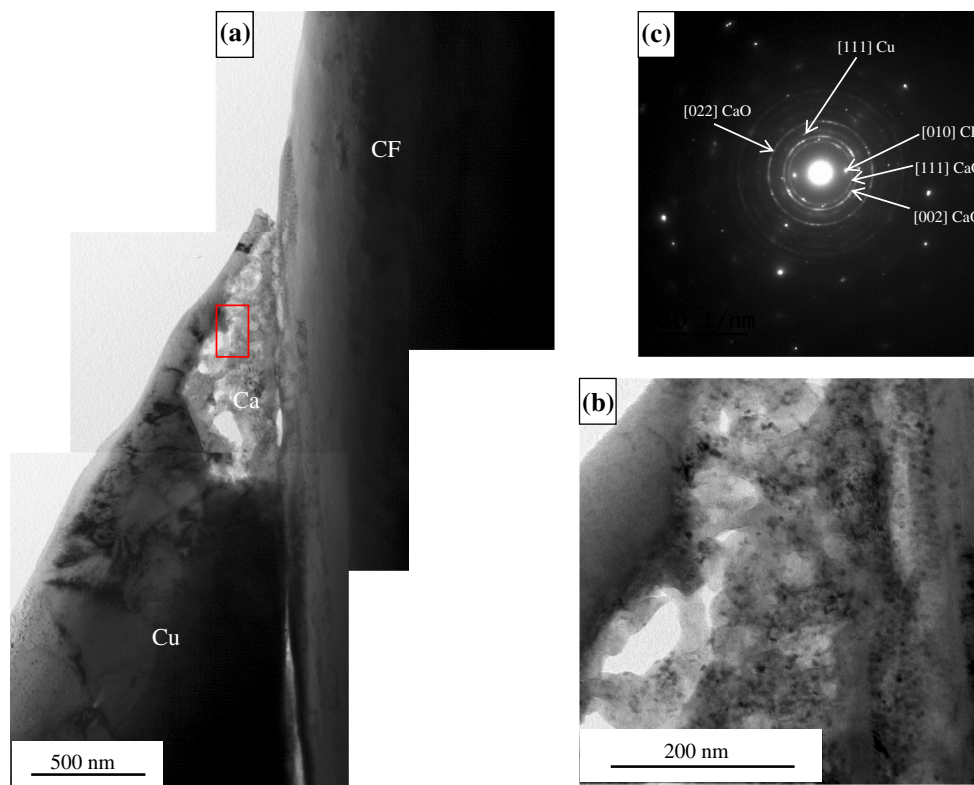
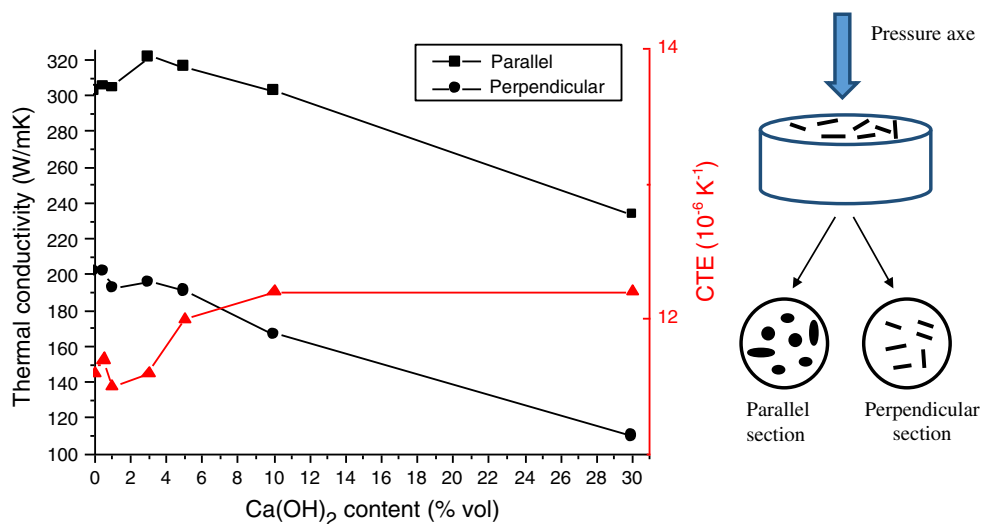


Fig. 4 Micrographs **a**, **b** and the electron diffraction pattern **c** of the compound Cu–Ca(OH)₂(3 %)-CF(30 %), obtained by TEM analysis. The micrograph **b** is a zoom of the micrograph **a** (red rectangle) (Color figure online)

Fig. 5 Thermal conductivities (circle and square) and CTE (triangle) of the composite Cu–Ca(OH)₂(X %)-CF(30 %) (with X = 0, 0.5, 1, 3, 5, 10, 30). The direction of the fibers is given according to the direction of the pressure axe. Consequently, for the parallel section, the conductivity is in the direction of the fiber



In order to study the interface/interphase and the interaction between calcium and CFs, the sample heat treated at 1000 °C for 24 h was analyzed using TEM. Several regions of the Cu/C materials were analyzed (i.e., eight different geographic zones were observed); representative micrographs and electron diffraction patterns are given in Fig. 7. The calcium layer (Fig. 7a) between the copper and CF had a thickness of about 1 μm, and it was several

micrometers long. CaC₂ phase, copper, and CFs can index the electron diffraction patterns performed at the CF–calcium and copper–calcium interfaces and the center of the rich calcium zone. Thus, calcium-rich zones were transformed into CaC₂. In comparison to the sample after sintering, the CaO phase was replaced by the CaC₂ phase. At 1000 °C, a CaO phase reacts with CF to form a CaC₂ interphase. Wu et al. and Li et al. [12, 19] report,

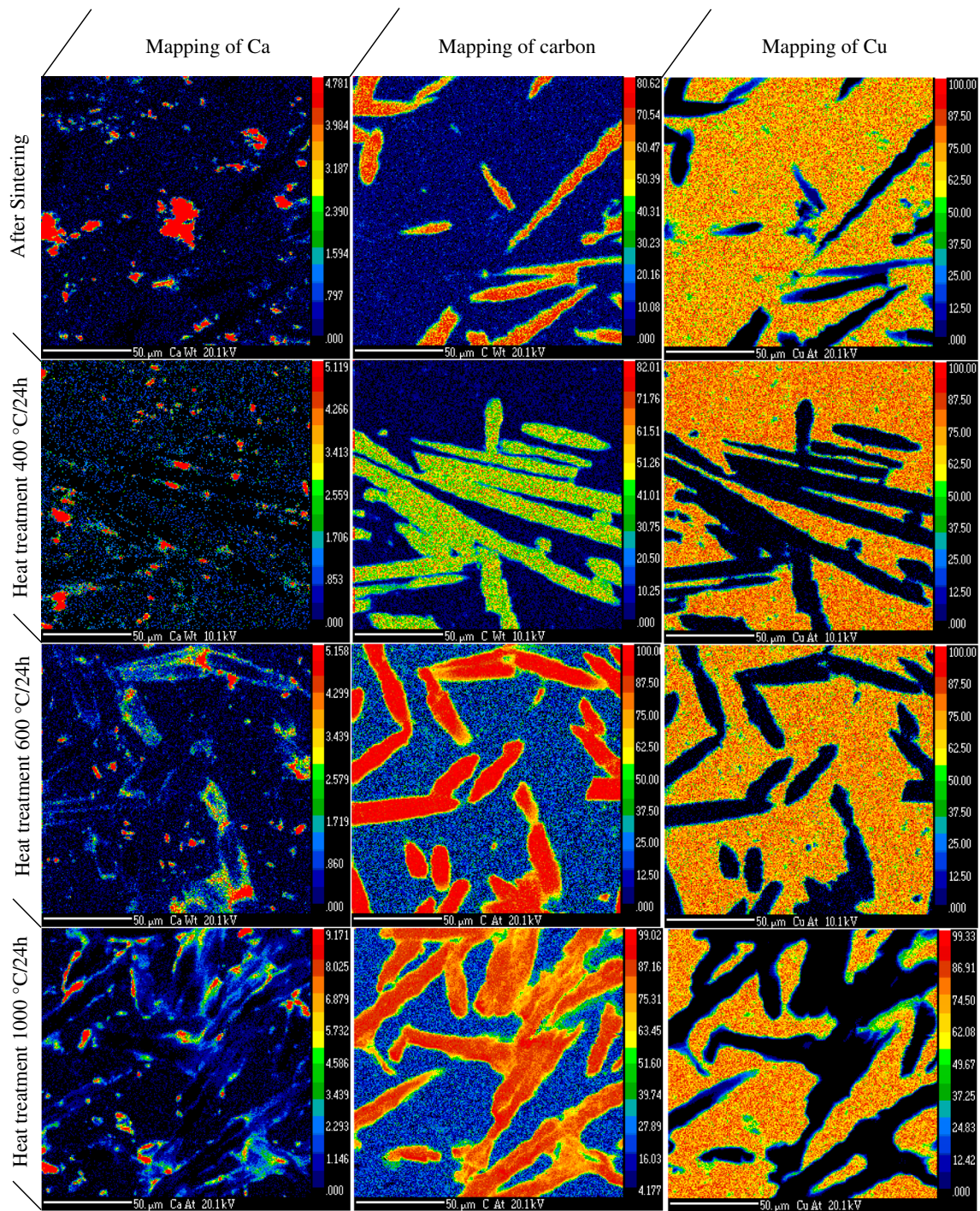


Fig. 6 Mapping of calcium, carbon, and copper elements inside the composite Cu–Ca(OH)₂(3 %)-CF(30 %) obtained by EPMA analysis. The scale bar is the same for each mapping and corresponds to 50 μm

respectively, a minimum of 1000 and 1550 °C for the start of the reaction between CaO and C. Therefore, our results are in good accordance with the literature, albeit with a lower temperature of reaction. Moreover, the formation of CaC₂ was considered total as calcium oxide was not observed around the CFs or inside the copper matrix. The

calcium-rich zones embedded in the copper matrix could not be observed by TEM analysis; therefore, the composition of these precipitates after the heat treatment is unknown.

To summarize, the heat treatment led to a diffusion of calcium oxide at the interface; and then a reaction occurred

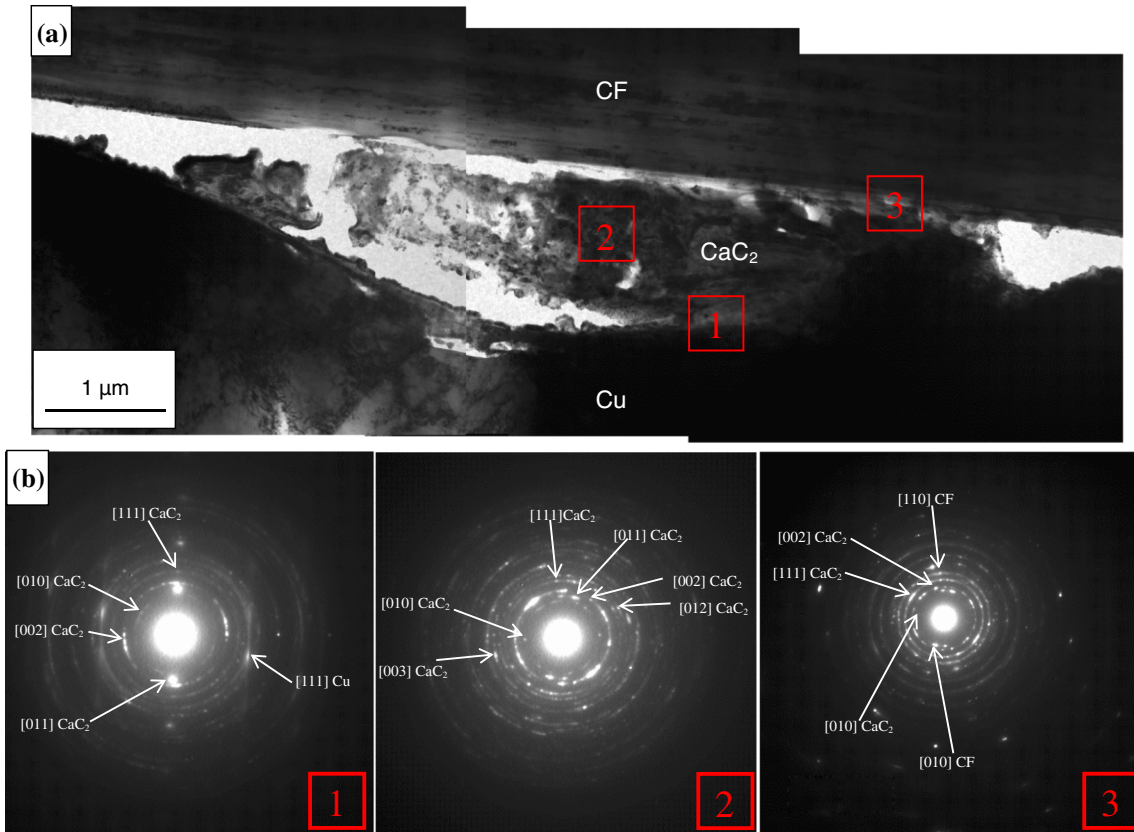


Fig. 7 Micrographs **a** and the electron diffraction pattern done on the selected zones 1, 2, and 3 **b** of the compound $\text{Cu-Ca(OH)}_2(3\%)\text{-CF}(30\%)$ after the heat treatment at $1,000\text{ }^\circ\text{C}$ during 24 h, obtained by TEM analysis

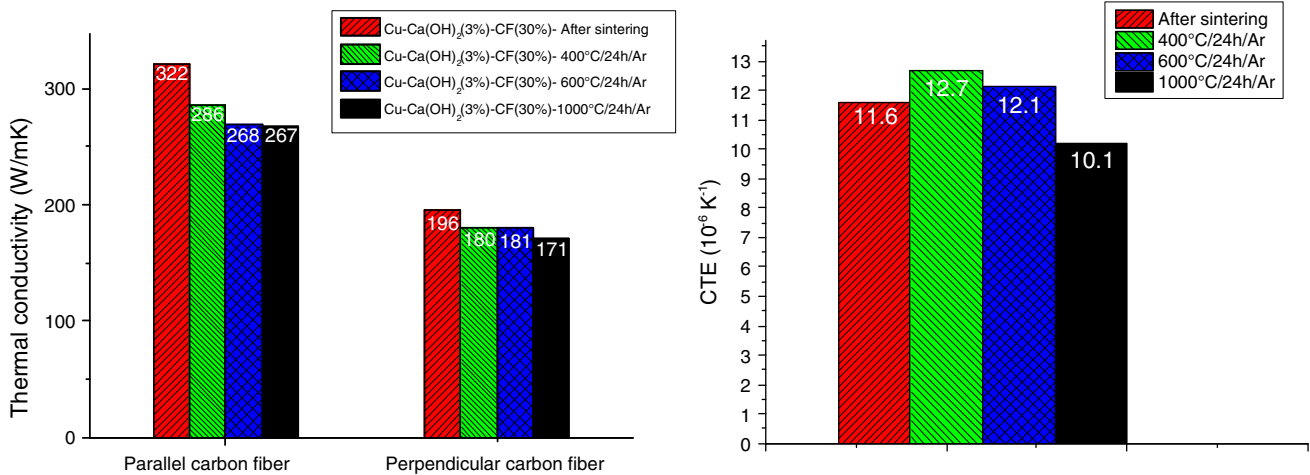


Fig. 8 Thermal conductivities and CTE of the composite $\text{Cu-Ca(OH)}_2(3\%)\text{-CF}(30\%)$ after sintering and heat treatment at 400, 600, and $1,000\text{ }^\circ\text{C}$

between calcium oxide and carbon to form carbide. The first step began at $400\text{ }^\circ\text{C}$; the second step was detected beginning at $1000\text{ }^\circ\text{C}$, which is at the lower end of the temperature range known for this reaction.

The TC properties and the CTE were measured for each sample heat-treated. The results are given in Fig. 8.

From a thermal point of view, the heat treatments induced a decrease in thermal conductivity. For example, the parallel TC decreased about 11 and 16 % after the heat treatment at 400 and $600\text{ }^\circ\text{C}$ (or $1000\text{ }^\circ\text{C}$), respectively. Thus, the calcium oxide diffusion at the interface acted as an obstacle to thermal conductivity. The precipitates of the

calcium oxide inside the copper matrix, for the sample with 3 % of $\text{Ca}(\text{OH})_2$, improved TC while the same calcium oxide in the interface matrix/fiber decreased it. This decrease could be explained by the localization of the calcium oxide. Indeed, the calcium oxide layers around the CFs change the interface/interphase properties. These insulator layers of about 1 micrometer limited the phonon and electron transfer between the copper and the CFs (i.e., formation of a thermal resistance). Nevertheless, the decrease at 1000 °C was unexpected. The formation of carbide should have increased the TC [6, 9, 10]. However, the carbide layers formed during the sintering must be controlled (i.e., strong interface, thin interphase,...) in order to maintain a maximum thermal interface conductance. They showed that more than 1 wt% of carbide elements induced a decrease in the thermal conductivity. The high content of $\text{Ca}(\text{OH})_2$ in our sample (i.e., 3 vol.%) and the thickness of the carbide layers ($\approx 1 \mu\text{m}$) could explain the decrease.

From a thermomechanical point of view, an increase of about 1 unit of the CTE was measured after annealing at 400 °C (i.e., $12.7 \times 10^{-6} \text{K}^{-1}$). Then, the CTE decreased until $10.2 \times 10^{-6} \text{K}^{-1}$ for the sample heat-treated at 1000 °C. These variations can be explained by the calcium diffusion and carbide formation. Indeed, for the composites after sintering, the precipitates of calcium oxide inside the copper matrix did not affect the CTE. At 400 and 600 °C, the diffusion of calcium oxide at the matrix/fiber interface led to the formation of calcium oxide layers. Consequently, the mechanical interface between the copper and the CFs was replaced by a new interface (i.e., copper–calcium oxide–CFs). Therefore, the formation of a brittle phase between copper and CF weakened the interface. The decrease in the CTE at 1000 °C can be explained by the formation of calcium carbide from calcium oxide. This reaction induced the formation of a chemical bond between the CFs and the calcium. This phenomenon led to a stronger interface which limited the thermal expansion.

Conclusion

The effects of adding calcium inside a composite Cu–CF(30 vol.%) were investigated. The goal was to form calcium carbide at the interface of copper and CFs in order to improve the thermal properties and the CTE. Because calcium is highly sensitive to air, the calcium hydroxide was chosen as the element to be added. During sintering, the calcium hydroxide was converted into calcium oxide, located in the copper matrix. For 3 vol.% of $\text{Ca}(\text{OH})_2$, a 7 % improvement in the parallel TC and a stable CTE were measured. Several heat treatments were then done on the sample with 3 vol.% of $\text{Ca}(\text{OH})_2$. At 400 and 600 °C, the

calcium oxide diffused from the copper matrix to the interface of the copper and CFs. This led to the formation of a brittle and insulator phase at the interface, which led to a decrease in the TC and an increase in the CTE. At 1000 °C, the calcium carbide formation occurred; and a decrease in the CTE was measured. Thus, we highlight the carbide formation from $\text{Ca}(\text{OH})_2$ in the Cu– $\text{Ca}(\text{OH})_2$ –CF composite using chemical reactions during sintering and heat treatment. Using the same method, with a lower content of $\text{Ca}(\text{OH})_2$ (i.e., 0–0.5 vol.%), could lead to the improvement of the global thermal mechanical properties correlating with a decrease in the CTE.

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