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# Sintered molybdenum for a metallized ceramic substrate packaging for the wide-bandgap devices and high temperature applications

Bassem MOUAWAD, Cyril BUTTAY, Maher SOUEIDAN, Hervé MOREL  
Ampère, UMR 5005  
Université de Lyon, INSA de Lyon  
21 Av. J. Capelle 69621 Villeurbanne, France  
[bassem.mouawad@insa-lyon.fr](mailto:bassem.mouawad@insa-lyon.fr),  
[cyril.buttay@insa-lyon.fr](mailto:cyril.buttay@insa-lyon.fr), [maher.soueidan@insa-lyon.fr](mailto:maher.soueidan@insa-lyon.fr),  
[herve.morel@insa-lyon.fr](mailto:herve.morel@insa-lyon.fr)

Vincent BLEY  
LAPLACE  
UPS, INPT  
118 route de Narbonne 31062 Toulouse, France  
[vincent.bley@laplace.univ-tlse.fr](mailto:vincent.bley@laplace.univ-tlse.fr)

Damien FABREGUE  
Mateis, UMR 5510  
Université de Lyon, INSA de Lyon  
7 Av. J. Capelle 69621 Villeurbanne, France  
[damien.fabregue@insa-lyon.fr](mailto:damien.fabregue@insa-lyon.fr)

**Abstract**—Silicon Carbide (SiC) is a good candidate for high temperature power electronic applications. To ensure good reliability, packaging materials with a coefficient of thermal expansion (CTE) matching that of SiC are needed. A metallized ceramic substrate based on aluminium nitride (AlN) and molybdenum (Mo) is reported in this paper. This substrate is built using a spark plasma sintering equipment. Results show that a dense Mo layer can be sintered on an AlN plate, with good adhesion, forming a Mo/AlN/Mo structure with well-matched CTEs.

**Keywords**—Wide-bandgap devices; aluminium nitride; molybdenum; Spark Plasma Sintering

## I. INTRODUCTION

Wide-bandgap semiconductor devices, such as silicon carbide (SiC), have pushed the boundaries in terms of power density, conversion efficiency or switching speed compared to silicon [1]. In particular, SiC devices can operate at much higher temperature [2]. These devices are assembled into modules, which provide electrical interconnects, thermal management (by using a metallized ceramic substrate, a heat-spreader and a heat sink) and electrical insulation (provided by the ceramic layer of the substrate). However, this package, which comprises many different materials (ceramic, metals, organic materials...) with vastly different Coefficients of Thermal Expansion (CTE), is very sensitive to thermal cycling. Indeed, any change in the operating temperature will result in a variation of the mechanical stresses experienced at the various interfaces. Repetitive temperature variations (thermal cycling conditions), and the associated stress changes, are a source of mechanical fatigue, which can lead to failure of the package.

With the extended temperature range (and therefore with the extended thermal cycling) permitted by SiC devices, the package becomes a limiting factor, and new technologies must be proposed to achieve more robust modules [3].

## II. PACKAGING

### A. Metallized ceramic substrate

In this paper, we focus on a specific part of the package, namely on the metallized ceramic substrate. This substrate concentrates many of the challenges described above: it has a large surface (usually in the order of around 10-cm<sup>2</sup>), associates materials with different CTEs (a ceramic and a metal), and must provide good electrical conductivity (of the metal layer) and thermal conductivity (of the metal and ceramic layers).

The most substrate used in conventional packaging is the Direct Bonded Copper (DBC). The DBC is formed by a ceramic layer (Aluminium oxide -Al<sub>2</sub>O<sub>3</sub>- or aluminium nitride -AlN-) between two copper layers. This DBC structure leads to a high differential stress due to the materials CTE mismatch that may cause failure at high temperature [4] (see table below).

### B. Materials

Some of the materials found in a high temperature power module are listed in Table I, along with their thermal properties. Recently, aluminium nitride, which has CTE properties very close to that of SiC, was proposed as a substrate material for high-temperature device packaging [5-7]. A gold thick-film metallization on AlN ceramic-based 500 °C packaging for SiC devices has been demonstrated [8]. The electrical interconnection system of this approach has been tested in air environment for over 5000h at 500 °C and a packaged SiC test device operated for over 1000h. Other approaches found in the literature include a molybdenum (Mo) thick film metallization performed by screen printing of molybdenum-manganese paste on AlN and tungsten (W) thin film metallization sputtered on AlN with a proprietary adhesion enhancer (i.e. Titanium (Ti)) since W does not adhere to AlN [9]. The adhesive strength for both, Mo and W

TABLE I. THERMAL PROPERTIES OF DIFFERENT MATERIALS OF A POWER ELECTRONIC MODULE [9]

Materials	Thermal properties	
	CTE ( $\times 10^{-6}/^{\circ}\text{C}$ )	Thermal conductivity (W/m.K)
Copper (Cu)	17	398
Molybdenum (Mo)	4.8	138
Tungsten (W)	4.5	178
Gold (Au)	14	315
Aluminium Oxide ( $\text{Al}_2\text{O}_3$ )	7.2	25
Silicon Carbide (SiC)	3.7	300
Aluminium Nitride (AlN)	4.1	200

metallizations, was found to be 87 MPa and 84 MPa respectively after a heat treatment of 1100 °C for 30 min.

As with these technologies, the work described here focuses on replacing the metallized ceramic substrate used in conventional packaging systems to a new one that will be able to withstand thermal cycling.

### C. Molybdenum / AlN

As discussed in the last section, for the high temperature operation (and wide-range thermal cycling), both the ceramic and the metal layers need to have a CTE close to that of the SiC devices. Mo was found to be a promising candidate for high temperature metallized ceramic substrate and has many good advantages: (i) similar CTE to AlN and SiC ( $4.8 \times 10^{-6}/^{\circ}\text{C}$  for Mo vs.  $4.1 \times 10^{-6}/^{\circ}\text{C}$  for AlN vs.  $3.7 \times 10^{-6}/^{\circ}\text{C}$  for SiC, see table I), (ii) good adhesion material, (iii) relatively low cost, (iv) it is widely used in electrical contact for high temperature. The disadvantages are: (i) it is very difficult in achieving full densification and (ii) very difficult to assembly with AlN without any additives using standard solution [9]. Preliminary results in the densification of pure Molybdenum shows that a full densification can be obtained using a new sintering technique such as Spark Plasma Sintering [10]. We suggest to use the same sintering technique to realize both molybdenum powder sintering, and Mo / AlN layers bonding simultaneously.

## III. EXPERIMENTAL PROCEDURE

### A. Spark plasma sintering

As with a classical sintering press, a Spark Plasma Sintering (SPS) system applies both pressure and heat to samples. The difference is that the heat is provided by a pulsed DC-current circulating through a graphite die (or through the sample if it is conductive), whereas the conventional sintering press uses external heating elements. SPS offers rapid sintering, with ramps of up to 700 °C/min using current pulses of several kA. This has great potential for achieving fast densification results with minimal grain growth in a short sintering time.

The experiments were carried out in a SPS FCT systeme GmbH, HP D 25 model. This apparatus consists of a uniaxial press with a maximum force of 250 kN and a power supply

capable of producing a pulsed current DC-current with a maximum of 8000 A at 8 V.

### B. Samples preparation

Commercial Molybdenum powder (Goodfellow, Cambridge, UK, 99.99+% purity) with a particle size ranging from 55  $\mu\text{m}$  to 355  $\mu\text{m}$  was consolidated using SPS technique. In this technique, molybdenum powders were poured directly, with no special treatment into the cylindrical graphite die of SPS without any additive or binder.

For the ceramic layer, disks of 19,8 mm in diameter were laser-cut into commercial plates of AlN with two different thicknesses (633  $\mu\text{m}$  and 1270  $\mu\text{m}$ ). The disks were cleaned using (i) ultrasonic cleaning in acetone, (ii) ultrasonic cleaning in microelectronic grade ethanol, (iii) immersion in  $\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2$  (1:1) to ensure non-existent of any metal particles (iv) followed by rinsing in DI water and blow drying with nitrogen gas.

Two sets of experiments were performed. The first set focussed on the molybdenum sintering parameters, and no AlN disks were used, Molybdenum powder samples of 20 mm in diameter and around 2 mm-thick were sintered in vacuum ( $10^{-2}$  Torr), without any additives. The second set of experiments was targeted at the Mo/AlN assembly. The 19,8 mm disks of AlN were put in 20 or 40 mm diameter graphite dies, and covered with a 1 mm-thick layer Mo powder. These samples were also sintered in vacuum ( $10^{-2}$  Torr).

Precise density measurements of the consolidated bulk molybdenum samples were made using the Archimedes' principle according to ASTM Standard B328-94. The relative density was calculated based on the theoretical density of Mo ( $10.22 \text{ g/cm}^3$ ).

At the end of each experiment, the samples were cross-sectioned, mounted in a hot mounting resin (Presi Phenolic resin) and then prepared metallographically (mechanical

TABLE II. MOLYBDENUM'S SPS SINTERING PARAMETERS

Sample	Molybdenum sintering parameters		
	Temperature ( $^{\circ}\text{C}$ )	Holding Time (min)	Relative Density (%)
1	1200	5	84.4
2	1350	5	91.2
3	1450	5	94.8
4	1550	5	96.5
5	1650	5	97.0
6	1750	5	98.4
7	1850	5	98.6
8	1950	5	99.5
9	1850	10	99.6
10	1850	20	99.8
11	1850	30	100

a. Molybdenum sintering parameters reported in [10]

grinding and polishing down to 0,05  $\mu\text{m}$  silica slurry). The samples were examined using optical and scanning electron microscopy (SEM). Energy dispersive X-ray spectroscopy (EDX) was used to identify the interface materials reaction.

### C. Mo powder Sintering

The sintering of the molybdenum powder using a SPS is presented in details in [10]. Table II presents the relative density in function of sintering temperature, ranging from 1200  $^{\circ}\text{C}$  to 1950  $^{\circ}\text{C}$  with a holding (sintering) time ranging from 5 to 30 min, under a fixed pressure of 77 MPa and a fixed heating rate of 700  $^{\circ}\text{C}/\text{min}$ . A relative density of 100% is achieved at a sintering temperature of 1850  $^{\circ}\text{C}$  hold for 30 min. Fig. 1 shows a cross-sectional optical micrograph of a bulk of molybdenum sintered at 1850  $^{\circ}\text{C}$  for 5 min. The sample was chemically etched in  $\text{H}_2\text{SO}_4:\text{HNO}_3:\text{H}_2\text{O}$  (1:1:3) solution. It shows that there are almost no pores between grains of Mo.

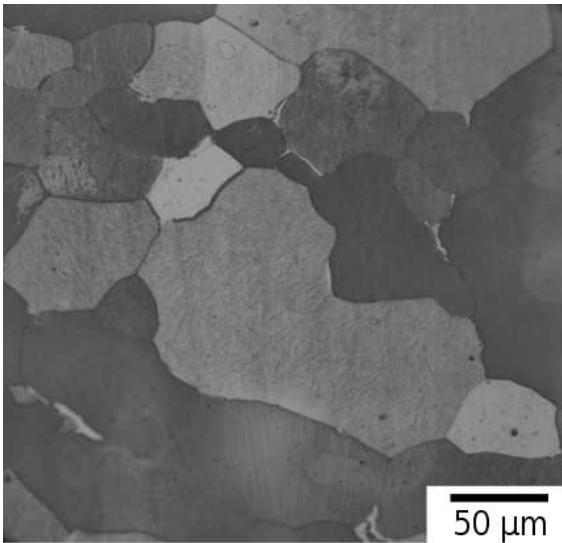


Figure 1. Cross-sectional optical micrograph of bulk molybdenum

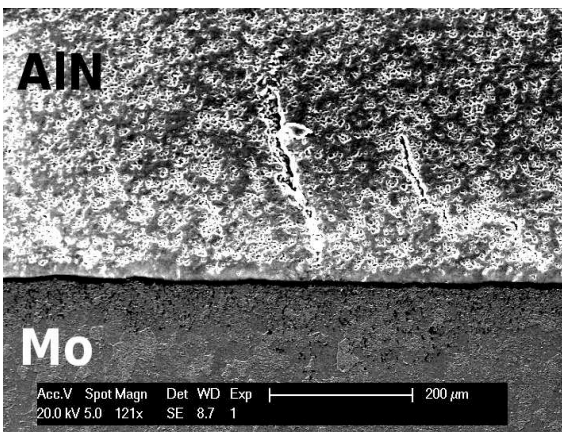


Figure 2. Cross-sectional SEM photo of Mo/AIN interface

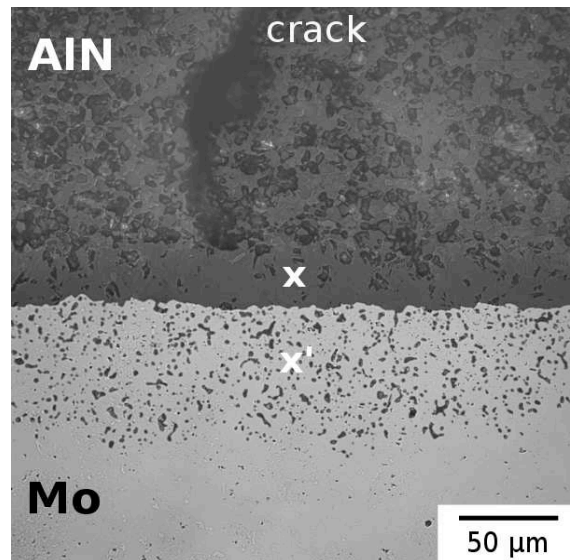


Figure 3. Optical microscope observation of the cross section of an AlN/Mo assembly. The “X”s indicate zones where EDX measurements were performed to analyze the chemical species present close to the interface

### D. Molybdenum / AlN assembly

First, Molybdenum powder was sintered on a disk of AlN with thickness of 633  $\mu\text{m}$  using a 20 mm diameter’s graphite die. We achieved a good assembly at the Mo/AlN interface for all samples. However, we noticed that the AlN layer fissured during the process. We attributed these cracks to the pressure applied by the SPS press. However, even at the lowest force setting (5 kN, or 16 MPa for a 20 mm sample diameter), cracks occurred. To avoid cracks, we used a disk of AlN with a higher thickness (1.27 mm), and placed it in a 40 mm-diameter graphite die.

Fig. 2 shows a cross-sectional SEM photo of a Mo/AlN interface, for a sample assembled at 1850  $^{\circ}\text{C}$  with a force of 5 kN applied by the press (which corresponds to 4 MPa in the 40 mm diameter die) with a holding time of 5 min. It shows the sublimation of a layer of AlN occurring during the sintering. Moreover, small cracks can also be seen, but they are limited to the neighbourhood of the interface. A close-up is given in fig.3. Some EDX characterization were performed and, shows that at the x marks, we have an area in the ceramic layer rich in aluminium. This means that some reaction occurred at the AlN/Mo interface.

## IV. DISCUSSION

SPS allows for molybdenum sintering at "low" temperature (much lower than the melting point of molybdenum, at 2623 $^{\circ}\text{C}$ ), with a high density (up to 100%). Using the same equipment, a bond between molybdenum and aluminium nitride was successfully achieved, although some sublimation of AlN occurred. To reduce this effect, we can reduce the sintering temperature. The microstructure at the interface between Mo and AlN must be conducted in order to get a better insight of the phenomena that have taken place. Moreover, a bending test will now be carried out to ensure that the bonding strength is good enough for our applications. At

last but not least an electrical characterization (dielectric strength) is required as well as a thermal cycling to study the reliability of the substrate.

#### V. CONCLUSION

Molybdenum powder was sintered to bulk using a Spark Plasma Sintering technique. This technique allows to achieve full densification in a few minutes. Molybdenum powder was sintered on disks of AlN, at 1850 °C for 5 min with a pressure of 4 MPa, showing a chemical reaction at interface level. Small cracks can also be seen, but they are limited to the neighbourhood of the interface.

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