NANOENERGETICS ON A CHIP: TECHNOLOGY AND APPLICATION FOR MICRO IGNITION IN SAFE ARM AND FIRE SYSTEMS

M. Petrantoni[1,2]*, M. Bahrami[1,2], L. Salvagnac[1,2], V. Conédéra[1,2], C. Rossi[1,2], P. Alphonse[3], C. Tenailleau[2,3]

[1]CNRS; LAAS; 7 avenue du colonel Roche, F-31077 Toulouse, France
[2]Université de Toulouse; UPS, INSA, INP, ISAE, LAAS; F-31077 Toulouse, France
[3]CIRIMAT, 118 route de Narbonne, 31062 Toulouse cedex 04, France
*Presenting Author: mpetrant@laas.fr

Abstract: A thin film (1 - 4 mm²) of nanoenergetic material made of Al and CuO multilayers is deposited on a chip by dc reactive magnetron sputtering method to produce electro-pyrotechnic initiators. It consists in 21 thin layers of Al (100 nm) and CuO (100 nm) successively stacked on a Cr/Pt resistance patterned on the pyrex microinitiator. Different tests have been performed to evaluate the ability of the Al/CuO nanothermite to be ignited by Joule effect and then to ignite a propellant placed close to the nanothermite. A few Watts are required for the electrical initiation. The electro-pyrotechnic initiator integrating the nanothermite can initiate a propellant in contact or through an air gap of ~ 270 µm. Beyond this distance ignition failed. This technology called "nanoenergetics on a chip" can open applications in many fields such as micropropulsion, microwelding and microinitiation.

Keywords: energetic material, nanothermite, propellant, aluminum, copper oxide, microinitiator

INTRODUCTION

For several years, technologies have been adapted and reviewed to realize the way between micro and nano-systems. Materials have known significant change and new materials have been developed, in particular among energetic materials (EM) which represent an interesting source of onboard energy. These materials could be integrated into micro and nanosystems to produce gas or release heat. Nanothermites consist of a metal (e.g. aluminum) and an oxidizer [1]. Al/CuO is interesting for "nanoenergetics-on-a-chip" because this material is a composite material which exhibits highly exothermic reaction (ΔH_{theor} = 21kJ/cm^3) [2] when it is heated and the aluminum and copper oxide are commonly used in a microelectronic process flow. To fasten the Al/CuO reaction, it has been already demonstrated the necessity to increase the specific surface area between CuO and Al in order to increase reactants intimacy and reduce the reactants diffusion distance [3-5]. We present in this paper a dc reactive magnetron sputtering method permitting to integrate multilayered Al/CuO nanothermites on a glass microinitiator for firing systems [5]: this is called electro-pyrotechnic microinitiator. First, the Al/CuO nanothermite is characterized by scanning electron microscopy (SEM) and differential scanning calorimetry (DSC). Then, the Al/CuO nanothermite is deposited onto a microinitiator to be ignited by Joule effect and to determine its ability to ignite a propellant in contact or through an air gap. Field of application covers, for example, the ignition of pyrotechnic chains for space, defense, aeronautics and automotive.

EXPERIMENTS

Glass microinitiator

A Cr/Pt microheater was realized to ignite by Joule effect the Al/CuO nanothermite. The process is described as follows.

Substrate preparation: the pyrex substrate is introduced for two minutes in a bath which composed of 50% in volume of hydrogen peroxide (H_2O_2) and 50% in volume of sulfuric acid (H_2SO_4). The step allows the elimination of organic contamination on substrate.

Step 1: First, a negative photoresist is spin coated on the pyrex substrate (see fig. 1). A photolithography is performed to pattern the resist. Three thin metallic layers are deposited by thermal evaporation: Cr (20 nm), Pt (120 nm) and Au (800 nm). Resist is removed in an acetone bath during ~ 12 hours.

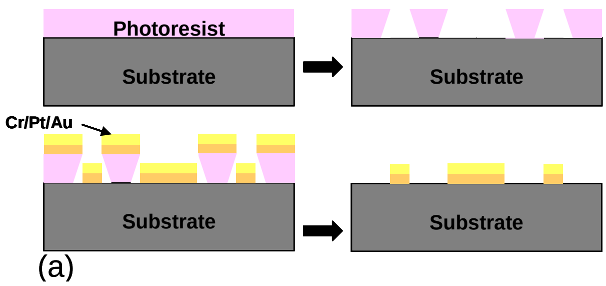

Fig. 1: Cr/Pt/Au deposition.

Step 2: Au is etched away on the filaments of the resistance by a KI-H_2O solution. The aim is to have the most conductive material on the contacts of the resistance and the most resistive materials close to the energetic material.

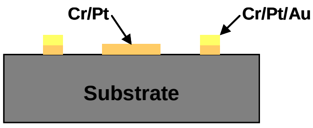

Fig. 2: Gold etching

Step 3: A Si_3N_4 membrane is deposited by Plasma-enhanced chemical vapor deposition (PECVD) on the resistance (see figure 3). This layer is deposited to prevent the potential short circuit in the following process. Its thickness is chosen to provide electric insulation and thermal conduction.

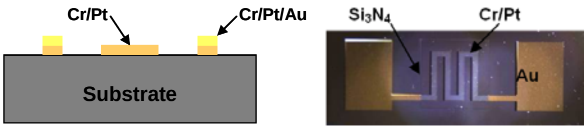

Fig. 3: Si_3N_4 deposition

All the resistances have a value of ~ 66 Ω and the dimensions are showed in the figure 4.

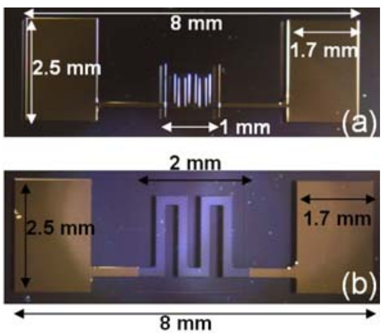

Fig. 4: Resistances dimensions for the initiation of (a): 1 mm^2 and (b): 4 mm^2 of nanothermite

Step 4: A multilayered energetic material is deposited by dc reactive magnetron sputtering. Aluminum and copper oxide layers are successively stacked (see figure 5). The aluminum layer was made from an aluminum target under argon plasma and the copper oxide layer was produced from a copper target under argon-oxygen plasma. The process was set up to produce low stress (< 50 MPa) multilayered Al/CuO, each individual layer having a thickness of 100 nm (see figure 6). The dc reactive magnetron sputtering parameters are summarized in the table 1.

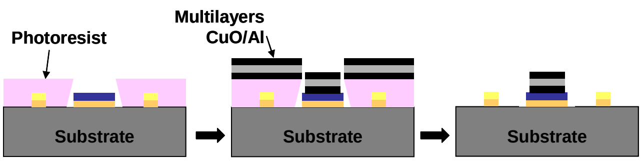

Fig. 5: Multilayered Al/CuO sputtering on the initiator

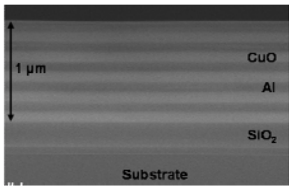

Fig. 6: SEM cross-section images of the CuO(100nm)/Al(100nm) multilayers magnetron sputtered.

Table 1: Multilayered Al/CuO deposition parameters.

	Al	CuO
Target-substrate distance (cm)	8.5	8.5
Argon flow rate (sccm)	50	100
Oxygen gaz flow rate (sccm)	0	25
Ultimate pressure (Pa)	2. 10^{-5}	2. 10^{-5}
sputtering pressure (Pa)	1.10^{-1}	5.10^{-1}
DC power (W)	800	400
Substrate temperature (K)	283	283

DSC analysis allows determining the heat release of the thermite reaction (see fig. 7): the onset temperature is ~ 740 K. The heat of reaction is released below the melting point of Al. And the total heat of reaction is ~ 1.2 kJ/g.

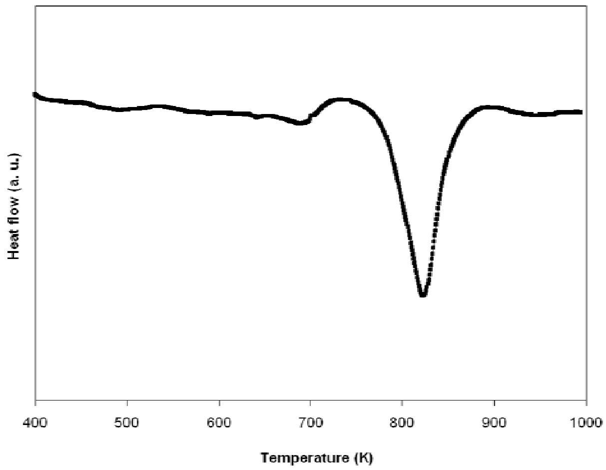

Fig. 7: DSC measurement of a multilayered Al/CuO sample

Components assembling: Electro-pyrotechnic microinitiator

To characterize the electro-microheater, we have designed a test vehicle using a Printed Circuit Board (PCB). The pyrex microinitiator on a chip is electrically bonded to the PCB using a conductive glue (see photo on figure 8a).

The central hole on the PCB allows placing the mass of propellant to be ignited in front of the nanothermite (see figure 8c). The distance between the nanothermite and the propellant is given by the thickness of the PCB (it must also take into account the thickness of gold contact, nanothermite, conductive glue…). The two vias are realized for electric connections on both sides of the PCB. The silicone ring around the glass chip insulates the system (see Figure 8b).

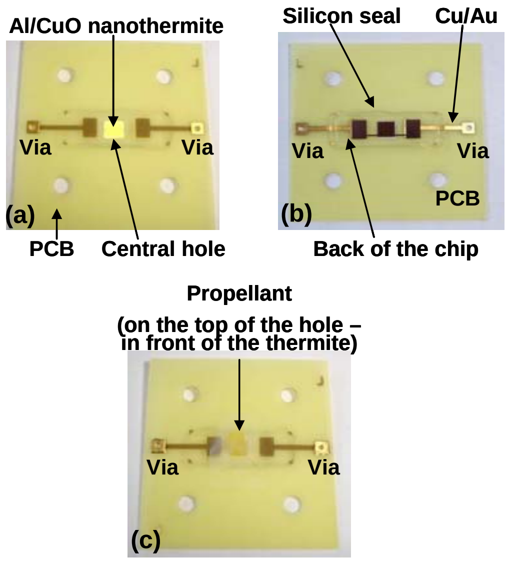

Fig. 8: Microinitiator bonded on PCB.

RESULTS AND DISCUSSION

Nanothermite initiation

The ignition and combustion testing of the electro-pyrotechnic microinitiator is achieved by inputting a current through the Au contact of the resistance. It means that the Al/CuO multilayered nanothermite is locally heated by Joule effect. The voltage and current flowing through the resistance and leading to the reaction of the nanothermite are registered to determine the initiation power. The current value is 0.17 – 0.25 A for an initiation of 1 mm^2 of nanothermite and 0.35 A for the initiation of 4 mm^2 (with the same resistance value). So, the nanothermite is ignited by few Watts (2 – 10 W). After the ignition, the reaction is accompanied by a bright flash of light: see figure 9.

Fig. 9: Successful nanothermite initiation by Joule effect (~ 38-39 µg of Al/CuO).

The figure 10 shows a microinitiator before and after a successful ignition.

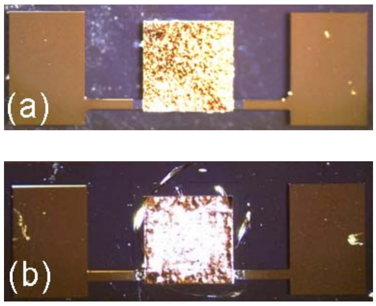

Fig. 10: Microinitiator (a): before and (b): after nanothermite initiation by Joule effect

If the input electrical current is too low (0.12 A for an initiation of 1 mm^2 of nanothermite), ignition fails: it means that the reaction starts on hot points (resistance filaments) but quenches. No spark and sustained heat release are observed (see figure 11).

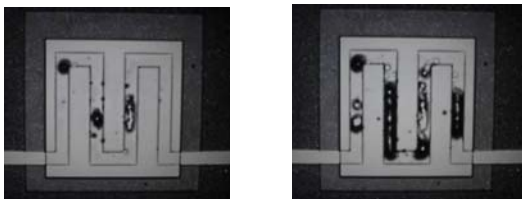

Fig. 11: Low initiation power. Nanothermite is ignited only on the hot points.

Initiation and combustion of a propellant by the nanothermite

A propellant composed by nitrocellulose (called double base) has been tested in open-air conditions. We proceeded to two tests:

- The propellant is placed directly in contact with the nanothermite (see Figure 12). In that configuration and under air, applying 0.35A to the electrical resistance, propellant is ignited (see fig. 13).

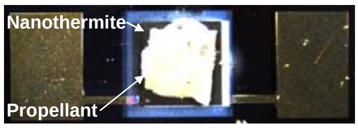

Fig. 12: Electric-pyrotechnic initiator with propellant placed in contact with the nanothermite.

Fig. 13: Propellant combustion after its ignition by the nanothermite.

- For the second test, we leave an air gap between the mitrocellulose propellant and the nanothermite (see Figure 14).

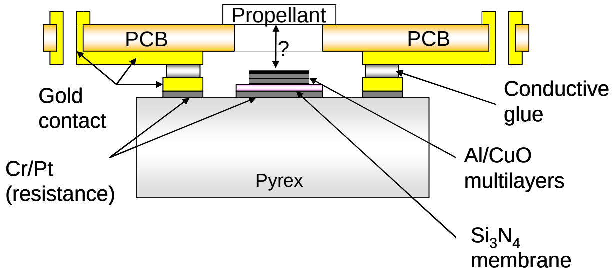

Fig. 14: Schematic view of the propellant initiation system by the nanothermite

Initiation of the propellant at different separation distances (fixed by the PCB thickness) are tested (see figure 14) under the same conditions: electrical current of 35 A.

For 150 µm PCB thickness, the double base propellant is ignited and it burns entirely (see figure 15). Beyond 150 µm, the nanothermite could not ignite the propellant. The air gap dissipates the thermite reaction energy.

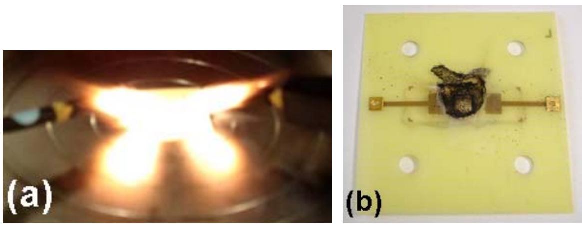

Fig.15: Microinitiator during (a) and after (b) propellant initiation by nanothermite. The distance between the two energetic materials is ~270 µm

Considering the actual thicknesses of the bilayer Cr / Pt (~ 167 nm), the Si_3N_4 membrane (~ 436 nm), the conductive adhesive (~ 34 µm), the gold contact on PCB (~ 81µm), the glass substrate of the microinitiator (~ 765 nm) and the nanothermite (2.1 µm), the maximum separation distance giving propellant ignition by the nanothermite is about 270 µm ± 10 µm.

CONCLUSION

We have developed a simple, reproducible and collective process to produce electro-pyrotechnic initiator integrating multilayered Al/CuO on a Cr/Pt resistance. The nanothermite deposition is done by reactive magnetron sputtering in argon-oxygen gas mixture plasma. It permits to obtain low stress (< 50 MPa) stacks of Al/CuO nanosized layers: the area of nanothermite deposition and Al and CuO individual thicknesses can be easily changed depending on the application.

We have also demonstrated that electro-pyrotechnic initiator integrating nanothermite can initiate a second energetic material such as a propellant: in contact or through an air gap. Under air open condition, the maximum distance between the nanothermite and the propellant is ~ 270 µm to achieve the propellant ignition. Beyond this distance ignition failed.

Finally, this work demonstrates the interest of this new technology called “nanoenergetics on a chip” to fabricate electro-pyrotechnic initiator without manipulation of dangerous products and using only collective processes. This technology can open applications in many fields where controlled ignition is required (such as micropropulsion, microwelding and microinitiation).

REFERENCES

[1] C. Rossi, K. Zhang, D. Estève, P. Alphonse, P. Tailhades, and C. Vahlas, J. microelectromechanical systems 16(4), 919 (2007)

[2] 16. S. H. Fischer and M. C. Grubelich, Conference: 24th International Pyrotechnics Seminar, Monterey, CA, 27-31 July (1998).

[3] K. B. Plantier, M. L. Pantoya, and A. E. Gash, Combustion and flame 140, 299-309 (2005)

[4] B. S. Bockmon, M. L. Pantoya, S. F. Son, B. W. Asay, and J. T. Mang, J. Appl. Phys. 98, 064903 (2005)

[5] M. Petrantoni, C. Rossi, L. Salvagnac, V. Conédéra, A. Estève, C. Tenailleau, P. Alphonse, Y. J. Chabal, Multilayered Al/CuO thermite formation by reactive magnetron sputtering: nano versus micro, Journal of Applied Physics, accepted