

Fabrication and electrical explosion of igniters based on Al/CuO reactive multilayer films

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Abstract

This paper presents novel energetic igniters prepared by integrating Al/CuO reactive multilayer films (RMFs) with Ti films. The as-deposited Al/CuO RMFs energetic materials were firstly characterized with varied analytical techniques. Results show that distinct layer-structure Al/CuO RMFs are deposited, giving a reaction heat of $2760\text{J}\cdot\text{g}^{-1}$. The structure of igniter is designed to be similar to a capacitor, which can form an electrical field across the igniter, and once initiated, instantaneous large current flew through the igniter. Firing characteristics of the igniter were determined using constant voltage firing set. Experimental results show that ignition delay time and total energy released of the igniter discharged in 30 V are 0.75 ms and 389.34 mJ, respectively. In addition, the explosion temperature over 3500 K could last 2.4 ms.

Keywords: dielectric structure, energetic igniter, electrical explosion

1. Introduction

Electrical igniters are applied widely in military and civilian functions such as triggering detonators, propulsion systems, inflation of airbags and so on; however, few attention have been focused on the developing of new igniters in the last decades. As two classical igniters, hot-wire and semiconductor bridge (SCB) have been widely used for a long time; nevertheless, there are some problems remaining such as smaller output energy compared with input energy as well as not very good integration with micro electromechanical system (MEMS) technologies.

In the past few years, reactive multilayer films (RMFs) have attracted extensive attention for their extraordinary advantages in terms of energy release and ignition properties. Usually, RMFs are composed of metal and oxidizer films alternately deposited in vacuum chamber. The specific surface area of RMFs increases dramatically as the monolayer thickness approaches nano-scale, which allows more fuel to be in contact with oxidizer. The layer structure makes the reactants in intimate contact and reduces interfacial impurities, so that the diffusion distances in RMFs are reduced 10–1000 times compared to their bulk counterpart, thereby enhancing atomic

mixing. For these reasons, exothermic reactions of RMFs often have higher flame temperatures and combustion rates.^{1)–10)}

The aim of this paper is to design and prepare dielectric structure energetic igniters by integrating Al/CuO RMFs with two Ti heating layers; furthermore, electrical explosion properties of the igniters are discussed.

2. Deposition and characterization of Al/CuO RMFs

Al/CuO RMFs were RF-magnetron sputtering deposited with ultrahigh purified Ar 30sccm onto a Pyrex 7740 glass substrate. The deposition pressure of the chamber was 0.4Pa and the purity of all the targets materials was above 99.9%. Al target was sputtered at 150 W and CuO target was sputtered at 200 W. Al/CuO RMFs are difficult to sputtering deposit because these materials are highly reactive. Heat generated during sputtering process could be sufficient to initiate the exothermic reaction. In addition, aluminum may be pre-oxidized by stray sputtered oxygen atoms from CuO target. These difficulties, however, were averted by cooling the substrates and shielding the sputter guns. In order to prevent RMFs mixing and reacting prematurely during deposition, the substrate carousel was water cooled at

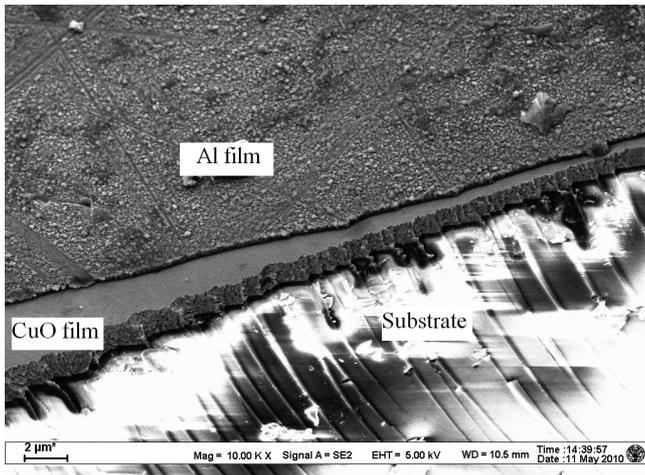


Figure 1 Cross-section-view SEM image of as-deposited Al/CuO bilayer period.

25°C and the sputter guns were shielded. The Al/CuO RMFs were then sputtering deposited without obviously premature reaction. Deposition process was programmed so that aluminum and copper oxide were deposited periodically for a certain number of runs to keep the total thickness consistent.

The modulation period of Al/CuO RMFs is a very important parameter, which has a great influence on stoichiometric reaction. In this paper, the modulation period is empirically determined as 1.2 μm which consists of 0.38 μm-thick Al film and 0.82 μm-thick CuO film. The morphologies of Al/CuO bilayer were investigated by using field emission scanning electron microscopy (FESEM, Sirion2000).

Figure 1 is a SEM image of the Al/CuO bilayer, the top 0.38 μm-thick layer is Al film and the bottom 0.82 μm-thick layer is CuO layer. The anticipated layer structure is clearly visible. The crystal structure of Al/CuO RMFs was analyzed by using X-ray diffraction (XRD, D8ADVANCE) operated at 50 kV and 150 mA. The XRD patterns of the Al/CuO RMFs showed the apparent existence of Al, CuO and a little Cu₄O₃, indicating that significant reduction of the CuO did not occur during deposition. The average grain sizes of Al and CuO films were calculated to be 30.3 nm and 12.3 nm, respectively, using Debye-Scherrer equation. These characteristics, together with the FESEM images, provide clear evidence that distinct Al and CuO films were sputtering deposited in a layered geometry. The exothermic reaction of Al/CuO RFMs was characterized with differential scanning calorimetry (DSC, NETZSCH-STA449C). Integration of the total exothermic peaks gives a heat of reaction equal to 2760 J·g⁻¹. This value is lower than the theoretical heat of reaction 4071 J·g⁻¹, which may be attributed to the emergence of amorphous or nanocrystalline Al₂O₃ during sputtering deposition, or Al/CuO RMFs are not in stoichiometric reaction¹¹.

3. Design and preparation of the igniter

Top view optical image and cross-sectional view schematic diagram of the igniter are shown in Figure 2.

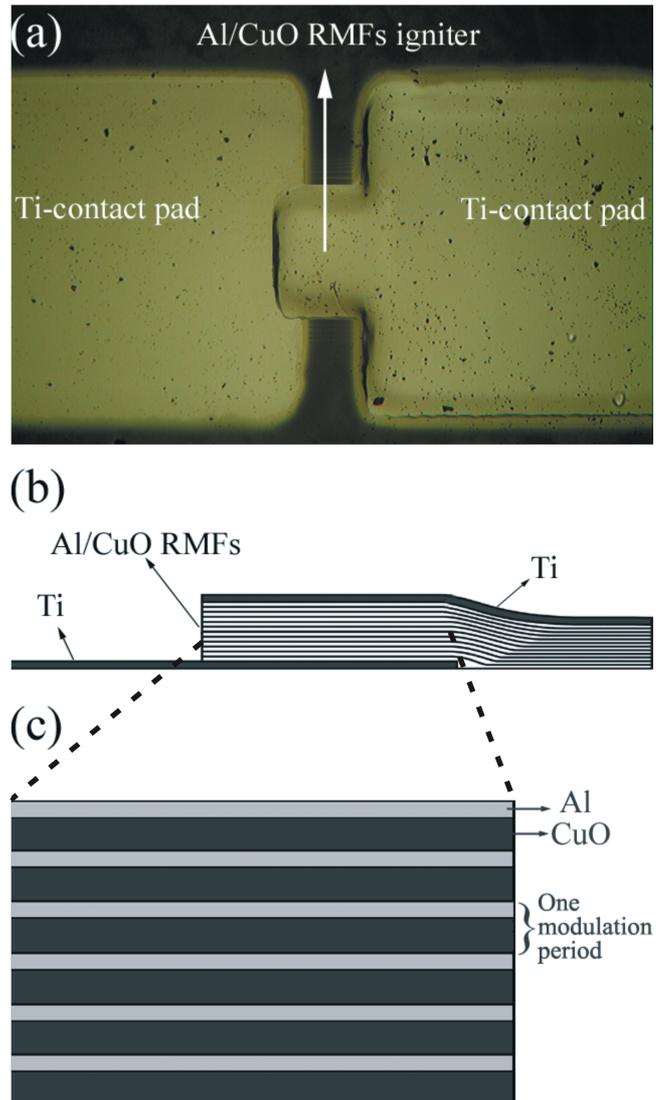


Figure 2 (a) Top-view optical image of the igniter. (b) Cross-section-view schematic diagram of the igniter. (c) Schematic diagram of the Al/CuO RMFs.

The structure consists of essentially three consecutive layers. Ti films act as both electrical heating layers and contact pads while six periods of Al/CuO bilayer separate Ti films. The dimensions of Ti films are of 1000 μm×1000 μm×2 μm, and the dimensions of middle Al/CuO RMFs are of 1000 μm×1000 μm×7.2 μm, each Al/CuO bilayer being 1.2 μm (0.38 μm-thick Al film and 0.82 μm-thick CuO film). The structure is similar to a capacitor. When the contact pads are connected to a voltage source, the source places an electric field across the igniter, and then the current is forced to flow through Al/CuO RMFs equally. The igniters were prepared by using standard MEMS techniques that allowed batch fabrication and high level of integration¹²⁻¹⁴. The main fabrication processes were illustrated in Figure 3.

The substrate was cleaned using acetone, thoroughly rinsed by deionized water, and blow dried in air. Then, the substrate was placed into an oven at 100°C for 30 min for further drying. Reversal photoresist (AZ5200) was spin coated onto the glass substrate and patterned using photolithography through a designed mask. The photoresist was exposed twice to generate a reentrant

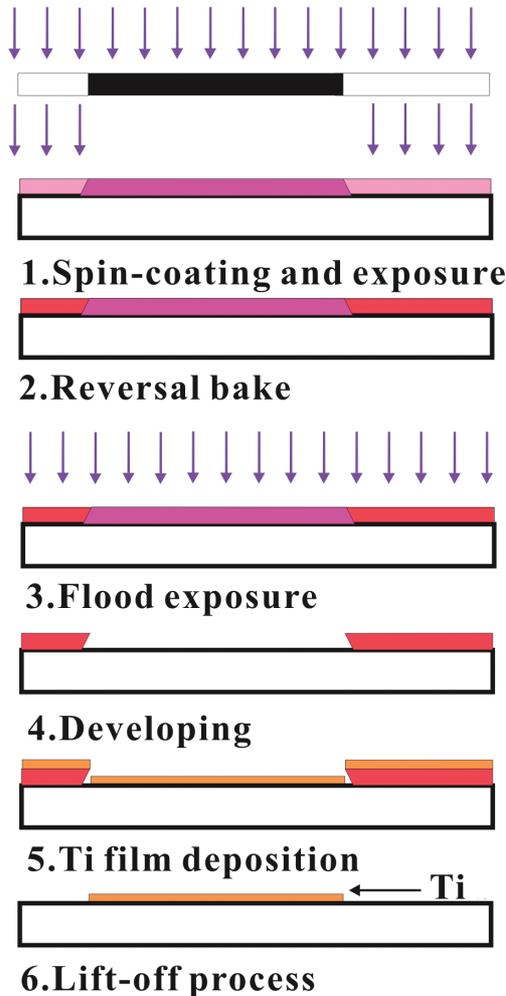


Figure 3 The main fabrication processes of Ti bottom layer. (The Al/CuO RMFs layer and Ti top layer can be obtained by using the same technique.)

profile. A2- μm -thick Ti film was deposited firstly. Then, the as-deposited Ti film was performed into acetone with ultrasonic lift-off for 30sec. Finally, the bottom Ti layer was achieved after deionized water cleaning. The bottom Ti film served as not only the electrical heating layer but also one contact pad of the igniter. According to the same process mentioned above, the middle 7.2- μm -thick Al/CuO RMFs, and the top 2- μm -thick Ti film were made respectively.

4. Open-air electrical explosion testing

Ignition power, ignition delay, energy release and electrical explosion temperature are important parameters for the practical application of energetic igniters. These parameters were obtained by using constant voltage firing set equipped with Multiscillograph (LeCory 44Xs-A) and electrical explosion temperature diagnosis system. Temperature diagnosis system was based on the "double-line atomic emission spectroscopy of copper element". The light emitted from the electrical explosion process was detected by optical fiber and guided into two monochromators (Omni- λ 300) for the diagnosis of explosion temperature. Diagram of the test apparatus was shown in Figure 4.

To perform an ignition test, an initial voltage was loaded

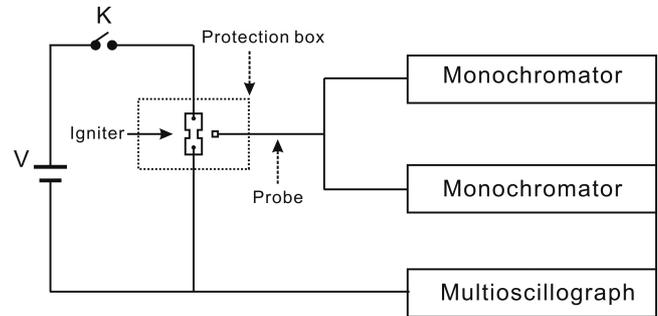


Figure 4 Diagram of the experimental arrangement.

on the igniter. If the first sample did not explode, the second sample will be tested at a relatively high potential. Testing results show that an initial discharge potential of approximate 30V is required to initiate the igniter. This characteristic should be attributed primarily to the specific structure of the igniter. The igniter will not explode until the external potential exceeds the breakdown potential of Al/CuO RMFs. This feature can not only reduce the early fracture probability of the igniter in joule-heating process, but also initiate the exothermic reaction of Al/CuO RMFs effectively. Additionally, the breakdown potential of igniter can be adjusted conveniently by selecting the appropriate thickness and surface areas of the RMFs. Fifty igniters were tested with discharge voltage 30V, and all of them exhibited the analogical results except few small variations of current and discharge time.

Figure 5 shows the voltage-current histories recorded simultaneously. After the electrical breakdown is realized, the current increases sharply and the instantaneous large current is forced to flow through Al/CuO RMFs. The absorbed electric energy quickly heats the igniter until parts of it undergo phase transition from solid into very high conductivity region of plasma. Current fluctuates slightly from triggering to the end, and more than one peak is observed, which may be attributed to the complexity of plasma discharge in the air combined with the exothermic reaction of RMFs. The electrical energy consumption of the igniter discharged in 30V was calculated to be 288mJ by integrating power and duration. For one energetic igniter, the surface area is 1000 μm^2

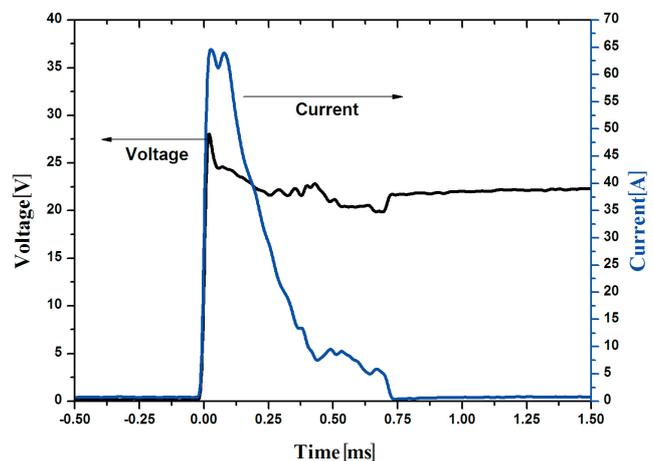


Figure 5 Voltage-current histories versus time.

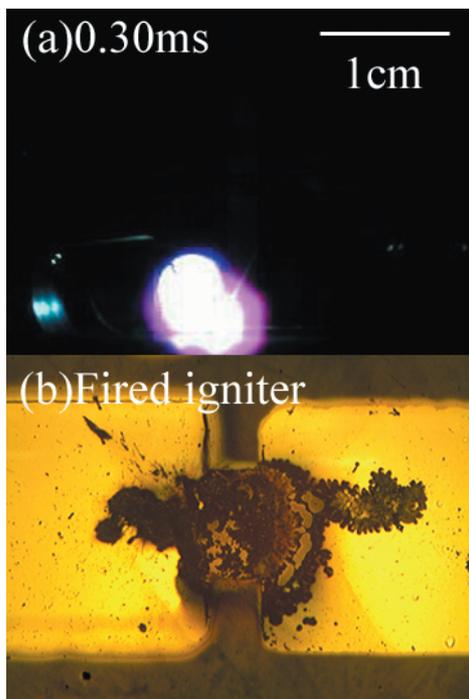


Figure 6 (a) Optical image of the firing igniter. (b) Optical image of the fired igniter.

1000 μm , and the thickness of films deposited is 7.2 μm . Therefore, the mass of the Al/CuO RFMs is estimated to be $3.672 \times 10^{-5}\text{g}$. As a result, the energy output is roughly determined as 101.34mJ. Therefore, the ignition delay time and total released energy of the igniter discharged in 30V are 0.75ms and 389.34mJ, respectively. For one igniter, the energy released by chemical reactions accounts for 26 percent of the total energy, which can be further improved by adjusting the deposition conditions of Al/CuO RFMs.

The electrical explosion process has been observed by using HG-100K high speed photographic apparatus which captures the photographs at 20000 frames per second. Figure 6 shows the optical images of one igniting sample discharged in 30V. For a given shot, a bright flash of light with the length close to 1cm was observed to be accompanied with products ejecting. The fierce explosion phenomenon is attributed to relatively high energy released by both electrical explosion and exothermic reactions. Figure 6(a) is one photograph captured after the igniter has been discharged for 0.3ms. The photograph correlated well with the current histories shown in Figure 5, where the breakdown of igniter happened and fierce explosion phenomenon appeared. Figure 6(b) shows that the main part of fired igniter has been vaporized due to electrical explosion and exothermic reactions.

The explosion temperature of the igniter and its duration were characterized by a systematic explosion temperature diagnosis model based on the "double-line atomic emission spectroscopy of copper element". The fundamental principle of the diagnosis model has been described in detail in another article published in *Journal of Applied Physics*¹⁵⁾. The explosion temperature as a function of time for one typical igniter discharged in 30V is shown in Figure 7.

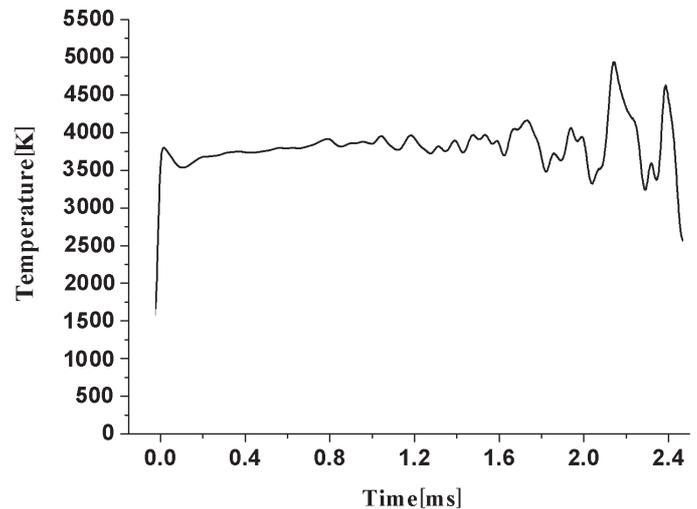


Figure 7 Explosion temperature histories versus its duration.

After the electrical breakdown was realized, the temperature increased initially with time and kept an approximately constant value of 3500K for 2.4ms, then decreased. The curve seemed unstable, and more than one temperature peak was observed due to the complexity of the plasma discharge in the air. The discharging time was 0.75ms, however, the explosion process of igniter was 2.4ms, so it could be deduced that the exothermic reaction of Al/CuO RFMs occurred, resulting in a further explosion process due to the highly energetic impact of the combustion wave and the extremely high local temperature generated. In addition, as the discharge voltage increases, the peak temperature becomes even higher, particularly when the discharge voltage approaches a huge value. The high temperature together with its duration is enough to initiate common pyrotechnic compounds. Furthermore, the ejected high temperature products may be able to initiate the attached energetic materials even if without physical contact.

5. Conclusion

Increasing attention has been focused on RFMs due to their high energy density, energy release rate and good compatibility with MEMS techniques in recent years; however, previous research work almost concerned about self-sustaining combustion reaction which propagated along the RFMs in a relatively slow rate. Here we designed and prepared a new prototype of energetic electric igniter based on Al/CuO RFMs, and electrical explosion performance of the igniter was analyzed empirically. Testing results indicate that the energetic igniter possesses the following advantages: 1) The structure of igniter is similar to a capacitor, which may allow the instantaneous large current to flow through Al/CuO RFMs under the influence of electrical field between two Ti films; 2) The energy released by exothermic reaction is able to help generating high local temperature and shock wave on igniter; 3) The igniter is fabricated by using standard MEMS techniques that allow batch fabrication and high level of integration and reliability. The findings are positive to current research on RFMs,

shedding light on new generation of electric igniters as well. Further work will be structural optimization of the igniter together with establishment of electrical explosion model.

References

- 1) C. Rossi, K. Zhang, D. Estève, P. Alphonse, J. Y. C. Ching, P. Tailhades, and C. Vahlas, *J. Microelectromech. Syst.*, 16, 919–931 (2007).
- 2) E. L. Dreizin, *Prog. Energy Combust. Sci.*, 35, 141–167 (2009).
- 3) F. Shimojo, A. Nakano, R. K. Kalia, and P. Vashishta, *Appl. Phys. Lett.*, 95, 043114 (2009).
- 4) K. J. Blobaum, M. E. Reiss, J. M. Plitzko, and T. P. Weihs, *J. Appl. Phys.*, 94, 2915–2922 (2003).
- 5) K. J. Blobaum, A. J. Wagner, J. M. Plitzko, D. Van Heerden, D. H. Fairbrother, and T. P. Weihs, *Appl. Phys.*, 94, 2923–2929 (2003).
- 6) K. J. Blobaum, D. Van Heerden, A. J. Gavens and T. P. Weihs, *Acta Mater.*, 51, 3871–3884 (2003).
- 7) S. Apperson, R. V. Shende, S. Subramanian, D. Tappmeyer, and S. Gangopadhyay, *Appl. Phys. Lett.*, 91, 243109 (2007).
- 8) K. Sullivan, G. Young, and M.R. Zachariah, *Combust. Flame.*, 156, 302–309 (2009).
- 9) Navid Amini Manesh, Saptarshi Basu, and Ranganathan Kumar, *Combust. Flame.* 157, 476–480 (2010).
- 10) N. Amini Manesh, S. Basu, and R. Kumar, *Energy.*, 36, 1688–1697 (2011).
- 11) S. H. Fischer and M. C. Grubelich, Theoretical energy release of thermites, intermetallics, combustible metals. 24th Int. Pyrotechnics Seminar, Monterey, CA, (1998).
- 12) K. Zhang, C. Rossi, and M. Petrantoni, *Microelectromech. Syst.*, 17, 832–836 (2008).
- 13) K. Zhang, C. Rossi, and G. A. Ardila Rodriguez, *Appl. Phys. Lett.*, 91, 113117 (2007).
- 14) K. Zhang, Y. Yang, E. Y. B. Pun, and R. Shen, *Nanotechnology.*, 21, 235602 (2010).
- 15) P. Zhu, R. Shen, N.N. Fiadosenka, Y. Ye, and Yan Hu, *Appl. Phys Lett.*, 109, 084523 (2011).