Fabrication and Characteristics of Al/PTFE Multilayers and Application in Micro-initiator

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Abstract: In this paper, a micro-initiator was designed and fabricated by integrating Al/PTFE multilayers with a Cu film bridge. The regularity layer structure and interface composition of Al/PTFE multilayers was analysed by transmission electron microscope and X-ray photoelectron spectroscopy, respectively. The heat release reaction in Al/PTFE multilayers can be triggered with reaction temperature of 430 °C, and the overall heat of reaction is 3192 J/g. Al/PTFE multilayers with bilayer thickness of 200 nm was alternately deposited on a Cu film bridge to improve the electric explosion performances. Compared to Cu film bridge, the Al/PTFE /Cu integrated film bridge exhibits improved performances with longer explosion duration time, more violent explosion phenomenon and larger quantities of ejected product particles.

1. Introduction
In recently years, the investigations for nano-energetic materials have triggered extensive interest for their various applications including welding and soldering [1], micro-initiator [2], initiation of secondary reactions [3], and airbags [4], et al. Nano-energetic materials composited of fuel and oxidizer in nanoscale exhibit better performances in many aspects such as rapid energy release, high energy density, and more elements to choice compared to conventional energetic materials. There are many ways to fabricate nano-energetic materials including power mixing, Magnetron sputtering, sol-gel, high-energy ball milling and so on [5]. Among these methods, alternately depositing two or more different multilayers exhibits superiority over others for maximum interfacial contact area, minimum distance among different layers and easy integration with microelectronic and mechanical systems. Integrating nano-multilayers with a metal film bridge initiator provides a fascinating method to improve the electric explosion performances of micro-initiator with compact size, and the performances are tunable upon changing the bilayer thickness and the number of layers. A lot of efforts have been devoted on integrating nano-multilayers with a micro-initiator such as Al/Ni [6, 7], Al/CuO [2, 8], Al/NiO [9], and B/Ti [10, 11]. Compared with these reactive nano-multilayers, Al/PTFE multilayers system could release gas during the reaction, which is beneficial for increasing the performances of micro-initiator. Moreover, Al/PTFE have an ultrahigh energy density of 21 KJ/cm³ [12].

In this paper, a micro-initiator (Al/PTFE /Cu integrated film bridge) was designed and fabricated by integrating Al/PTFE multilayers with a Cu film bridge. The structure and interface composition of the Al/PTFE multilayers was characterized by TEM and XPS analysis, respectively. The electric explosion performance of Cu film and Al/PTFE /Cu integrated film bridge were characterized through
an electric ignition measurement system, and high speed camera was utilized to record the reaction processes.

2. Experimental Section

![Schematic representation of fabrication process for Al/PTFE/Cu integrated film bridge.](image)

Figure 1 shows the manufacture process of Al/PTFE/Cu integrated film bridge. Before deposition, an alumina substrate was cleaned in an ultrasonic bath with acetone, alcohol, and deionized water for 10 min sequentially. Then, the substrate was dried by nitrogen gas and heat treated at 150 °C for 20 min. A Cu target (99.995 wt. %) with a diameter of 100 mm was applied for sputtering. 2 μm thick Cu film was deposited on the alumina substrate, and the sputtering temperature, pressure and power of 100 °C, 0.55 Pa and 100 W, respectively. After that, the Cu film spin-coated with photoresist (PRI-4000A) was patterned with photolithography through a designed mask. Then, the exposed Cu film was directly wet-etched in FeCl₃ solution.

Afterwards, the reversal photoresist (AZ5200) was spin-coated onto the Cu film bridge, which was exposed twice to generate a reentrant profile. Then, Al/PTFE multilayers with bilayer thickness of 200 nm (Al, 50 nm; PTFE, 150 nm) were alternately deposited on the Cu film bridge from Al and PTFE targets by DC magnetron sputtering and DF magnetron sputtering. The sputtering power and pressure for Al film and PTFE film were set at 0.5 Pa, 100 W and 1.5 Pa, 300 W, respectively. The gross thickness of Al/PTFE multilayers was 2 μm. After removing the developed photoresist, two Cu bound pads were deposited on two sides of Al/PTFE multilayers and lead wires were soldered for the connection to the voltage source.

The periodic layer structure and interface composition of the Al/PTFE multilayers were verified by TEM and XPS, respectively. The heat release properties of Al/PTFE multilayers were tested by DSC with a heating rate of 10 °C/min from 50 to 800 °C in flowing nitrogen. The electric explosion performances were characterized through an electric ignition measurement system, and high speed camera was utilized to record the reaction processes.
3. Results and Discussion

![Figure 2](image)

**Figure 2.** Cross-sectional TEM image of Al/PTFE multilayers.

Figure 2 reveals the periodic and regularity layer structure of as-deposited Al/PTFE multilayers. The Al layer and PTFE layer can be clearly defined, and the thickness is controlled. We also can see some surface roughness between Al layer and PTFE layer, which are beneficial for the intermetallic reaction between Al layer and PTFE layer to release energy.

![Figure 3](image)

**Figure 3.** XPS spectra of Al 2p core level (a) and F 1s core level (b).

The interface composition of the Al/PTFE multilayers were investigated by XPS analysis, as shown in Figure 3. The Al 2p core level of the sample with 1 nm PTFE film deposition on Al layer. Two peaks of the Al 2p core level appeared at BE of 72.2 and 75.6 eV. The peak at 72.2 eV of the Al 2p spectra of Al 2p1/2 and Al 2p3/2 is Al film. The peak at 75.6 eV of the Al 2p core level was weight than the Al 2p core level of metallic Al, which could be ascribed to the electronic feature induced by the deposition of PTFE film on Al layer. Meanwhile, compared to PTFE film, the F 1s core level of the sample with 1 nm PTFE film deposition on Al layer shifted as well. These results indicates that the Al/PTFE multilayers is composed of Al layer, Al-F bond (inert layer) and PTFE layer assembling in a metastable system. The Al-F bond existed between Al Layer and PTFE layer is important component to keep high energy density of Al/PTFE multilayers.

Figure 4 reveals the heat release characteristics of Al/PTFE multilayers with bilayer thickness of 200 nm. There are only one major exothermic peaks at 535°C, and the onset reaction temperature is 430 °C. The total reaction heat is calculated at 3192 J/g through integrating the positive exothermic heat flow. The high heat energy release is conductive to enhance the electric explosion performances of Cu film bridge.
Figure 4. DSC trace for Al/PTFE multilayers using a heating rate of 10 °C/min in flowing nitrogen.

For the electric explosion analysis of Cu film bridge and Al/PTFE/Cu integrated film bridge, a discharge capacitor (1800 V, 0.22 μF) was adopted to apply voltage. Figure 5 reveals the experimental results of Voltage-current plots. After applying voltage to film bridge, the resistance and temperature of film bridge rise instantly due to the rapidly increasing current density. When the film bridge begins to vaporize, the voltage stops increasing and reaches the maximal value. Thus, the maximal voltage is regarded as the electron explosion voltage, and the time is regarded as ignition delay. The explosion voltage is measured to be 1575 V for Cu film bridge, and 1655 V for Al/PTFE/Cu integrated film bridge. The ignition delay is 78 ns for Cu film bridge, and 71 ns for Al/PTFE/Cu integrated film bridge. The explosion voltage and ignition delay for two types of film bridges are similar, this might be explained that the room temperature resistivity of Cu film is far less than that of PTFE layers in static, most of the current is expected to pass through the Cu film bridge.

Figure 5. Voltage and current plots of electron explosion processes: (a) Cu film bridge, (b) Al/PTFE/Cu integrated film bridge

As shown in Figure 6, a high-speed camera was utilized to record the reaction dynamic processes of film bridges during the voltage-current tests. The interval between adjoining pictures is 50 μs. The specific flame structures in various electron explosion stages were obviously observed. We can observe a fiercer electric explosion phenomenon accompanied with larger quantities of ejected product particles occurred on the Al/PTFE/Cu film bridge. The explosion duration time is about 300 μs for Cu film bridge and 350 μs for Al/PTFE/Cu film bridge. It can be definitely concluded that the self-propagation exothermic reaction of Al/PTFE multilayers is triggered during the electric explosion process, and the reaction can improve the explosion performances of Cu film bridge substantially.
Figure 6. Explosion processes of Cu film bridge (a) and Al/PTFE/Cu integrated film bridge (b).

4. Conclusion
In this work, an integrated film initiator is realized by integrating Al/PTFE multilayers with a Cu film bridge on alumina substrate. From the comparison of the electric explosion characteristics of the Cu film bridge and Al/PTFE/Cu integrated film bridge, it can be concluded that the combination of Al/PTFE multilayers with Cu film bridge can improve the electric explosion performances substantially. In addition, the electric explosion performances of Al/PTFE/Cu integrated film bridge could be tuned by simply altering the films thickness, and the Al/PTFE/Cu integrating film bridge can be fabricated by standard microfabrication technique that allow batch fabrication and high level of integration.

5. References
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