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基于Al/CuO_x复合薄膜半导体桥间隙点火性能研究

倪德彬，于国强，史胜楠，徐 栋，陈利魁，朱雅红，王培勇

(陕西应用物理化学研究所，陕西 西安 710061)

摘　要： 为了提升半导体桥(SCB)的点火能力，尤其是点燃钝感药剂的能力，采用磁控溅射技术将Al/CuO_x复合薄膜与半导体桥相融合，形成含能点火器件，并研究了该含能点火器件的发火感度和点火能力。采用扫描电子显微镜(SEM)、X-射线能谱仪(EDS)、X-射线衍射仪(XRD)研究了Al/CuO_x复合薄膜的微观形貌和组成。结果表明，在溅射过程中氧化铜薄膜主要以黑铜矿($Cu_2^{1+}Cu_2^{1+}O_3$)形式存在；复合薄膜中Al、Cu、O三种元素质量分数分别为28.8%，32.5%和38.7%，且Al与Cu原子比例接近于理论比1∶1；差示扫描量热仪(DSC)显示Al/CuO_x复合薄膜放热量约为2175.4 J·g^{-1}；高速摄影技术测试Al/CuO_x复合薄膜的燃烧速率约为3.0 m·s^{-1}；兰利法测得该含能点火器件50%发火电压为8.45 V，99.9%发火电压为12.39 V。点火能力实验表明，在点火间隙为4 mm时，该含能器件能够点燃钝感点火药硼-硝酸钾(B/KNO_3)药片，显著提升了半导体桥的点火能力。

关键词： Al/CuO_x；复合薄膜；半导体桥(SCB)；间隙点火

中图分类号： TJ55；TJ45　　**文献标志码：** A　　**DOI：**10.11943/CJEM2018167

1 引言

半导体桥点火器件具有作用时间快、发火能量低和安全性高等优点，但是其输出能量较小，发火持续时间短，点火能力依赖于输入能量，当半导体桥与药剂之间存在间隙时，则不能可靠点火[1]。采用微电子技术将含能复合薄膜与半导体桥芯片进行集成，形成含能半导体桥点火器件[2-4]，可以提高半导体桥点火器件的点火能力。

含能复合薄膜是将两种或两种以上可反应的材料(金属/金属氧化物，金属/金属、金属/有机材料)按照一定的厚度和周期性交替沉积的多层薄膜材料，在较小的热脉冲或者电能激励下，能够引发自持放热反应并释放大量热量[5-6]。常见的含能复合薄膜有Al/CuO[7]，Al/MoO_3[8]，Al/NiO[9]，Al/Ni[10-11]等，其中Al/CuO复合薄膜具有较高放热量备受关注，国内外针对Al/CuO复合薄膜开展了大量研究，包括制备方法[12-13]、反应机理[14]、反应模型[15-17]、电爆性能[18-22]等。研究表明Al/CuO复合薄膜在发生自持燃烧反应时，具有更好的反应活性和更好的爆发温度，能够提升半导体桥点火器件的点火能力。李勇等[23]制备的基于Al/CuO复合薄膜纳米含能半导体桥在激励电压45 V，点火间隙为2 mm时，能够点燃硫氰酸铅/氯酸钾点火药。Zilong Zhen等[24]制备的基于Al/Co_3O_4半导体桥含能点火器件，激励电压为30 V，点火间隙为3.7 mm时，能够点燃锆/四氧化三铅点火药，极大提高了半导体桥的点火能力。但是，这些含能点火器件的工作电压均为几十伏，不利于点火系统的智能化、微型化发展。

为此，本研究采用磁控溅射技术制备了基于Al/CuO_x复合薄膜的半导体桥含能器件，并测试了该含能器件的发火感度、电爆性能和点火能力。

2 实验部分

2.1 试剂与仪器

Al靶材，直径10.8 mm，厚度7 mm，纯度99.99%，CuO靶材，直径10.8 mm，厚度7 mm，纯度99.99%，均为江西科泰新材料有限公司；丙酮，乙醇均为分析纯，上海国药集团；去离子水，自制；光刻胶，

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作者简介： 倪德彬(1987-)，男，高级工程师，主要从事纳米铝热剂研究，e-mail：nidebin@hust.edu.cn

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扫描电镜（SEM），Hitachi，S-4800；X-射线衍射仪（XRD），Bruker，D8 Advance；差示扫描量热仪（DSC），Netzsch，STA449C；高速摄影仪，Redlake HG-100K；高速数字存储示波器，LeCroy WaveSurfer44Xs。

2.2 实验过程

首先将半导体桥电极塞依次置于丙酮、乙醇、去离子水中超声清洗 10 min，用氮气吹干，放置于溅射模具中，准备溅射。当腔室气压达到 2.6×10^{-4} Pa 开始溅射，Ar 流量为 20 $mL\cdot cm^{-3}$，冷却水温度为 20 ℃，旋转速率 50 $r\cdot min^{-1}$。Al 靶材溅射参数为：直流溅射，溅射功率：250 W，溅射速率为：15 $nm\cdot min^{-1}$，100 nm/层；CuO 靶材溅射参数：射频溅射，溅射功率：200 W，溅射速率：10 $nm\cdot min^{-1}$，200 nm/层。Al/CuO_x 复合薄膜总厚度 3 μm，溅射过程由电脑控制，依次交替沉积 Al、CuO 薄膜，直至完成整个溅射过程。

样品制备及热分析测试：将光刻胶均匀旋涂在硅片表面，110 ℃下烘干 30 min，然后将 Al/CuO_x 复合薄膜溅射在光刻胶表面。溅射结束后，将硅片在丙酮中浸泡 3 次，过滤，烘干，得到片状薄膜。测试温度为室温~1000 ℃，升温速率为 20 $℃\cdot min^{-1}$，氮气保护，氮气流量为 10 $mL\cdot min^{-1}$。

3 结果与讨论

3.1 Al/CuOx 复合薄膜的形貌分析

采用 SEM 观察了 Al/CuO_x 复合薄膜的横截面形貌，结果见图 1。从图 1 可以看到 Al 层与 CuO_x 层之间连接紧密，无断层，且与基底结合良好。通过 EDS 分析（图 2）可以看到，复合薄膜中包含了 Al、Cu、O 三种元素，三种元素的质量分数分别为 28.8%，32.5% 和 38.7%，且 Al 与 Cu 的原子比例接近于理论比 1∶1。

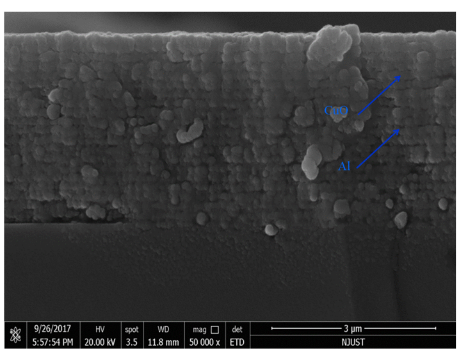

图 1 Al/CuO_x 复合薄膜的横截面 SEM 形貌图

Fig.1 SEM image of cross-section morphology for Al/CuO_x multilayer films

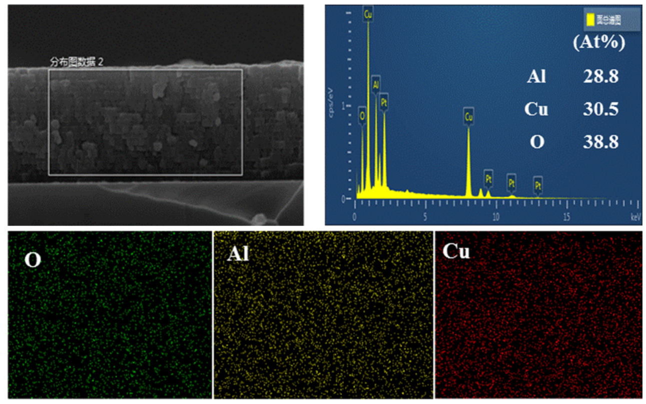

图 2 Al/CuO_x 复合薄膜的 EDS 谱图

Fig.2 EDS images of Al/CuO_x multilayer films

3.2 Al/CuO_x 复合薄膜的热分析研究

采用 DSC-TG 研究了 Al/CuO_x 复合薄膜的放热行为，结果如图 3 所示。从图 3 可知，整个放热过程有两个明显的放热峰。第一个小放热峰的起始反应温度为 312.9 ℃，最大放热量是温度为 336.7 ℃，放热量为 33.4 J。该峰的出现，可能是 Al-Cu-O 界面在加热过程中结晶或者是低温下氧化还原反应释放的热量造成的，由于该峰的放热量很小，对整个放热过程的贡献可以忽略。第二个放热峰的起始反应温度为 570.3 ℃，最大放热量时温度为 600.5 ℃，放热量达到了 2142 $J\cdot g^{-1}$。由此可以推断出，在 Al（熔点：660.4 ℃）熔化之前，Al/CuO_x 复合薄膜就已经发生了反应，说明该反应经历的是固-固反应过程[25-26]。虽然 Al/CuO_x 复合薄膜的放热量比理论放热量（4077 $J\cdot g^{-1}$）低，但并不影响其点火能力。整个放热过程，薄膜总质量基本保持不变，说明制备薄膜过程中的光刻胶完全被去除。

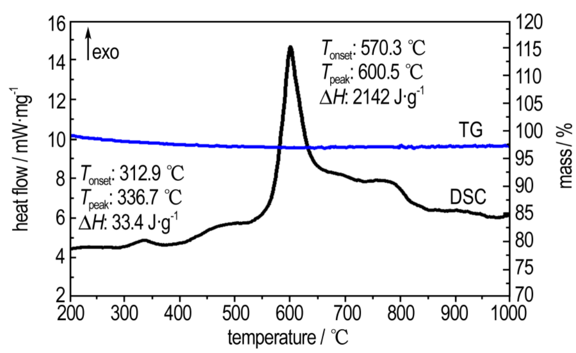

图 3 Al/CuO_x 复合薄膜的 DSC-TG 曲线

Fig.3 DSC-TG curves of Al/CuO_x multilayer films

Al/CuO_x 复合薄膜的 XRD 测试结果如图 4 所示。由图 4 可知，反应前（红色曲线）38.47°，44.74°，65.13°，78.22°衍射峰分别是 Al（JCPDS04-0787）的

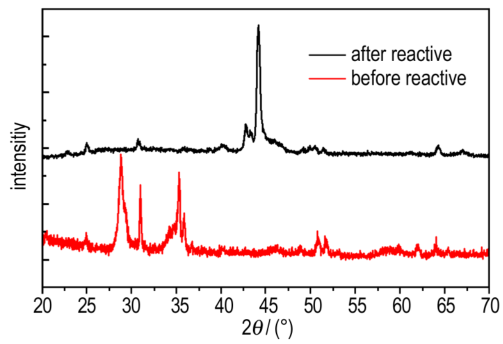

图4 Al/CuO_x复合薄膜反应前后的XRD图谱

Fig.4 XRD patterns of Al/CuO_x multilayer films before and after reaction

(1 1 1),(2 0 0),(2 2 0),(3 1 1)面的衍射峰;35.64°,36.34°,58.31°,63.93°,65.03°和75.51°衍射峰,分别来源于Cu_4O_3(JCPDS49-1830)的(2 1 1),(0 0 4),(2 2 4),(4 0 0),(4 2 2)面的衍射峰。溅射沉积得到的氧化铜是黑铜矿,其化学结构式为$Cu_2^{1+}Cu_2^{1+}O_3$,含铜量比CuO稍高,溅射过程中有一部分氧被消耗,也造成了溅射薄膜中的氧含量比预计的CuO低[12]。Al/CuO_x复合薄膜反应后产物的XRD图谱(黑色曲线)显示,43.29°,50.43°和74.13°来源于Cu(JCPDS04-0836)(1 1 1),(2 0 0)和(2 2 0)面的衍射峰。产物Al_2O_3是无定形态材料,在XRD中未能检测出来。

3.3 Al/CuO_x复合薄膜的半导体桥发火器件发火感度

采用兰利法[27-28]测试了Al/CuO_x复合薄膜半导体桥点火器件的发火电压感度,以100 μF电容为激励电源。预先估计发火电压的上限x_u为20 V,下限x_l为0 V,连续实验17发,经计算机处理得到该点火器件的50%发火电压为8.45 V,99.9%发火电压为12.39 V,由表1可知较低的发火电压,更有利于该含能发火器件的在微点火系统中的应用。

表1 兰利法测试Al/CuO_x薄膜的电压发火感度

Table 1 The firing-voltage sensitivity of Al/CuO_x multilayer film measured by Lanley's method

i	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
x_i/ V	10	5	7.5	13.75	10.62	7.81	9.22	11.48	10.35	9.08	4.54	6.81	8.58	7.69	6.12	6.91	7.74
N_i	1	0	0	1	1	0	0	1	1	1	0	0	1	1	0	0	1
x'_j	x_l	x_1	x_u	x_3	x_2	x_5	x_4	x_7	x_6	x_l	x_{10}	x_9	x_{12}	x_{11}	x_{14}	x_{13}	

Note: i is experimental quantity. x_j is experimental stimulus quantity. 1 means ignition. 0 means non-ignition. x_u is the upper limit of ignition. x_l is lower limit of ignition. x'_j is the value of another stimulus quantity except x_j.

3.4 Al/CuO_x复合薄膜的燃烧速率

将厚度为3 μm的Al/CuO_x复合薄膜溅射在聚酰亚胺基底上进行燃烧速率测试。根据图5所示,采用高速摄影和激光点火技术测高速摄影拍照速率为10000帧/s,计算得到,Al/CuO_x复合薄膜的燃烧速率约为3.0 m·s^{-1},燃烧速率较低。

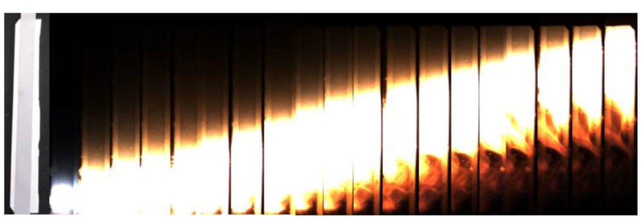

图5 用高速摄影测得Al/CuO_x复合薄膜的燃烧速率

Fig.5 The burning rate of Al/CuO_x multilayer films obtained with high speed cameral

3.5 Al/CuO_x复合薄膜半导体桥发火器件的电爆性能

电爆实验采用储能电容放电仪,电容为100 μF,充电电压为12.39 V,高速数字存储示波器记录点火过程电压和电流变化。Al/CuO_x复合薄膜电爆过程电压-电流变化曲线,结果见图6。由图6可以看到含能薄膜半导体桥发火电压曲线呈现出先上升后降低的过程,没有出现典型半导体桥电爆过程中的升温、融化、汽化和生成等离子四个阶段,表明整个作用过程中电流仅经过半导体桥,半导体桥将热量传递给Al/CuO_x复合薄膜,引起复合薄膜发生反应,点火机制为热点火[23]。Al/CuO_x复合薄膜半导体点火器件的临界发火时间约为3.89 μs,临界发火能量为29.2 mJ,作用总时间约为231 μs。临界发火时间变长、临界发火能量变大是由于Al/CuO_x复合薄膜在点火时带走了一部分半导体桥热量造成的。Al/CuO_x复合薄膜半导体含能器件作用总时间显著长与半导体桥作用总时间,可以有效提升点火能力。

采用高速摄影仪记录整个点火过程,拍照速率为25000 fps,结果见图7。从图7中可以看到,在40 μs时,Al/CuO_x复合薄膜已经被完全点燃,且火焰明亮,并且稳定向前传播,同时伴随大量炙热粒子向外散射,火焰面积在360 μs时达到最大,火焰长度约4 cm;随

后火焰有效面积在逐渐减小，亮度逐渐变暗，直到800 μs时基本消失。如此长火焰传播距离和大量固体离子提高了Al/CuO$_x$复合薄膜半导体桥点火器件的点火能力。

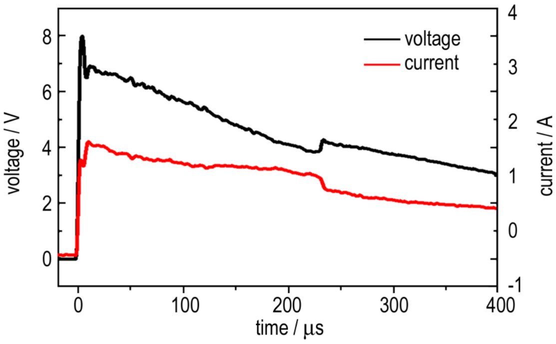

图6　基于Al/CuO$_x$复合薄膜半导体桥的电流-电压曲线

Fig.6　The voltage-current curves of SCB based on the Al/CuO$_x$ multilayer films

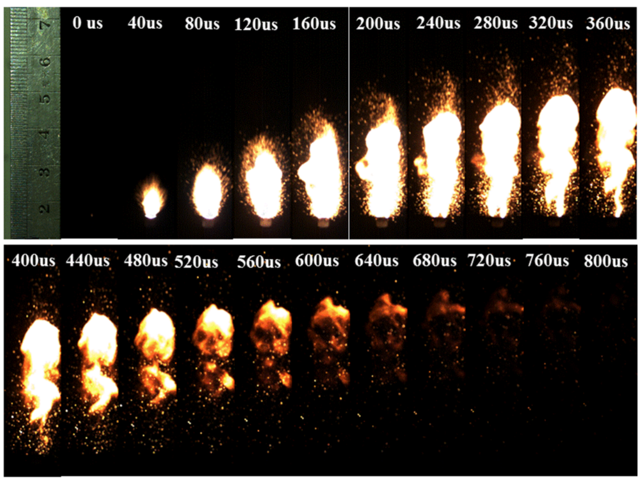

图7　Al/CuO$_x$复合薄膜发火器件燃烧过程的高速摄影图

Fig.7　The high-speed photography images of Al/CuO$_x$ multilayer films during the combustion process

3.6　Al/CuO$_x$复合薄膜点火能力

为了考察Al/CuO$_x$复合薄膜的点火能力，开展了半导体桥含能复合薄膜点燃钝感点火药B/KNO$_3$药片实验。实验条件为：B/KNO$_3$药片药量为50 mg，密度为2 g·cm^{-3}，电容充电电压12.39 V。首先将B/KNO$_3$药片紧贴在Al/CuO$_x$复合薄膜表面，装在点火模具中，等待点火。闭合电路后，B/KNO$_3$药片被点燃，测试装置如图8所示。调节B/KNO$_3$药片与Al/CuO$_x$薄膜的距离，考察在一定间隙条件下Al/CuO$_x$复合薄膜的点火能力，结果见表2，不同间隙距离进行点火实验5次，从表2可以看到，当间隙距离大于4 mm时，5发实验均不能将B/KNO$_3$药片点燃。对照实验显示在相同发火条件下，半导体桥（不含Al/CuO$_x$复合薄膜）不能点燃B/KNO$_3$，说明Al/CuO$_x$复合薄膜能够显著提高半导体桥的点火能力。

表2　Al/CuO$_x$复合薄膜的间隙点火能力实验结果

Table 2　The experimental results of gap ignition ability of Al/CuO$_x$ multilayer film

gap distance / mm	0	1.03	2.10	3.13	4.07	5.04
number of experiments	5	5	5	5	5	5
number of ignition	5	5	5	5	5	0

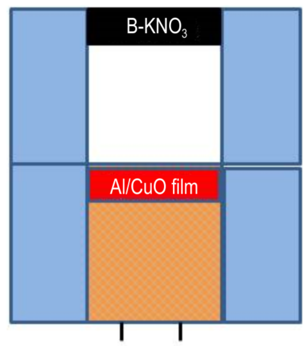

图8　Al/CuO$_x$复合薄膜点火能力测试装置结构示意图

Fig.8　Schematic diagram of setup structure used to test the ignition ability of Al/CuO$_x$ multilayer film

4　结论

通过磁控溅射方法制备了Al/CuO$_x$复合薄膜半导体桥发火器，获得了复合薄膜的热力学性能和电爆性能：

（1）Al/CuO$_x$复合薄膜放热量达到2175.4 J·g^{-1}，燃烧速率约3 m·s^{-1}。复合含能点火器件的临界点火时间3.89 μs，临界发火能量为29.2 mJ，作用总时间约为231 μs，燃烧生成的火焰长度超过了4 cm，燃烧时间持续约8 ms。

（2）在点火间隙为4 mm的情况下，能够点燃密度为2 g·cm^{-3}的钝感点火药B/KNO$_3$药片，显著的提升了半导体桥的点火能力，有望应用在间隙点火系统、微点火芯片等点火装置中提高点火效率和点火安全性。

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The Gap Ignition Performances of Semiconductor Bridge Based on Al/CuO$_x$ Multilayer Films

NI De-bin, YU Guo-qiang, SHI Sheng-nan, XU Dong, CHEN Li-kui, ZHU Ya-hong, WANG Pei-yong

(*Shanxi Applied Physics and Chemistry Research Institute, Xi'an* 710061, *China*)

Abstract: To improve the ignition ability of semiconductor bridge (SCB), especially the ability to ignite insensitive compositions, Al/CuO$_x$ multilayer film was fused and combined with semiconductor bridge by magnetron sputtering technology to form an energetic ignition device and the ignition sensitivity and ignition ability of the energetic ignition device were studied. The micro morphology and composition of Al/CuO$_x$ multilayer films were studied by scanning electron microscopy (SEM), X-ray energy dispersive spectrometer (EDS) and X-ray diffractometer (XRD). Results show that the copper oxide film mainly exists in the form of black copper ore (Cu_2^{1+} $Cu_2^{1+}O_3$); the mass fractions of Al, Cu and O in the multilayer film are 28.8%, 32.5% and 38.7% respectively, and the ratio of Al to Cu atom is close to the theoretical ratio of 1∶1; the results obtained by differential scanning calorimeter(DSC) show that the quantity of heat release of Al/CuOx multilayer film is about 2175.4 $J \cdot g^{-1}$. The burning rate of Al/CuO$_x$ multilayer film is measured by high-speed photography is about 3.0 $m \cdot s^{-1}$. The 50% ignition voltage of the energetic ignition device measured by Lanley's method is 8.45 V and 99.9% ignition voltage is 12.39 V. The ignition ability experiment shows that when the ignition gap is 4 mm, the energetic device can ignite the insensitive ignition composition of B/KNO_3 tablets, which significantly improves the ignition ability of the semiconductor bridge.

Key words: Al/CuO$_x$; multilayer films; semiconductor bridge(SCB); gap ignition

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