



Effect of aluminum micro- and nanoparticles on ignition and combustion properties of energetic composites for interfacial bonding of metallic substrates

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ABSTRACT

In this study, the effect of micro- and nanoscale energetic materials in the formulation of aluminum microparticles (Al MPs)/aluminum nanoparticles (Al NPs)/iron oxide nanoparticles (Fe_2O_3 NPs) as a heat energy source for melting solder microparticles (SAC 305 MPs) on the interfacial bonding properties of Cu metallic substrates, is investigated. The optimized mixing ratio is Al MP:Al NP: Fe_2O_3 NP = 30:30:40 wt%, which generates a maximum total exothermic energy of $\sim 2.0 \text{ kJ g}^{-1}$. The presence of Al NPs is essential to make stable ignition and initiation of Al MPs, which enable to attain relatively long combustion duration. The use of highly reactive Al NPs/ Fe_2O_3 NPs can improve the aluminothermic reaction, while the addition of Al MPs to the Al NPs/ Fe_2O_3 NPs is also required to maintain their high thermal energy for a longer duration. An energetic material (EM) layer composed of Al MP/Al NP/ Fe_2O_3 NP composites is employed as a heat source between solder material (SM) layers composed of SAC305 MPs. The SM/EM/SM multilayer pellets are assembled and ignited between interfacial Cu substrates for bonding. Thus, interfacial bonding between the Cu substrates is successfully achieved, and the resulting maximum mechanical strength for the bonded Cu substrates using the SM/EM/SM multilayer pellets increases by $\sim 40\%$ compared to that when using a pure SM layer pellet. Hence, EM layers can act as both an effective heat energy generation source and a mechanical reinforcing medium, while the interfacial bonding process using SM/EM/SM multilayer pellets demonstrated herein provides an easy and versatile means of welding and joining for industrial applications.

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1. Introduction

Several interfacial bonding processes, such as anodic bonding, direct bonding, solder bonding, and adhesive bonding, have been developed [1–7]. For these processes, the interfacial bonding properties (including the bonding strength and airtightness) are highly dependent on the bonding conditions, including the temperature, pressure, and type of substrate [8–15]. In particular, soldering with molten solder at high temperatures inherently creates compounds between the solder and substrate, which can affect the interfacial bonding properties [16–18]. High-temperature conditions can release residual stresses in multi-layered bonding substrates, while they can simultaneously result in a weakening of the

interfacial bonding strength [19,20]. When low-pressure conditions are applied, the actual contact area between the interfacial bonding parts can be reduced, and thus, this can result in a significant decrease in the interfacial bonding strength. Therefore, the interfacial bonding process generally requires a closed system, in which temperature- and pressure-controlled conditions are applied to both the solder and interfacial substrates [21,22].

Generally, a heat source, such as a furnace, welding torch, or microwave plasma burner is employed to preheat and coalesce the solder and substrate. For example, several bonding processes have been developed, including eutectic bonding, activated bonding, cast bonding and solid-state joining, which rely on the use of a furnace to supply heat energy. However, these are subject to a range of restrictions on the material selection and bonding structures. Simultaneously, to suppress the formation of cracks, defects, and residual stress for interfacial bonding, the precise control of the operating conditions, such as the preheating temperature, dwell time, and cooling rate, is also required [23]. However, relatively high

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temperatures and pressures, applied over an extended duration, are usually adopted for interfacial bonding processes, so that the structural changes in the substrates to be bonded occur due to thermal deformation, surface oxidation, and the formation of intermetallic compounds between the solder and substrate. This can eventually result in reducing the mechanical strength while adversely affecting the physical properties of the bonded substrates [24–26].

Energetic materials (EMs) are composites consisting of fuel and an oxidizer. They store chemical energy, which can be rapidly turned into thermal energy via a homogeneous exothermic reaction when ignited by an external energy input [27–39]. The heat energy generated by the ignition of highly reactive EMs can be used to bond interfacial substrates by melting the solder inserted between those substrates. The use of EMs as a heat source for interfacial bonding offers several advantages including, (i) there is no need to heat either the solder or the interfacial substrates, (ii) additional, possibly complex, heating equipment is not necessary, and (iii) the simple and versatile interfacial bonding process can be implemented for a range of interfacial structures.

In the present study, we systematically investigated the effects of using EMs as a heat energy source on the interfacial bonding properties of metallic substrates. Solder material (SM) and EM were pelletized with multiple layers, and were then inserted between the metallic substrates to enable interfacial bonding. Specifically, micro- and nanoscale aluminum (Al) was used as the fuel, while iron oxide (Fe_2O_3) was used as a weak oxidizer. SAC 305 was used as a SM. Various SM/EM/SM multilayer pellets were fabricated and then tested to determine their efficacy at bonding the interfacial metallic substrates. The ignition, combustion, and physical properties of the SM/EM/SM multilayer pellets and the mechanical properties of the bonded Cu metallic substrates were systematically examined using several different techniques, including a high-speed camera, pressure-cell tester (PCT), differential scanning calorimetry (DSC), scanning electron microscopy (SEM) and universal tensile-strength testing.

2. Experimental

In the present study, a mixture of Al nanoparticles (Al NPs; Nano Technology Co., Ltd., Korea) with an average diameter of around 80 nm, and Al microparticles (Al MPs; Player Metal Co., Ltd., Korea) with an average diameter of around 8 μm , was used as the fuel source. As the oxidizer, we used Fe_2O_3 NPs (Sigma-Aldrich, Korea) with an average diameter of around 90 nm. SAC 305 powder (Sn: 96.5 wt%, Ag: 3 wt%, Cu: 0.5 wt%; Senju Metal Industry Co., Ltd., Japan) with an average diameter of around 70 μm was used as the SM.

Figure 1 is a schematic diagram of the fabrication of EMs composed of Al NPs, Al MPs, and Fe_2O_3 NPs. The SM/EM/SM multilayer pellets were formed by a subsequent die-compaction process. The fabricated SM/EM/SM multilayer pellets were inserted between the Cu substrates, and then the interfacial bonding process was implemented by igniting the multilayer pellets using a tungsten hot-wire under an applied voltage of around 4 V and a current of around 2 A.

Briefly, EM powders composed of Al NPs/Al MPs/ Fe_2O_3 NPs were dispersed in an ethanol solution, and were subsequently mixed by ultrasonication (ultrasonic power ≈ 170 W, ultrasonic frequency ≈ 40 kHz) for 30 min with a mixing ratio of Al NP:Al MP: Fe_2O_3 NP = 30:30:40 wt%, which was empirically determined as the optimum combustion condition for exothermic reactions, as shown in Fig. S1 in Supporting information. The EM powders were finally dried in a convection oven at 80 $^\circ\text{C}$ for 30 min. EM (i.e., Al NP/Al MP/ Fe_2O_3 NP composites) and SM (i.e., SAC305 MP, Sn: 96.5%, Ag: 3%, Cu: 0.5%) powders were sequentially poured into a mold in the die-compaction process, and were then pelletized into

disks approximately 7 mm in diameter and around 1 mm in height under a pressure of 300 MPa.

Differential scanning calorimetry (DSC; Setaram, LABSYS evo, France) measurements of the EM powders were performed with a temperature increase rate of 10 $^\circ\text{C min}^{-1}$ at 30–1000 $^\circ\text{C}$ under an air atmosphere. The physical structure of the fabricated SM/EM/SM multilayer pellets was observed using scanning electron microscopy (SEM) (Carl Zeiss, Supra 40VP, Germany) operating at around 20 kV. The elemental mappings of interfacially bonded Cu substrates and EM powders were observed using a field emission scanning electron microscope equipped with an energy dispersive X-ray analyzer (EDS, INCA, Oxford Instruments, Abingdon, UK). The X-ray phase analyses of EM powders were performed using X-ray diffractometry (XRD, Empyrean series2, PANalytical, Almero, Netherlands) with Cu $K\alpha$ radiation in the range of 20–90 $^\circ$. A high-speed camera (Photron, FASTCAM SA3 120K, Japan) was employed to observe the ignition and combustion reaction of the EM powders and pellets fabricated in the present study. The high-speed camera had a maximum frame rate of 1,200,000 fps, a minimum frame rate of 60 fps, a 17.4 mm \times 17.4 mm CMOS image sensor, a pixel size of 17 $\mu\text{m} \times 17 \mu\text{m}$, and a AC operating voltage 100–240 V, drawing a current of 60 A. The PCT was used to analyze the pressure trace and pressurization rate by igniting the composite powders in a sealed pressure cell (around 13 ml). Briefly, EM powder (around 16 mg) was placed in the sealed pressure cell, and then ignited by using a tungsten hot-wire igniter, which was operated at around 4 V, drawing a current of 2 A. The explosion pressure generated by the hot-wire ignition was measured by a piezoelectric pressure sensor (113A03, PCB Piezotronics) attached to the pressure cell. Simultaneously, the detected pressure signal was amplified and transformed into a voltage signal through a combination of an in-line charge amplifier (422E11, PCB Piezotronics) and signal conditioner (480C02, PCB Piezotronics). Finally, the signal was captured and recorded using a digital oscilloscope (TDS 2012B, Tektronix). We calculated the pressurization rate by measuring the maximum pressure with the rise time when the composite powder was ignited. After performing the interfacial bonding process, the mechanical bonding strength of the Cu substrates was measured using a universal tensile strength tester (LRXPlus 5 kN, Lloyd Instruments Ltd., UK) with a strain rate of around 10 mm min^{-1} .

3. Results and discussion

Figure 2 shows the SEM images and energy dispersive X-ray spectroscopy (EDS) elemental mappings for the Al NP/Al MP/ Fe_2O_3 NP composite powder used in the present study. The Al NPs and Al MPs, used as a fuel, were both spherical with average diameters of around 100 nm (Fig. 2a) and 8 μm (Fig. 2b), respectively. The Fe_2O_3 NPs, used as an oxidizer, were also spherical with an average diameter of around 90 nm (Fig. 2c). After mixing the Al NPs/Al MPs/ Fe_2O_3 NPs, the resulting EM composite powders were observed using SEM and EDS, as shown in Fig. 2d–f. It was found that the Al NPs/ Fe_2O_3 NPs were homogeneously distributed and attached to the surfaces of the Al MPs.

We systematically examined the effects of Al NPs and Al MPs on the ignition and combustion properties of EMs. Figure 3 shows schematics and still images of the ignition and combustion reactions of the EMs addressed in the present study. First, Al MP/ Fe_2O_3 NP-based EMs without any Al NPs failed to ignite when using a tungsten hot-wire, even when heated to around 800 $^\circ\text{C}$ with an applied voltage of around 12 V [40,41]. This suggests that a high thermal energy input is required to directly ignite Al MPs, which have a relatively low reactivity due to their large primary size. However, when Al NPs were present in EMs (i.e., the Al NP/ Fe_2O_3 NP and Al NP/Al MP/ Fe_2O_3 NP composites), the ignition and subsequent combustion occurred stably using the same tungsten hot-wire heated

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