Fabrication and Characterization of Nanoscale Heating Sources ("Nanoheaters") for Nanomanufacturing

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ABSTRACT

Nanoheater systems based on the exothermic reaction between aluminum and nickel were developed. Multilayered and powder-based heterostructures were fabricated and characterized, before and after ignition experiments, for their geometries and structures as well for the ignition and exothermic transformation kinetics. A novel ultrasonic powder consolidation (UPC) technique[1] was employed to produce ignitable Al/Ni compacts from elemental powders. Oxide free, fully dense Al matrix and intimate Al/Ni interfaces were obtained. Consolidates were successfully ignited and the reaction self-propagated throughout the compact resulting in the formation of NiAl phase. The ignition characteristics of multilayered structures and sequential thermal generation and conduction were investigated by IR thermal camera measurements.

Keywords: thermal manufacturing, heat sources, nanostructures

1. INTRODUCTION

Active use of heat to alter the geometry, structure and properties of solids is central to material removal, deposition, joining, shaping, and transformation processes in macroscale manufacturing. However as the nanoscale is approached, traditional heat sources are not always compatible because of fundamental technical limits. Spatial and temporal dimensions of nanoscale structures and phenomena pose a difficulty in application of traditional heat sources. The characteristic lengths and times of heat transfer approaches in macroscale are incompatible with the process requirements at nanoscale. Also there are difficulties in controlling temperature distribution and duration. Therefore, there is an outstanding need for new disruptive heat sources, enabling fine local selectivity and time exposure control in nanoscale thermal processing. Such sources could revolutionize manufacturing as well as on-board thermal actuation and autonomous powering of miniature devices and systems.

This paper addresses research in manufacture and operation of nanoheater systems based on the exothermic reaction[2] between aluminum and nickel. Multilayered nanoheaters comprised of alternating thin layers of Al and Ni (ranging around 20-100 nm each) are fabricated with physical vapor deposition, while powder-based Al-Ni composites are developed using ultrasonic joining. These nanoheaters, which can be either embedded within the bulk or on the surface of another material, can be ignited by an external ignition source. Reaction enthalpies released as a result, act as localized heat fluxes transferred to desired areas, primarily by conduction. The spatial and temporal profile of this heat source can be tailored by altering the structures of the nanoheaters. Nanoheaters with sufficiently fine-scale distributions of Al-Ni interface may ignite spontaneously when moderate bulk heating is applied to the material that contains the nanoheaters. Such nanoheaters can be ignited at low temperatures, at which no harm occurs to the substrate or bulk material itself. Applications for this new technology may include, but are not limited to, biomedical therapies[3] (e.g., heating cells and tissues), clean-energy enablers (e.g., pre-heating fuel cell membranes), sustainable products (e.g., reconfiguring polymer microstructures), MEMS and lab-on-a-chip applications.

2. NANOHEATER FABRICATION AND CHARACTERIZATION

To achieve controlled heat generation and distribution, one must have (1) full understanding of the effect of process parameters on the fabricated structure and (2) validated models of the heat output. These require information and validation from characterization of the composition, geometry and configuration of the fabricated nanoheaters

2.1 Multilayered Thin Films

Multilayered Al-Ni thin films are fabricated by a sputtering system at the University of Cyprus. The samples were characterized using JEOL 7401 field-emission scanning electron microscopy (FE-SEM), **Figure 1**. For this specimen the alternating layers of Al and Ni were in the 70-100 nm range. Additional SEM results were used to help establish sputtering process parameters. Since the specimens were fragile, they were sandwiched between two layers of backing material, e.g., silicon wafers, and glued by thin layers of epoxy. Fine polishing with 0.3 μm diamond suspension produced artifact-free smooth surfaces essential for microscopy. All the samples were observed for layer thickness, uniformity, degree of intermixing, defects, chemical composition, and phase identification after reaction.

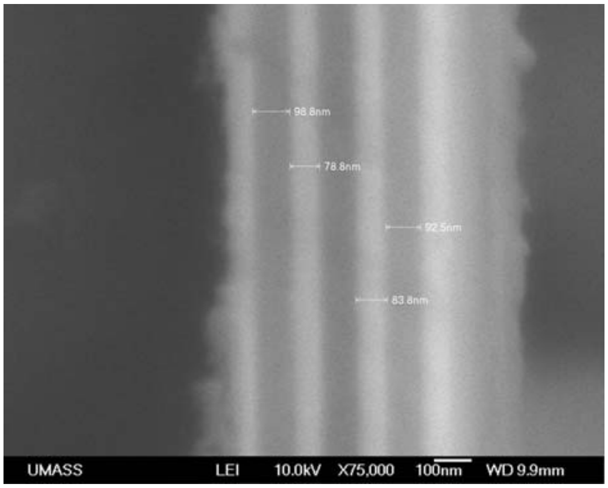


Figure 1 FE-SEM image of alternating thin films of Al and Ni deposited by sputtering at the University of Cyprus.

2.2 Powder-based Composites

Ultrasonically consolidated compacts were produced from Al and Ni powders. Al (99.5% pure) and Ni (99.8% pure) powders, 7-15 and 45-150 μm in size, respectively, were mixed in 1:1 molar ratio for 45 minutes in a cylindrical powder mixer revolving at 500 rpm. The powder mixture was subjected to ultrasonic consolidation at 300 °C in air using a punch and die arrangement as schematically shown in **Figure 2A**. The die is made of a 0.78 mm thick nickel plate with a die hole of 3.4 mm in diameter. Ultrasonic vibration was applied parallel to the die surface at a frequency of 20 kHz with vibration amplitude of 10 μm under uniaxial pressures of 70 to 250 MPa to study the effect of the stress on the microstructure, ductility and ignition behavior of the samples produced. The consolidation time was set at 1 s, which was followed by an additional 0.02 s of an after burst time. Compacts 3.3-3.8 mm in diameter were produced.

Figure 2B shows an SEM image of a compact ultrasonically consolidated under a uniaxial loading of 210 MPa. The compact shows a high degree of densification and a composite microstructure where Ni particles (lighter grey) are well dispersed in the Al matrix (darker grey). Thus, the Al powder particles took most of the deformation needed to produce the consolidated composite while the Ni particles being harder remained virtually undeformed. In most of the specimen volume, densification was complete and the contact between the Al matrix and the Ni particles was well-established as seen in the inset. Some porosity, however, was also noticed at the Al/Ni interface in the edge regions of the specimen where insufficient supply of aluminum around the Ni particles resulted. EDX results of the consolidated specimen showed no evidence of oxide formation in the matrix as well as in the Al/Ni interface.

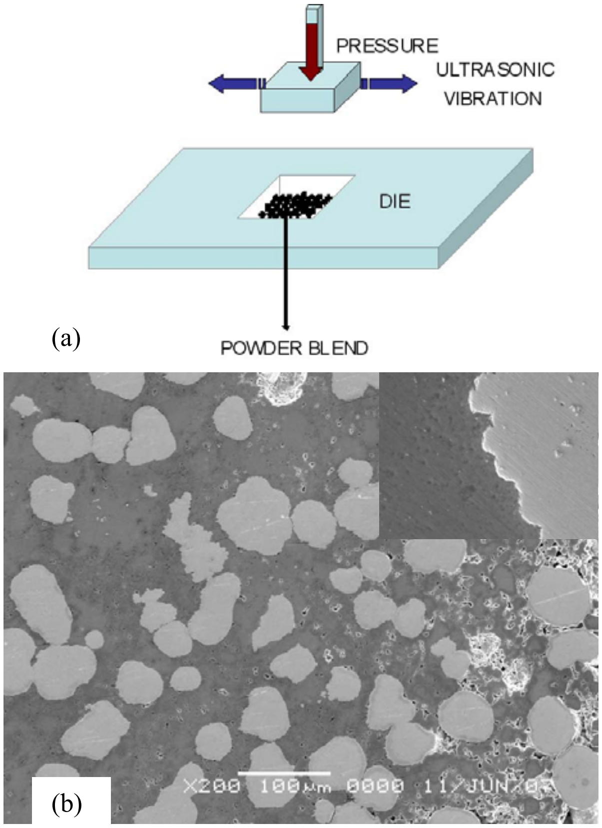
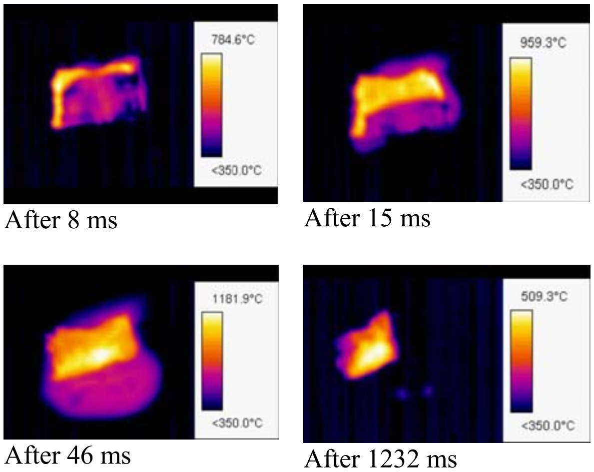


Figure 2 (A) Schematic of ultrasonic powder consolidation (UPC). **(B)** FE-SEM image of an Al/Ni compact consolidated at 300 °C and 210 MPa for 1 s. Inset reveals intimate contact between the Al matrix and the Ni particles.

Preliminary experiments were also performed with nano-flakes, 200 - 300 nm thick and 50 - 100 μm across, produced by a proprietary hammer milling technique. Fully dense consolidates with no delamination were obtained at 300 °C under 160 and 200 MPa.

3. THERMAL CHARACTERIZATION OF NANOHEATERS

A commercial multilayered nanoheater sample (RNT, Inc., Hunt Valley, MD) was ignited and the electromagnetic radiation (infrared band) was captured using an infrared pyrometry camera (SC4000, FLIR Systems, Billerica, MA). Thermal images reveal that the self-propagating reaction reached a peak temperature of 1181 °C in 46 ms. **Figure 3** shows 4 instances at time intervals as the reaction progressed.



After 8 ms

After 15 ms

After 46 ms

After 1232 ms

Figure 3 Infrared (IR) images of the exothermic reaction of an ignited multilayered Al-Ni film at different times.

Ignition tests were also performed on the ultrasonically consolidated specimens by heating them with a torch in open air (**Figure 4A**). Compacts with sufficient densification (consolidated at 160 MPa or above) ignited after a very brief exposure to the propane torch flame. **Figure 4B** shows the microstructures of the specimen after the ignition. The prior Ni particles are hardly seen in the ignited specimen, indicating that the exothermic reaction has propagated throughout the compact. EDX analysis indicated that the resultant phase produced was NiAl and that slight oxidation occurred during ignition due to the ambient conditions.

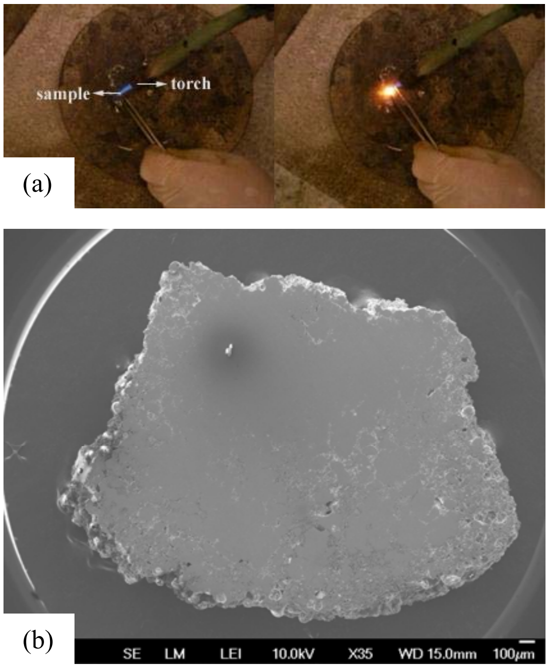


Figure 4 (A) A compact subjected to heating with a torch ignites in 150 ms. **(B)** SEM image of the sample after the self-propagation of the exothermic reaction upon ignition.

Reaction did not self-propagate in powder-based compacts with insufficient densification. Consolidates made from the nanoflakes were ignited even more readily.

4. THERMAL MEASUREMENT AND MODELING

One key advantage of nanoheaters is the ability to provide controlled, intense, and rapid heat to a very localized region, thus reducing energy, processing time, and collateral damage. To design the nanoheaters properly, it is important to predict the heat output and the affected adjacent area and time-temperature profile developed in that area after the nanoheater ignition. A series of experimental studies were initiated that can be used to validate the models for (1) 1-D infinite plane heat conduction and (2) 2-D sequential ignition.

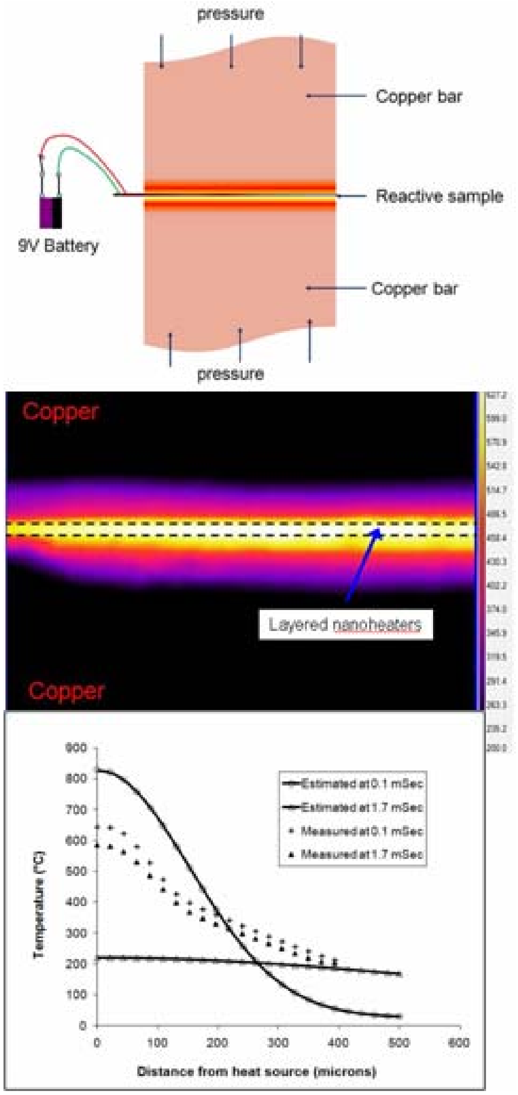


Figure 5 (A) Schematic of 1-D heat conduction setup **(B)** Temperature profile from FLIR IR measurements. **(C)** Comparison of model (lines w/dots) and experimental (dots) measurements of 1-D temperature distribution in infinite length copper rod after instantaneous heat input of 53 kJ/m^2.

4.1 1-D Infinite Plane Heat Conduction

The 1-D transient heat conduction generated by a multilayered nanoheater film (80 μm) was estimated and measured. The nanoheater was in intimate contact with the entire bottom surface of a copper rod (k=383-391 W/m·K), which is used as a bulk material. Air was used for insulation purposes. An estimate was made by applying transient heat conduction equations for 1-D conduction in bulk media.[4] It was assumed that nanoheater heat release was instantaneous and no temperature gradient existed along the thickness of the nanoheaters. If the diameter and length of copper are much larger than the thickness of the nanoheater film (with insulated sides), the temperature distribution can be predicted. **Figure 5A** shows a schematic of this 1-D setup. **Figure 5B** shows the thermal imaging measurements of temperature distribution with the FLIR camera. **Figure 5C** shows estimated and measured temperature profile along the copper rod. For estimated values (lines), the increase in temperature is initially localized to within 500 μm of the heat source at 0.1 ms, and the material beyond that distance is still at room temperature. Within 2 ms, the temperature has dropped rapidly, as it is a decaying function of time, and the heat flow has traveled beyond the 1000 μm distance. However, there is large difference between estimated and experimental values, probably due to the assumption of instantaneous heat generation. Further study is currently under investigation.

4.1 2-D Sequential Nanoheater Ignition

In addition to localized heating, the nanoheaters can be designed to ignite in a sequential manner. Initial experiments have been conducted to quantify the control of the sequential ignition. **Figure 6A** shows a schematic of three nanoheater “islands” connected by a narrow nanoheater strip (all materials are multilayered films). The first island can be ignited and if sufficient heat is transmitted by the inter-connecting strips to the second one, that island will ignite too. In the experimental setup, rectangular films were fixed to a silicon wafer and then narrower strips of films were attached between each pair of films. The nanoheaters were ignited in open air, and the reaction propagated rapidly from island to island until all of the material had been reacted (**Figure 6B**). Further studies are planned to quantify this process.

5. CONCLUSIONS

Fabrication of ignitable Al/Ni heterostructures has been successfully accomplished by sputtering of multilayer films and powder-based deformation bonding (UPC). Al and Ni powders as well as nanoflakes were consolidated under different processing conditions. An oxide and pore free Al matrix and intimate interface between the Al matrix and Ni particles were achieved. Torch heating ignited the powder consolidates, causing a self-propagating reaction that produced NiAl with only slight oxidation due to external effects. The UPC route will be further explored to establish consolidation conditions for nanoflakes.

The ability to capture and quantify ignition and temperature distribution with microscale temporal and spatial resolution was demonstrated. Improved quantification of heat output and ignition conditions will be conducted using differential scanning calorimetry (DSC) and infrared cameras for the model cases and the sequential ignition studies. Models of both the exothermal reaction kinetics and the macroscale heat conduction are in progress and results will be compared with the experimental results.

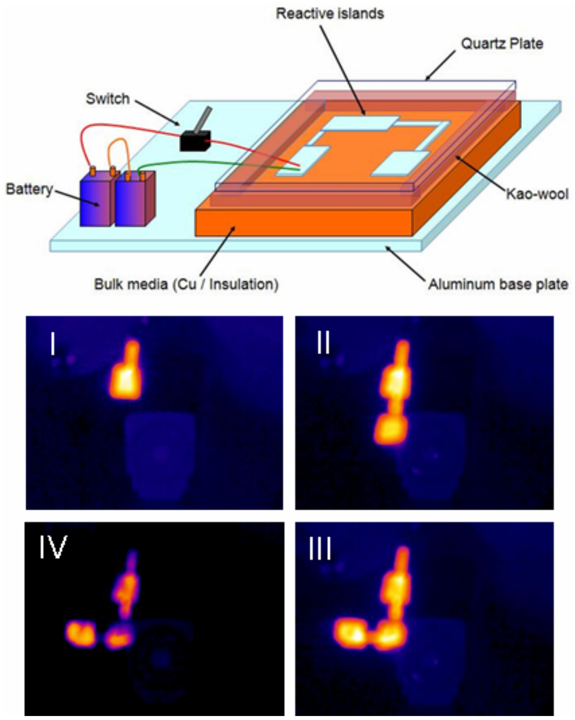


Figure 6 (A) Schematic of 2-D sequential ignition set-up. **(B)** Sequential ignition recorded through a quartz plate by the FLIR camera (red color indicates ignited nanoheaters).

ACKNOWLEDGEMENTS

The authors acknowledge the financial support for the research by the National Science Foundation (DMI 0531127 and 0738253), the European Commission through Marie Curie Chair “UltraNanoMan” (MEXC-CT-2004-006680) and Marie Curie Excellence Team Project “NanoHeaters” (MEXT-CT-2005-0023899). They would also like to thank FLIR Systems (Billerica, MA) for their assistance.

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