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Room-temperature soldering with nanostructured foils

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Self-propagating formation reactions in nanostructured multilayer foils provide rapid bursts of heat and can act as local heat sources to melt solder layers and join materials. This letter describes the room-temperature soldering of stainless steel specimens using freestanding, nanostructured Al/Ni foils. The products, heats, and velocities of the reactions are described, and the microstructure and the mechanical properties of the resulting joints are characterized. A tensile shear strength of 48 MPa was measured for the reactive foil joints, compared to 38 MPa for conventional joints. Both numerical predictions and infrared measurements show limited heat exposure to the components during reactive joining. © *2003 American Institute of Physics.* [DOI: 10.1063/1.1623943]

Over the last decade, exothermic formation reactions have been reported to self-propagate in a variety of nanostructured multilayer foils, such as Al/Ni, Al/Zr, Ni/Si, and Nb/Si foils.[1–6] Self-propagating formation reactions in multilayer foils are driven by a reduction in chemical bond energy. With a small thermal pulse, atoms diffuse normal to the layering as shown schematically in Fig. 1(a), and A–A and B–B bonds are exchanged for A–B bonds. This local bond exchange produces a large quantity of heat that is conducted down the foil and facilitates more atomic mixing and compound formation. Reactions can be ignited in these foils at room temperature with a small spark, and the reactions self-propagate along the foils at speeds ranging from 0.1 to 30 m/s.[3–6] As the atoms in the alternating layers mix, heat is released very rapidly and the temperature of the complete foil can rise above 1200 °C in milliseconds.[6]

Self-propagating reactions in multilayer foils offer an exciting opportunity to dramatically improve conventional joining technologies by acting as local heat sources to melt solder or braze layers and thereby join components [Fig. 1(b)]. Since heat is localized to the interface being bonded, temperature sensitive components or materials can be joined without damage. In addition, since the nanoscale layers in the foils provide very small diffusion distances for mixing, and thereby promote self-propagation, this method of soldering and brazing can be performed at RT, and in air, argon, or vacuum.

In this letter, we briefly describe exothermic reactions in nanostructured Al/Ni multilayer foils, and demonstrate that they can be used to solder Au-coated stainless steel specimens. The products, the heats and the velocities of these reactions were characterized using x-ray diffraction (XRD), differential scanning calorimetry (DSC), and optical measurements. The total time for joining and the temperature rise in the components were estimated using infrared imaging. Lastly, the microstructure and mechanical strength of the resulting joints were characterized using scanning electron microscopy (SEM) and tensile shear lap tests.

Multilayer foils of different thicknesses were fabricated by sequentially magnetron sputter depositing many Al and Ni layers onto cooled substrates. All the foils contained 2000 nanoscale layers of Al and Ni. The relative Al and Ni layer thicknesses were maintained at a 3 to 2 ratio to obtain foils with a 50/50 atomic ratio of Al and Ni. Bilayer thicknesses ranged from 35 to 85 nm, and therefore the total foil thicknesses ranged from 70 to 170 μm. The base pressure of the chamber prior to deposition was less than 1×10^{-6} Torr and

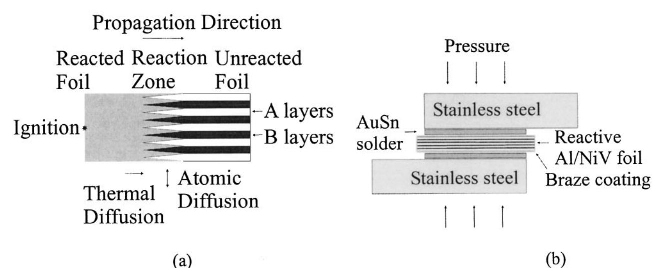

FIG. 1. (a) Schematic drawing of a self-propagating reaction in a multilayer foil, showing a cross-sectional view of the atomic diffusion between A and B layers and thermal diffusion along the foil. (b) Schematic showing the reactive joining of Au-coated stainless steel shear lap specimens. The joints were made using Al/Ni reactive foils, sheets of AuSn solder, and an applied pressure of 100 MPa.

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the pressure of Ar during deposition was 1 mTorr. To enhance wetting of the foils by the AuSn solder during joining, the foils were coated with a 1-μm-thick wetting layer of braze (Incusil ABA). The braze was deposited under 5 mTorr of Ar, both before and after the Al/Ni multilayer was deposited. Afterwards the reactive multilayer foils were removed from their substrates for use as freestanding foils.

DSC was performed on freestanding Al/Ni foils using a Perkin Elmer DSC. The foils were heated from 50 to 725 °C at 40 °C/min in Ar. By integrating the heat flow with respect to time, the heat of reaction was obtained.[7] The integrated heats were constant with bilayer thickness. This suggests that the intermixing between layers that occurs during deposition was very limited in depth, relative to the bilayer thicknesses examined.[5–7] The average heat of reaction for the Al/Ni foils was measured to be 1168 ± 21 J/g.

The phases in as-deposited and reacted Al/Ni foils (without the Incusil coating) were characterized using symmetric XRD. For the as-deposited foils all the peaks in X-ray diffraction scan correspond to Al and Ni. To characterize the Al-Ni products, these foils were ignited in air and then ground into powders for XRD examination. All major peaks correspond to the ordered B2 AlNi compound, which is the equilibrium compound for this composition. The same final compound is expected when reacting the foil during joining.[6]

Reaction velocities were measured using a series of optical fibers, as described earlier.[4] As the reaction flame propagates in front of the fibers, the fibers are illuminated sequentially and the total absorbed light is coupled and sent to one photodiode. Using the resulting voltage signal and the spacing of the optical fibers, reaction velocities can be determined. Since the reaction heats were measured to be constant over the range of bilayers examined, reaction velocity should be determined by the average diffusion distance, and therefore should vary inversely with bilayer thickness.[5,6,8,9] As expected, the reaction velocity does increase from 3.5 to 6 m/s, as bilayer thickness decreases from 85 to 35 nm.

To attempt reactive soldering, stainless steel specimens were cut to the following dimensions, $0.5\ \text{mm} \times 6\ \text{mm} \times 25\ \text{mm}$, and they were electroplated with Ni and then Au to enhance wetting and bonding. Joints were fabricated by stacking two 25-μm-thick sheets of AuSn solder and one reactive foil between two stainless steel samples at RT in air under approximately 100 MPa of pressure, as shown schematically in Fig. 1(b). Note that to wet the components and form a strong joint between them, the freestanding solder layers must melt across their complete thickness. The joint area was approximately $5\ \text{mm} \times 6\ \text{mm}$.

Ten stainless steel shear lap specimens were joined, and then tested in tension at RT using an Instron testing machine and a cross-head speed of 0.1 mm/min. The average shear strength was 48 ± 3 MPa. While the heat available for bonding scales linearly with foil thickness, the measured shear strengths did not increase with total foil thickness, as it varied from 70 to 170 μm. This suggests that additional heat provided by the thicker foils does not lead to stronger joints when using this particular set of materials and geometry.

Cross sections of untested stainless steel joints were characterized using SEM. Figure 2 offers a typical example of the interfacial region after stainless steel specimens were

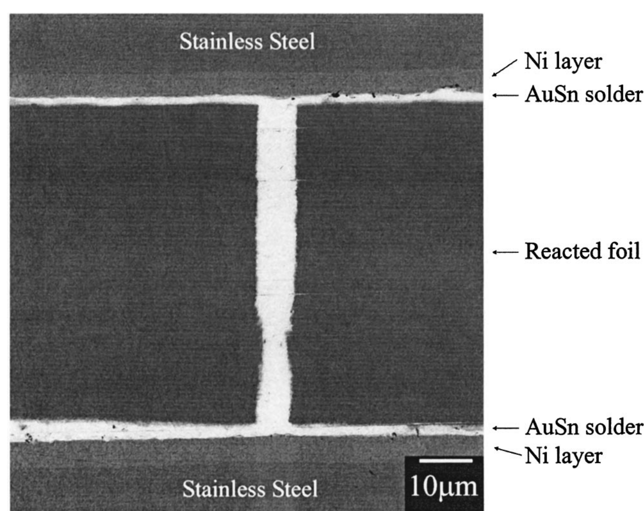

FIG. 2. SEM micrograph of stainless steel components joined using reactive Al/Ni foils and sheets of free standing AuSn solder (25 μm thick). Note that most of the AuSn solder flows out of the joint and into cracks that form within the foil on reaction and on cooling. It also shows the distinct interfaces between the reacted foil, the AuSn solder, and the Ni coating on the underlying stainless steel.

joined using two pieces of freestanding AuSn solder (originally 25 μm thick) and one Al/Ni reactive foil (80 μm thick). The cross-sectional images identify several important points. First, when the foils react they contract due to densification; they also contract due to cooling from the high reaction temperatures. Both sources of contraction can lead to cracking. However, molten solder typically flows into these cracks. Second, note that the AuSn solder layers have shrunk in thickness from 25 μm to several microns, suggesting the majority of the solder flows into cracks and out of the bond area, due to the applied pressure. Lastly, the combination of strong joints and this image suggests that the AuSn solder layers are melting across their full 25 μm thickness and enabling wetting of the Au-coated stainless steel specimens. Since the melting point of the AuSn solder is 280 °C, such evidence suggests solder or braze layers with higher melting temperature could be used, particularly with thicker foils.

For comparison, some stainless steel specimens were joined using a furnace instead of a reactive foil to heat the solder. In this case, two stainless steel specimens and one piece of AuSn solder were clamped together and heated above the melting temperature of AuSn solder in air. Afterwards the stainless steel joints were tested in tension under the same conditions. The average shear strength of these specimens was 38 ± 1 MPa. The lower strength can be attributed to coarser microstructures, compared to the reactive multilayer joints that cooled very rapidly.

A numerical study was performed to predict the evolution of the temperature profiles within the bonded stainless steel components. The model is based on a simplified description of the self-propagating reaction that relates the nanoscale transport and kinetic phenomena, which govern the self-propagating reaction in the foil, with the thermal transport and phase evolution, which occur in the AuSn solder layers and the stainless steel components. The model assumes one-dimensional motion of the reaction front, which is described using experimentally determined heats of reaction and velocities of the foils. Our computation focuses on

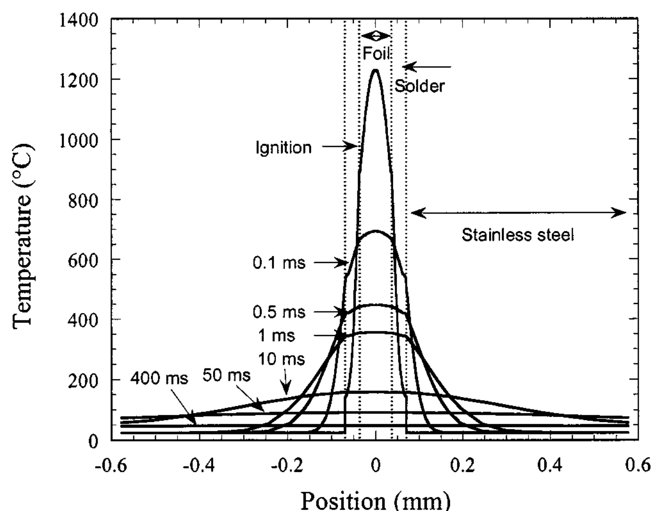

FIG. 3. Temperature profiles across the stainless steel joint obtained from numerical predictions.

simulating heat flow into the solder layers, phase-changes within these layers, and temperature evolution within the bonded components. The temperature evolution can be obtained by integration of the energy conservation equation, which is independently solved within the reactive foil, solder layers and stainless steel components:

$$\rho \frac{\partial h}{\partial t} = \nabla \cdot q + \dot{Q},$$

where ρ and h are the density and enthalpy of the corresponding layer, respectively, t is time, q is the heat flux vector, and $\dot{Q}$ is the heat release rate. The enthalpy h is related to the temperature through a relationship that involves the material's heat capacity c_p and the latent heat h_f.

Figure 3 shows the temperature profiles across the joint obtained from numerical predictions at the moments of reaction, 0.1, 0.5, 1, 10, 50, and 400 ms after reaction. It shows that the temperatures across the joint decrease very quickly to 48 °C at 400 ms after reaction, and that the heat effected zone in the stainless steel samples is spatially very limited, effectively to less than 250 μm. Figure 4 is a plot of temperature versus time in the stainless steel components, 100 μm from the solder/stainless steel interface. The numerical results predict that following reaction, temperature will decrease to 60 °C within 0.2 s, and that the heat exposure to the stainless steel components is very limited and localized.

To assess heating and cooling rates experimentally, temperatures in the stainless steel components were measured during the reaction using an FLIR SC500 infrared camera. Before the joining process, the sides of the stainless steel specimens were polished and painted white, to ensure a uniform emissivity. The temperatures at the side surfaces of the components were then monitored using the infrared camera with a spatial resolution of 108 μm and a temporal resolution of 0.2 s. Based on a series of thermal profiles that start prior to ignition, it can be estimated that the total heating time is less than 0.2 s. After the reaction, temperatures in the stainless steel specimens decreased very quickly. In the stainless steel components at 100 μm from the interface between the solder layer and the stainless steel, the temperature decreased

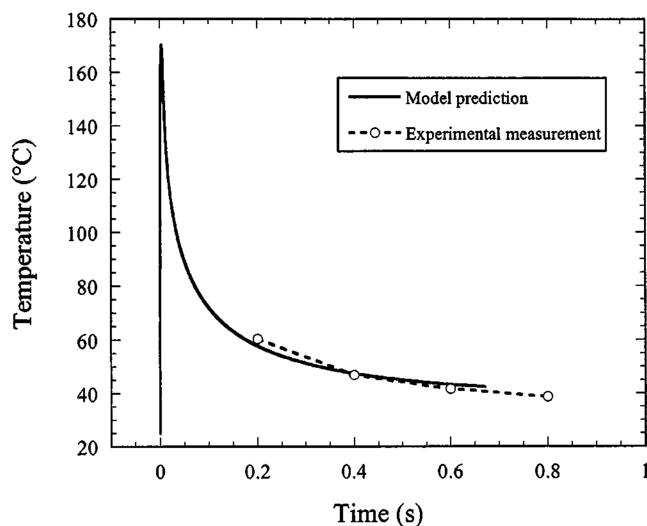

FIG. 4. Temperature changes in a stainless steel component, 100 μm from the solder/stainless steel interface, from both numerical predictions and experimental measurements.

to 60.4 and 38.8 °C at 0.2 and 0.8 s after reaction, respectively, as shown in Fig. 4, which is consistent with the numerical prediction. Here the cooling rate is estimated to be $>$1000 °C/s. This demonstrates that the heating of components is very limited, particularly when compared to traditional soldering that utilizes furnaces.

In conclusion, we have demonstrated a method of RT soldering using reactive Al/Ni nanostructured multilayer foils. The reactive foils have heats of reaction equal to 1168 $\pm$ 21 J/g, an ordered final product of AlNi, and reaction velocities ranging from 3.5 to 6 m/s. The self-propagating reactions within the foils can act as very localized heat sources that completely melt freestanding sheets of AuSn solder under pressure, thereby joining stainless steel specimens. The stainless steel joints formed using this method have an average shear strength of 48 $\pm$ 3 MPa, which is higher than conventional AuSn solder joints (38 $\pm$ 1 MPa) that cool far more slowly. The very rapid and localized heating of the reactive nanostructured foils offers the ability to join temperature-sensitive materials.

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