Thermochemical Metrology of Interfacial Stabilities

Hetero-interfaces are present in the majority of electronic materials and affect the operating characteristics of many devices. For successful fabrication and optimal performance of advanced designs, data on the behavior of interfaces during thermal processing is essential. Efficient measurement and prediction of the interfacial thermochemical stabilities of potentially useful materials combinations require new technology and new approaches, which are being developed in our laboratories.

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By using thin-film differential scanning calorimeters (DSCs), it is possible to study materials interactions on a very fine scale — both in terms of the mass of the samples involved, as well as the magnitude of the thermodynamic quantities measured. A further advantage of the thin-film DSC approach to thermochemical metrology is that it lends itself to combinatorial studies, in which compositionally graded thin films can be deposited over a DSC array. In this way, interfacial stabilities of various materials combinations can be rapidly evaluated. Our plan is to correlate thermochemical data from DSC arrays with characterization by x-ray microdiffraction, using an area detector and an automated stage to sample each element of the array. The products formed during interfacial reaction will be identified, and these experiments will form the basis for more detailed examination of the kinetics of reaction for appropriate materials combinations.

To date, our efforts on thin-film DSC have concentrated on proof-of-concept using well-known thermochemical events such as the melting of Sn (232 ºC), the ε/α transition in metallic Co (422 ºC), and the reaction between elemental Si and Ni thin films (kinetically determined). Studies of the latter two reactions are still in progress, but our preliminary studies of Sn melting have been completed. Figure 1 shows a 200 nm thick layer of Sn deposited on the sample side of a thin-film DSC sensor. The sample was deposited by pulsed laser deposition (PLD) through a 60 µm x 100 µm mask; a range of particle sizes is evident. Figure 2 is an uncalibrated average of multiple DSC scans from a sample like that in Figure 1. The melting (endothermic) and crystallization (exothermic) scans show multiple events, possibly related to differences in thermal behavior of different size fractions of the particles. A large thermal hysteresis between melting and crystallization is evident. This example suggests that the thin-film DSC is sensitive to variations in materials properties, in this case probably influenced by the interfacial tension between the metallic Sn in the core of the particles and the thin SnO_2 skin on the surface of the particles.

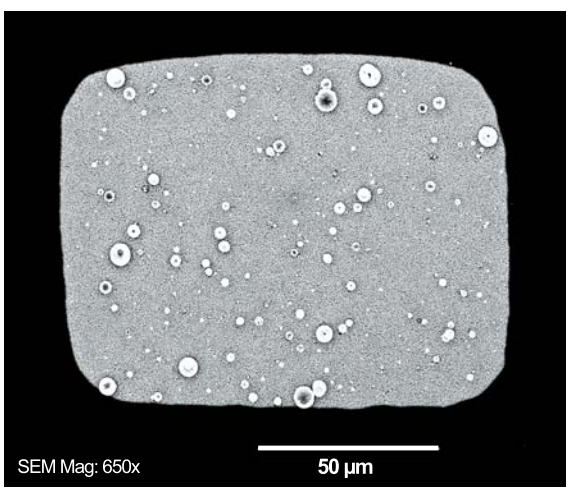

Figure 1: *PLD-deposited Sn on DSC sensor.*

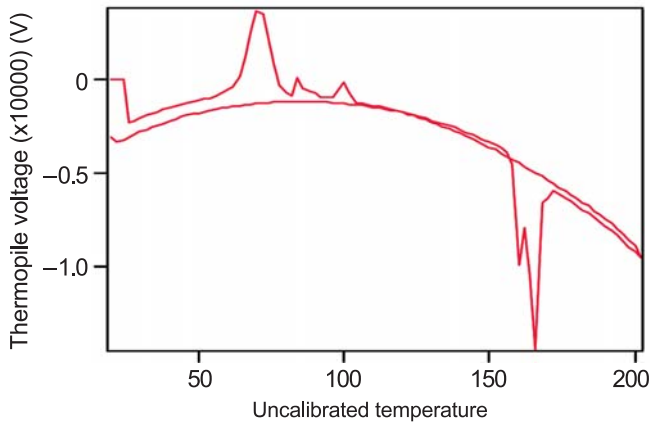

Figure 2: *Thin-film DSC of Sn in Figure 1.*

Work has already begun on a second generation of thin-film DSCs that will be optimized to give true nanometric sensitivity. This work is being done in collaboration with the University of Illinois at Urbana–Champaign. Currently, instrumentation is being set up to utilize the enhanced sensitivity that will be achieved through a chip design minimizing thermal mass. With the newer devices, near-adiabatic measurements will be possible.

Contributors and Collaborators

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