### 18. *Gas Sensing with Microhotplate Sensor Arrays*

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**Objective:** Demonstrate feasibility of silicon micromachined, thin-film sensor measurement technology for multi-analyte, real-time detection and concentration determination of gases.

**Problem:** Increasing global competition has placed new demands on the chemical process industry for more efficient use of materials, better process reproducibility, and environmental safety. Meeting these demands requires a low-cost technology for the measurement of gas species, which can provide immediate, onsite analysis for the detection of reaction products, exhaust gases, leaks, etc. Gas-phase measurements are also needed for applications ranging from environmental monitoring at hazardous waste sites to chemical agent detection.

**Approach:** Advances in micro-fabrication technology now make possible miniaturization of conventional conductometric low-cost metal oxide sensors into a planar array form. A sensor array platform has been developed which uses a microhotplate as the generic device structure. The microhotplate has three functional layers: a heater, a thermometer/heat distribution plate, and electrical contacts for monitoring the conductivity of sensing films. NIST holds three patents on this technology. There are three key components to the microsensor research program: advancing sensor materials, understanding transducing mechanisms, and developing new methods for sensor operation and signal analysis. Catalyst-doped metal oxide materials are used for sensing films and evaluated on the basis of sensitivity, selectivity, and stability. The dominant sensing mechanisms under investigation include catalyzed reactions, adsorption/desorption, grain boundary diffusion, and electronic effects related to surface states created by chemisorbed species. Surface analytical techniques combined with electrical measurements are used to address these issues. New sensing modes are possible that use the ability of these devices to be heated and cooled in milliseconds over a large operating temperature range (>500 °C). In temperature programmed sensing, the sensor is subjected to a repeating pattern of temperature pulses. Effects that produce a response signal are based on thermally-activated processes, such as adsorption, reaction, and desorption. The sensor generates repetitive response signatures that are characteristic of adsorbed species/sensing material combinations. Neural network and chemometric based approaches to this problem are being used to optimize the generation of patterns and to analyze signals during sensing.

**Results and Future Plans:** A nanoparticle-based sensor material was developed that exhibited sensitivity to methanol in air at concentrations below 10 ng/g, while providing stable performance in on/off cycle test with methanol over a time period of 100 hours. Nanoparticle tin oxide films were created by spinning a colloidal suspension of nanoparticles and then sintering with the device's microheater. Nanophase oxides can produce high sensitivities without the fouling effects that are often observed on metal catalyst-doped films. These materials will be further investigated using arrays with 4 to 48 elements, to relate microstructure and stoichiometry to sensing performance. We have also in the past year developed new temperature-pulsing techniques that combine mass spectrometry with electrical sensing to study sensing mechanisms. These methods will be used to characterize the gas-surface interactions for new sensing materials in different gas mixtures. Aspects of this work are funded by DTRA for chemical agent detection and DOE-EMSP for developing sensors for environmental management.

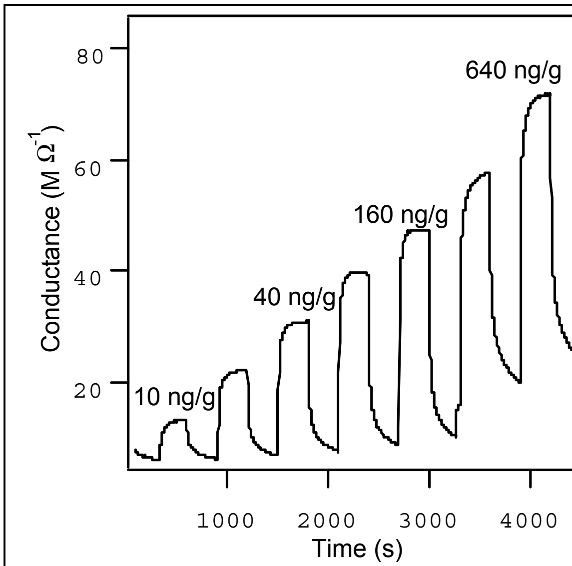


Figure 1. Response of a tin oxide nanoparticle sensing film to methanol in air, (concentration increases twofold at each step as indicated).