Use of Microhotplate Arrays as Microdeposition Substrates for Materials Exploration

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An approach for high-throughput rapid screening of chemical vapor deposition (CVD) materials using micromachined silicon microheater arrays is described. To illustrate this approach, titanium dioxide was deposited by CVD, using titanium(IV) nitrate and titanium(IV) isopropoxide at temperatures between 130 and 815 °C. Deposition was confined to the microhotplate elements within 4- and 16-element arrays. Film microstructure was examined by scanning electron microscopy. In situ electrical measurements were made with integrated microcontacts during the deposition of TiO_2 using titanium(IV) isopropoxide. A novel approach using temperature-programmed deposition with temperature ramp rates up to 800 °C/s was also employed for microstructure modification during deposition. Additionally, the steep temperature gradients present on the microhotplate supports have been demonstrated to provide an excellent platform for investigating temperature-dependent microstructures.

Introduction

It has been widely observed that the composition and microstructure of solid-state materials strongly influence their properties. Processing methods are developed, with considerable effort, to optimize properties for a wide spectrum of technological areas. However, a large number of techniques and conditions may need to be explored to produce the most desirable material. For example, in chemical vapor deposition (CVD) one must choose suitable precursor chemistry, reagent gas concentrations, and precursor partial pressure to control the composition and microstructure of metal and metal oxide thin films.[1−5] Sputter deposition processes are typically sensitive to target condition, sputtering power, reactant concentrations, and substrate orientation. Nearly all deposition processes are strongly influenced by substrate temperature during (and after) deposition.

Given the number of process variables that can affect a material, it is not surprising that combinatorial methods are becoming more commonly used to produce and identify promising candidate materials for development into future products.[6−15] In the area of electronic materials, for example, it would be useful if one could deposit varied forms of materials in a survey approach and characterize their electrical properties after deposition and postprocessing. In many cases, it would be beneficial if the electrical properties of the materials, such as resistance or capacitance, could be measured during the processing of multiple samples in parallel.

We have been using micromachined silicon devices called microhotplates as platforms for developing metal oxide thin film gas microsensors.[16−19] In this paper, we emphasize the utility of arrays of such micromachined devices as materials research tools. Microarrays of 4, 16, 36, and 48 individually addressable elements, each element with its own independent heating and electrical probe contacts, are currently in use. A representative sketch of a single microhotplate and an optical micrograph of a 16-element array are shown in Figure 1. The nominal size and mass of the suspended structure are 100 × 100 μm and 0.2 μg, respectively. The arrays provide excellent platforms for performing multiple

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(1) Smith, R. C.; Taylor, C. J.; Roberts, J.; Campbell, S. A.; Tiner, M.; Hegde, R.; Hobbs, C.; Gladfelter, W. L. *Chem. Mater.* **2000**, *12*, 2822−2824.
(2) Crawford, N. R. M.; Knutsen, J. S.; Yang, K.-A.; Haugstad, G.; McKernan, S.; McCormick, F. B.; Gladfelter, W. L. *Chem. Vap. Deposition* **1998**, *4*, 181−183.
(3) Barreca, D.; Depero, L. E.; Franzato, E.; Rizzi, G. A.; Sangaletti, L.; Tondello, E.; Vettori, U. *J. Electrochem. Soc.* **1999**, *146*, 551−558.
(4) Barreca, D.; Massignan, C.; Daolio, S.; Fabrizio, M.; Piccirillo, C.; Armelao, L.; Tondello, E. *Chem. Mater.* **2001**, *13*, 588−593.
(5) Becht, M.; Morishita, T. *Chem. Vap. Deposition* **1996**, *2*, 191−197.
(6) Briceno, G.; Chang, H. Y.; Sun, X. D.; Schultz, P. G.; Xiang, X. D. *Science* **1995**, *270*, 273−275.
(7) Bozovic, I.; Matijasevic, V. *Trends Adv. Mater. Processes* **2000**, *352*, 1−8.
(8) Buyevskaya, O. V.; Bruckner, A.; Kondratenko, E. V.; Wolf, D.; Baerns, M. *Catal. Today* **2001**, *67*, 369−378.
(9) Cawse, J. N. *Acc. Chem. Res.* **2001**, *34*, 213−221.
(10) Chang, H.; Xiang, X. D. *Integr. Ferroelectr.* **2000**, *29*, 113−125.
(11) Pescarmona, P. P.; van der Waal, J. C.; Maxwell, I. E.; Maschmeyer, T. *Catal. Lett.* **1999**, *63*, 1−11.
(12) Siu, T.; Yekta, S.; Yudin, A. K. *J. Am. Chem. Soc.* **2000**, *122*, 11787−11790.
(13) Tuinstra, H. E.; Cummins, C. L. *Adv. Mater.* **2000,** *12*, 1819−1822.
(14) van Dover, R. B.; Schneemeyer, L. F.; Flaming, R. M.; Huggins, H. A. *Biotechnol. Bioeng.* **1999**, *61*, 217−225.
(15) Xiang, X. D. *Mater. Sci. Eng. B* **1998**, *56*, 246−250.
(16) Suehle, J. S.; Cavicchi, R. E.; Gaitan, M.; Semancik, S. *IEEE Electron Device Lett.* **1993**, *14*, 118−120.
(17) Semancik, S.; Cavicchi, R. E. *Appl. Surf. Sci.* **1993**, *70−1*, 337−346.
(18) Semancik, S.; Cavicchi, R. E.; Kreider, K. G.; Suehle, J. S.; Chaparala, P. *Sens. Actuators B* **1996**, *34*, 209−212.
(19) Semancik, S.; Cavicchi, R. E.; Wheeler, M. C.; Tiffany, J. E.; Poirier, G. E.; Walton, R. M.; Suehle, J. S.; Panchapakesan, B.; DeVoe, D. L. *Sens. Actuators B* **2001**, *77*, 579−591.

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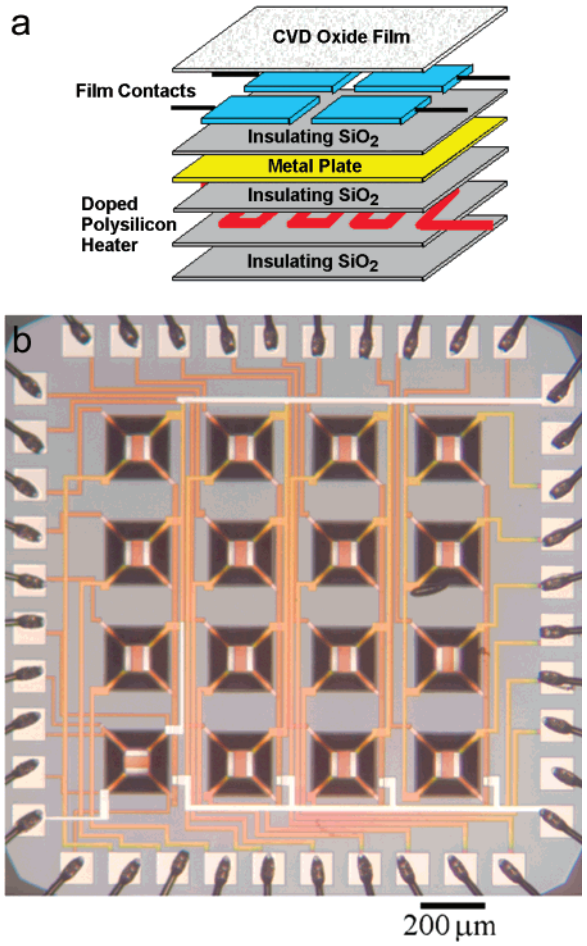

Figure 1. (a) Schematic diagram of a single microhotplate shown with optional metal heat distribution plate. (b) Optical micrograph of a 16-element microhotplate array.

experiments on a single chip. The low thermal mass and embedded heater enables them to be heated at rates of 10^5 to 10^6 °C/s, and operated with sustained temperatures in excess of 500 °C. Metal contacts (shown in Figure 1) allow the real-time electrical characterization of growing films for all elements of an array. Their small size/power output, coupled with their good thermal isolation, permits high temperatures with little heating of the surroundings. Thermally activated, self-lithographic processing makes it possible to deposit material only on the heated microsubstrate, thus reducing precursor consumption due to gas-phase depletion found in more standard reactors caused by deposition on other heated surfaces such as reactor walls and substrate holders.[18]

We concentrate here on CVD processing of TiO_2 using single-source precursors and SEM analysis as a model to demonstrate the feasibility and power of this approach. While we have demonstrated the approach on a known system, we emphasize its utility for examining new processing chemistry for pure and mixed materials. In addition to microstructural analysis by SEM, the microarrays may be used in conjunction with elemental analysis techniques such as EDS, and small area AES and XPS to determine film composition or for the preparation of mixed metal oxides prepared by low-temperature atomic layer deposition (ALD) where precursor chemistry plays a significant role in film growth.[20−23] They would also be useful for the processing of multilayer films that require different operating temperatures for the deposition of successive layers. Indeed, they make it possible to efficiently examine the effect of process parameters for new chemical precursors and rapidly identify processing recipes to make them work properly for a wide range of materials and technologies.

To demonstrate the microhotplate array's utility as a growth substrate for materials investigation, 16-element arrays were employed for the CVD of titanium dioxide films in the temperature range 100 to 410 °C under isothermal and temperature-programmed conditions using titanium(IV) nitrate as the precursor. Higher temperature depositions were performed using a 4-element array capable of operating in excess of 800 °C. Real-time electrical measurements during the CVD of TiO_2 using titanium(IV) isopropoxide (TTIP) at 400 °C were also made.

Experimental Procedures

Microhotplate arrays were fabricated at MIT-Lincoln Laboratories[24] using multilevel mask designs prepared at NIST. Completed wafers were received with a protective layer of photoresist. After the samples were diced, individual microhotplate arrays were bonded to a 0.75 × 0.75 cm piece of thermal oxide coated silicon wafer using epoxy for easier manipulation. Photoresist was removed using acetone, and the arrays were etched using tetramethylammonium hydroxide-ammonium persulfate etchant[25,26] to create the suspended microhotplate structure shown in Figure 1. Following etching, the microhotplate arrays were placed in standard 40-pin dual in-line ceramic packages and wirebonded using a Kulike and Soffa (model 4523)[24] wirebonder. Photographs of the packaged microhotplate array are shown in Figure 2. Experiments were carried out in a low-pressure CVD reactor constructed using a six-way 2.75 in. conflat cross. It is equipped with three stainless steel precursor lines, a fused silica viewport, and a 40-pin electrical feedthrough for making connection to the packaged microarrays. The remaining port is used to mount the cross on a larger chamber pumped by a Welch (1397)[24] vacuum pump. The 1/4-in. stainless steel precursor lines are equipped with VCR fittings and mass flow controllers (MKS M200 and 1179)[24] and bypass lines. Precursor vessels were constructed of Pyrex glass tubes with glass to metal seals equipped with mini-CF fittings for easy precursor loading. VCR fittings were used for connecting stainless steel bonnet valves to the vessels. Depositions were performed with precursor vessels held at constant temperature using heating tapes controlled by Eurotherm (91e)[24] temperature controllers and type K thermocouples.

Titanium Oxide CVD. Titanium oxide was first deposited using titanium (IV) nitrate (TN). This precursor is capable of depositing titanium oxide that is free of residual carbon

(20) Ritala, M.; Asikainen, T.; Leskela, H. *Electrochem. Solid State Lett.* **1998**, *1*, 156−157.
(21) Ritala, M. *Appl. Surf. Sci.* **1997**, *112*, 223−230.
(22) Ritala, M.; Leskela, M.; Dekker, J. P.; Mutsaers, C.; Soininen, P. J.; Skarp, J. *Chem. Vap. Deposition* **1999**, *5*, 7−9.
(23) Ritala, M.; Kukli, K.; Rahtu, A.; Raisanen, P. I.; Leskela, M.; Sajavaara, T.; Keinonen, J. *Science* **2000**, *288*, 319−321.
(24) Disclaimer: certain commercial instruments are identified to adequately specify the experimental procedure. In no case does this identification imply endorsement by the National Institute of Standards and Technology.
(25) Klaassen, E. H.; Reay, R. J.; Kovacs, G. T. A. *Sens. Actuators A* **1996**, *52*, 33−40.
(26) Tabata, O.; Asahi, R.; Funabashi, H.; Shimaoka, K.; Sugiyama, S. *Sens. Actuators A* **1992**, *34*, 51−57.

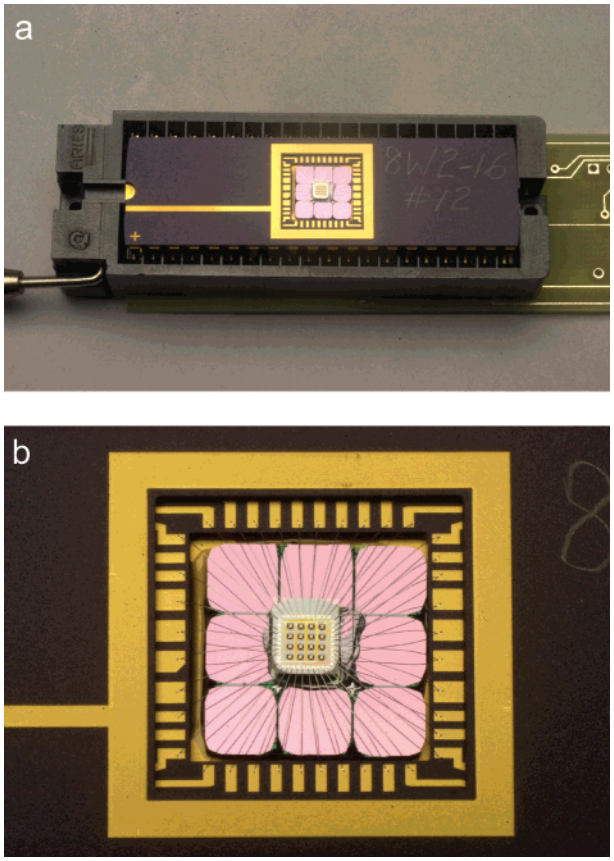

Figure 2. (a) Photograph of a packaged 16-element microhotplate array mounted in a standard socket similar to those used in this research. (b) Detail of packaged array.

without the use of an external oxidant.[27,28] Upon receipt, the titanium(IV) nitrate (Aldrich Chemical)[24] was ground under inert atmosphere using an agate mortar and pestle prior to transferring it to the precursor vessel. In these experiments, the precursor was maintained at 35 °C. High purity argon was used as the carrier gas with a one sccm flow rate. Reactor pressure was typically 500−650 mTorr. Microhotplate heaters were calibrated for temperature in situ with the carrier gas flowing through the bypass line. Following calibration, carrier gas flow was diverted into the precursor vessel, and after stabilization, the microhotplate was turned on to begin deposition. Experiments were carried out under isothermal conditions. Deposition times were 5 min for each sample. The 16-element arrays were used for deposition temperatures up to ∼450 °C, while four-element arrays without metal plates were used for deposition at temperatures in excess of 800 °C.

A second study was performed to demonstrate the effect of rapid temperature programming during the deposition. Previous research has shown that temperature-programmed deposition can be useful in optimizing film properties,[29,30] and it was speculated that it might be possible to obtain film microstructures that are not attainable under isothermal deposition conditions. The short thermal time constant ($\tau \sim 3-5$ ms) for the hotplate permits almost instantaneous temperature changes. Two temperature programs were used in this second study: a square wave where the heater is oscillated between two

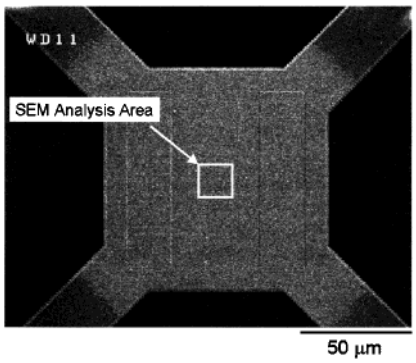

Figure 3. SEM image of a single microhotplate identifying the area used for collecting SEM microstructure data. Notice that deposition only occurs on the heated portion of the device.

different temperatures and a triangle wave where the temperature is ramped over a fixed temperature range. In all of the experiments presented here, the samples were prepared sequentially; we are currently developing a more sophisticated interface for performing all of these experiments simultaneously using this system. Nevertheless, the results illustrate the efficiency of deposition/processing with microarrays.

Results and Discussion

Film microstructure was examined by field emission scanning electron microscopy (Hitachi S-4100).[24] Uncoated samples were analyzed using a 5 kV accelerating voltage. Unless otherwise noted, images were acquired in the portion of the microsubstrate outlined in Figure 3. Figure 4 contains representative data for the isothermal deposition experiments performed using a single 16-element array and incremental deposition temperatures to illustrate the microstructure's temperature dependence when one uses TN as the precursor. Note the dramatic changes in microstructure that occur as substrate temperature increases. At the lowest temperature, large grains appear that eventually coat the microsubstrate surface. Substrate peak attenuation in the EDS data acquired from the samples after deposition indicate that films deposited above 170 °C are of the same approximate thickness (∼500 nm), while those deposited below 170 °C were considerably thinner (75 to 150 nm), consistent with reaction-limited growth. As the deposition temperature increases (175 to 250 °C), the large grains seem to subdivide along a particular crystallographic direction resulting in a platelike microstructure. This platelike microstructure may be explained in terms of the anisotropic nature of the TiO_2 surface. It is possible that the lone pairs on the nitrate ligands participate in specific donor−acceptor interactions with the surface. Such interactions might enable surface migration of adsorbed precursor to be more rapid in some directions than in others. The resulting deposits would likely be anisotropic along this preferred direction. Increasing deposition temperature further causes these plates to subdivide into smaller grains. This may be the result of a shortened diffusion length prior to desorption or reaction. These microstructures obtained below 400 °C exhibit large voids, indicating a relatively low surface mobility of the surface adatoms during film growth.

The results obtained when a 4-element array was used for deposition at 647, 695, 754, and 815 °C, using TN as the precursor, are shown in Figure 5. As shown

(27) Gilmer, D. C.; Colombo, D. G.; Taylor, C. J.; Roberts, J.; Haugstad, G.; Campbell, S. A.; Kim, H. S.; Wilk, G. D.; Gribelyuk, M.; Gladfelter, W. L. *Chem. Vap. Deposition* **1998**, *4*, 9−11.
(28) Taylor, C. J.; Gilmer, D. C.; Colombo, D. G.; Wilk, G. D.; Campbell, S. A.; Roberts, J.; Gladfelter, W. L. *J. Am. Chem. Soc.* **1999**, *121*, 5220−5229.
(29) Cale, T. S.; Richards, D. F.; Yang, D. W. *J. Comput.-Aided Mater. Des.* **1999**, *6*, 283−309.
(30) Yang, D.; Kristof, J. J.; Jonnalagadda, R.; Rogers, B. R.; Hillman, J. T.; Foster, R. F.; Cale, T. S. *J. Electrochem. Soc.* **2000**, *147*, 723−730.

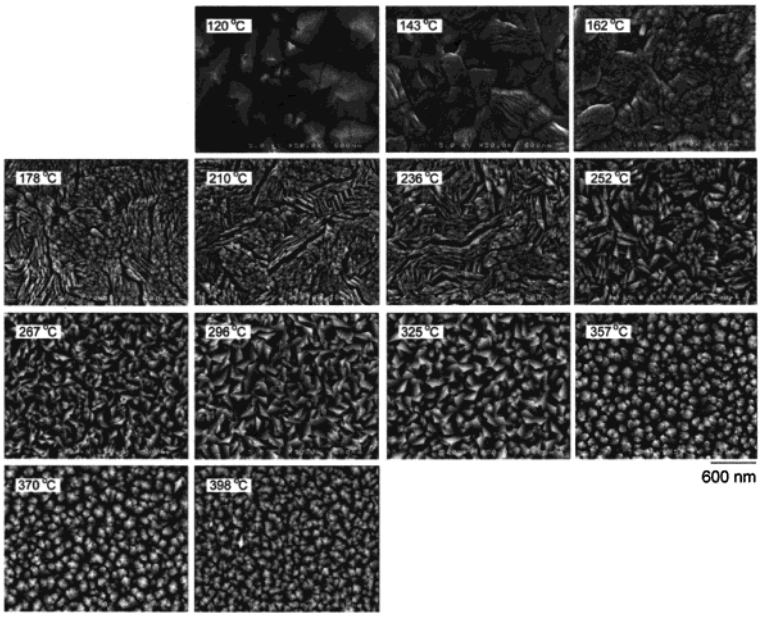

Figure 4. SEM microstructure data for 13 elements within a 16-element microarray onto which TiO_2 films were deposited on individual microhotplates in at temperatures ranging from 100 to 410 °C. Images were taken at the center of the hotplate.

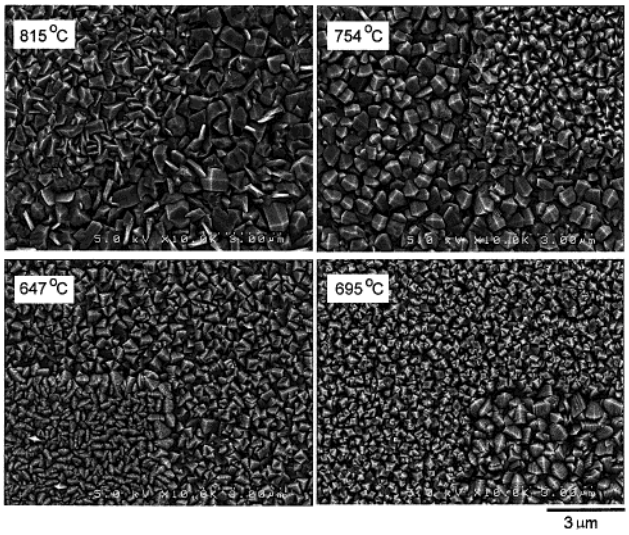

Figure 5. Electron micrographs showing the microstructure data for films deposited on a 4-element array using TN in the range 640 to 815 °C. Images were taken just off-center to illustrate the differences in microstructure on and off the contacts.

here, for temperatures above 640 °C, grain structures become better defined and elongated plates narrow into a transition microstructure of large and small grains with tapered tips. Grains exhibit pronounced faceting and increase in size with deposition temperature as predicted by the modified structure zone model (SZM) for thin films.[31]

While we have not yet performed definitive studies on the crystal structure of the deposited material, it is believed that the TiO_2 deposited below 700 °C is anatase based on previously reported X-ray diffraction data.[28] We are currently exploring the use of X-ray microdiffraction to confirm this belief. Rutherford backscattering spectrometry performed on the same set of samples indicated that the films deposited using TN, in the temperature range 159 to 890 °C, were all stoichiometric TiO_2. The main difference was that the films varied in density by ~10%. We do not currently have ready access to a scanning Auger microscope that would be needed to perform confirmation of the film stoichiometry.

The entire range of microstructures can also be viewed by examining the microhotplate legs, where steep temperature gradients exist during device operation. A composite of four SEM images (Figure 6) taken along one of the microhotplate legs illustrates the entire range of microstructures accessible in the temperature range 25 to 815 °C. The observed microstructures agree well with the results obtained using discrete elements, making it possible to interpolate the temperatures present on the surface of the microhotplate leg. Such a steep gradient (790 °C/70 μm) is not likely to be realized on larger samples, and as such, microhotplates offer an additional unique opportunity to study microstructural changes very efficiently. The presented result required four images to be carefully overlayed, but traditional larger samples unlike microhotplates would require dozens of images to be laboriously combined. Although a variety of microstructures may be demonstrated using this approach, the electrical characterization must be performed on discrete samples. The information obtained using the microhotplate legs can be used to assist planning future experiments where film microstructure is the main variable. It should be noted that the observed changes in microstructure are similar to those reported on Si (100) substrates under higher vacuum conditions than those employed in this study.[27,28]

The three different temperature profiles used in the temperature-programmed deposition experiments are shown in Figure 7. Examples of microstructure data from the temperature-programmed deposition experiment are shown in Figure 8. As would be expected, samples that were prepared using a square wave applied to the heater that is symmetric about 0 V (i.e., ±12.5 V) appear the same as those prepared at the same temperature in an isothermal experiment. It appears

(31) Messier, R.; Giri, A. P.; Roy, R. A. *J. Vac. Sci. Technol. A* **1984**, *2*, 500−503.

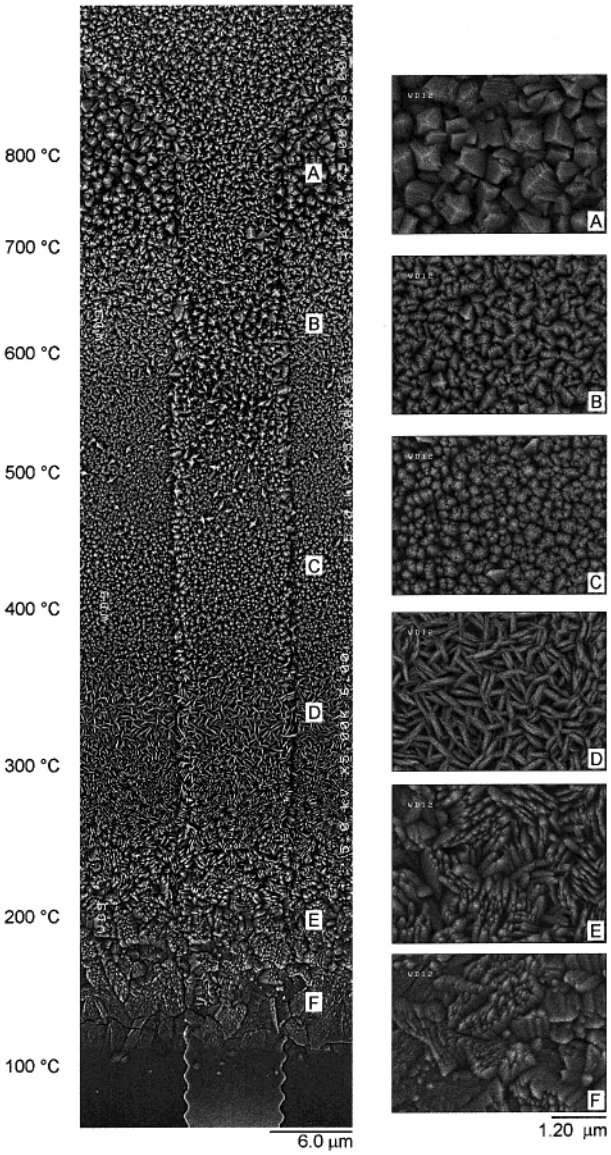

Figure 6. Composite electron micrograph taken along one of the supports of the microhotplate operated at 815 °C. Note the range of microstructures exhibited due to the steep temperature gradient. The labels (A−F) correspond to the location where the higher magnification images were acquired. The temperature scale is approximate and is based on the microstructures obtained in Figures 4 and 5.

as though TiO_2 films deposited using alternating temperature pulsing (labeled asymmetric square) are a combination of the microstructures obtained at each component temperature. The long, platelike grains are subdivided into smaller well-defined bundles most likely due to increased thermal energy at elevated temperatures. Films deposited at higher temperatures exhibit finer, well-faceted grain structures that appear more uniform than their low temperature counterparts. This is evidenced by the fact that two devices operating in this mode alternating between a common low temperature (170 °C) and 370 and 330 °C, respectively, show similar grain morphologies, but the sample that received the 370 °C pulse has better defined bundles than those in the sample that was prepared with the 330 °C pulse. Using a ramping temperature profile, it is possible to prepare microstructures that are distinctly different from those prepared under isothermal conditions. As for

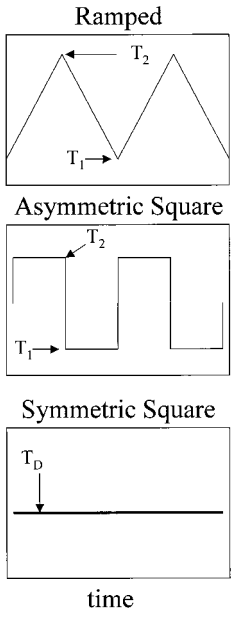

Figure 7. Temperature profiles used in the temperature-programmed deposition experiments. When a square wave that is symmetric about 0 V is applied to the microheater, the resulting temperature profile is identical to that of an isothermal deposition.

the case of pulsed temperature deposition, the ramped temperature morphologies have attributes in common with samples prepared isothermally. Although grain bundles are also observed, they are morphologically different. They are smaller in size than those observed in pulsed temperature deposition, possibly because no dominant microstructure develops due to the rapidly changing nature of the temperature program. The heating rates used in the temperature ramping experiments are still orders of magnitude slower than the platform's maximum heating rate, so future experiments will investigate the effects on film microstructure caused by faster temperature programs.

Electrical Measurements. Electrical measurements can also be performed on growing films using functionality designed into the elements of the microhotplate arrays. The TiO_2 films deposited using TN discussed above are too resistive for such monitoring, given the contact geometry used in these experiments. Even with interdigitated comb contacts,[32] limited success was attained for monitoring the growth of TN-deposited films. To demonstrate real-time conductance measurement on a growing film, TiO_2 was grown using TTIP as the CVD precursor. A plot of conductance vs time for a 4-element array is shown in Figure 9. There is an initial period of approximately 15−20 s where the film is too resistive to measure. Following this brief induction period, the conductance increases, roughly linearly with time, as might be expected for a constant growth rate.[33] The ripples in the conductance between 3×10^{-7} and 6×10^{-7} Ω^{-1} are likely artifact in nature caused by the autoranging function on the multimeter used to acquire the data. The fact that the slope of the plots is consider-

(32) Cavicchi, R. E.; Walton, R. M.; Aquino-Class, M.; Allen, J. D.; Panchapakesan, B. *Sens. Actuators B* **2001**, *77*, 145−154.
(33) DiMeo, F.; Cavicchi, R. E.; Semancik, S.; Suehle, J. S.; Tea, N. H.; Small, J.; Armstrong, J. T.; Kelliher, J. T. *J. Vac. Sci. Technol. A* **1998**, *16*, 131−138.

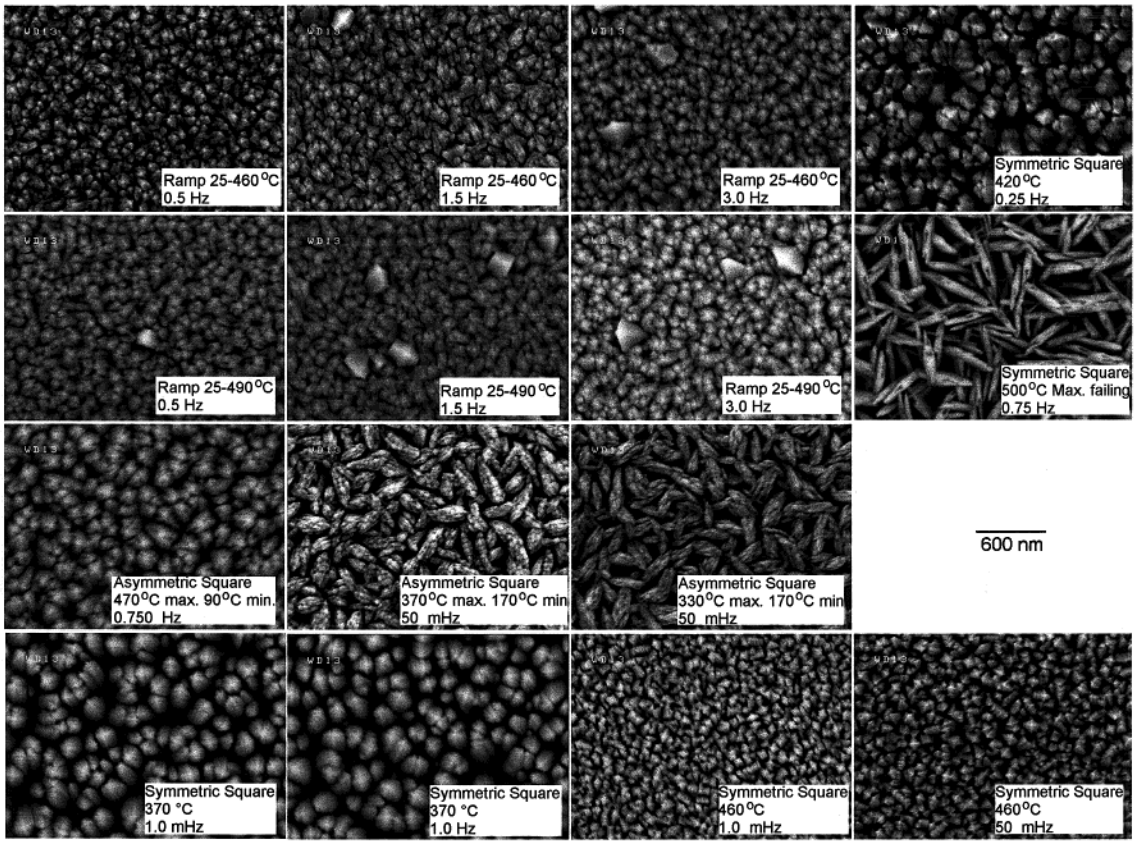

Figure 8. Microstructure data for TiO_2 films deposited on individual microhotplates using temperature-programmed deposition. Three types of depositions were performed: ramped temperature, in which the temperature is ramped between two temperatures, pulsed temperature (labeled Asymmetric Square) where the device alternates between two temperatures, and symmetric square where the absolute value of the heater voltage is unchanged resulting in an isothermal deposition.

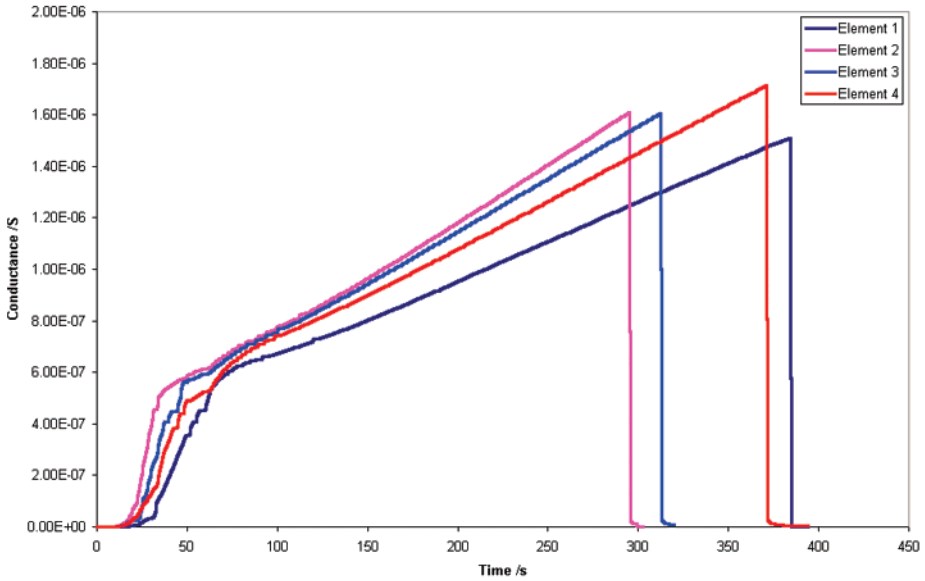

Figure 9. Conductance data obtained during the deposition of TiO_2 using titanium(IV) isopropoxide at 400 °C. Such real-time data may be used to further tune the film properties.

ably steeper in the early stages of film growth is not completely unexpected since measured resistance is related to both film thickness (which is increasing with time) and the chemoresistive nature of the film that allows changes in the ambient environment to change the film's electrical properties. In the early stages of film growth, the deposited material is similar in nature to a nanoparticle film in that the increased surface area-to-volume ratio of the growing film makes the device more sensitive to gases than a one with thicker film.[32,34] In the case of TiO_2 deposited using TTIP, this may be due to the increased sensitivity of the device toward the reaction products such as isopropyl alcohol, propylene, and water.

Modifications for Improved Array Experimentation. As stated earlier, devices used in this study were

(34) Zhang, G.; Liu, M. L. *Sens. Actuators B* **2000**, *69*, 144−152.

not designed with the intentions of operating at temperatures in excess of 550 °C, and as such, a few minor problems have been encountered. The tungsten contacts present in the elements of some microarrays were found to be unsuitable for making contact with the films at elevated temperatures. New microhotplate designs will address the contact material issues by incorporating platinum contacts on all of the devices, for more stable high temperature work. In addition, new designs will include a variety of new contact geometries for working with a broader range of materials. More resistive materials may be prepared using arrays with interdigitated comb contacts, while materials that are more conductive may be prepared using devices that have a more conventional contact geometry. Alternate electrical measurements such as capacitance and AC impedance could also be implemented using such platforms. The temperature gradient on the legs of the microhotplate is simply an added benefit of the platform employed here, but the legs have not been optimized for a survey-based approach to microstructure optimization. An alternative device with a better-defined gradient could, for example, be developed with a heater in the suspended part of a cantilever, which would provide better thermal isolation. We are also implementing the necessary changes to allow computer interfacing for automating the simultaneous deposition and monitoring of all 16 elements. Once in place, this system will be able to prepare samples either isothermally or using temperature-programmed deposition and will have the capabilities to prepare mixed composition films through the simultaneous use of multiple precursors. Simultaneous electrical probing of all 16 films during growth would also be possible.

Conclusions

We have demonstrated how arrays of microhotplates may be used to investigate efficiently the role that temperature plays in the development of film morphology under isothermal and temperature-programmed deposition conditions. Indeed, the temperature-dependent microstructures obtained using TN provide an excellent example of the power of this microarray approach. It is possible to perform multiple experiments on a single chip, probe the film electrically during deposition, and analyze the samples in an efficient manner. Since all of the experiments may be performed in a short time and without breaking vacuum, this approach also prevents errors caused by longer-term drift or variability in precursor partial pressure, pressure sensor calibration, and mass flow controller calibration. An alternate use for the large arrays would be to use redundant elements for examining reproducibility or for performing annealing studies. Initially developed for use as a gas microsensor, the microhotplate platform is ideal for investigating film microstructure as a function of deposition temperature or for evaluating the effects of postdeposition thermal processing. Although we have not performed them on these samples, microanalytical techniques, such as small area AES or XPS, would certainly add to the arsenal of tools available to the materials research chemist and engineer for studying more complex systems.

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Supporting Information Available: Optical micrographs of illustrating the loss of tungsten from the microhotplate surface as well as supporting EDS data confirming the loss of tungsten (PDF). This material is available free of charge via the Internet at http://pubs.acs.org.

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