Tailoring Residual Stress in Multilayered Polysilicon Produced Near 600°C Expands MEMS Opportunities

When a microelectromechanical system (MEMS) is integrated on a chip with complementary metal oxide semiconductor (CMOS) circuitry, the MEMS components are invariably fabricated first. This sequence is required because the MEMS structures are usually fabricated from polysilicon films that require high-temperature annealing (over 1000°C) to eliminate the residual stresses and stress gradients that would otherwise distort the MEMS structures on their release from the substrate. The shallow junctions in the CMOS elements and metallic interconnects would not survive such a heat treatment. Researchers at Case Western Reserve University have developed a technique to solve this problem; they can produce large-area polysilicon films by low-pressure chemical vapor deposition (LPCVD) using process temperatures that never exceed 615°C and that possess zero stress and zero stress gradients after deposition. This drastically reduced thermal budget allows MEMS components to be fabricated directly on top of CMOS circuitry, leading to enhanced performance and functionality.

The technique, called the MultiPoly™ process,* involves the deposition of polysilicon layers with alternating compressive and tensile residual stresses. The individual layers are engineered to obtain the desired overall film stresses and net stress gradients. It has been known for some time that changing the deposition temperature of LPCVD polysilicon can change the sign of the residual stress. Films deposited between ~550°C and ~590°C display fine-grained, equiaxed microstructures with as-deposited tensile stresses, while films deposited between ~600°C and ~700°C display columnar (110) textured microstructures with as-deposited compressive stresses. The fine-grained microstructure results from the homogeneous nucleation and growth of silicon crystallites within an as-deposited amorphous silicon film; the tensile stresses are generated by the modest volume decrease associated with crystallization (amorphous Si has a slightly lower density than crystalline Si). The columnar microstructure results from the formation of crystalline-silicon films during deposition, in which growth occurs fastest in ⟨110⟩ directions. While the origin of the compressive stresses is not as well understood, it almost certainly involves nonequilibrium point defects in the as-deposited films.

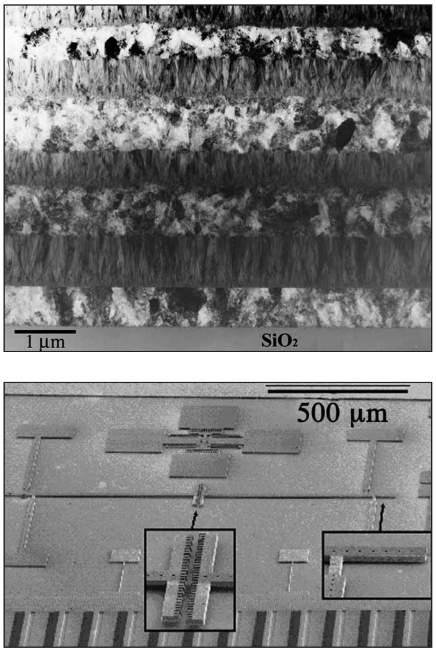

Figure 1. (a) Cross-sectional transmission electron micrograph of a nine-layer MultiPoly film deposited alternately at 570°C and 615°C. Total thickness is 6.3 μm. Layer thicknesses (from substrate to free surface) are 0.67 μm, 0.86 μm, 0.77 μm, 0.64 μm, 0.87 μm, 0.67 μm, 0.57 μm, 0.61 μm, and 0.67 μm, where the five odd layers were deposited at 570°C and the four even layers were deposited at 615°C. (b) Scanning electron micrograph of surface-micromachined devices fabricated from a 10-layer MultiPoly film. The insets show the two ends of a 700-μm-long beam (part of a microstrain gauge); there is no measurable difference in the height of the two ends.

Figure 1a shows a cross-sectional transmission electron micrograph of a nine-layer MultiPoly film deposited on an oxidized Si wafer. The initial layer was grown at 570°C and was amorphous as deposited. The second layer was deposited at 615°C and developed the columnar microstructure characteristic of growth at this temperature. During deposition of the second layer, crystallization of the first layer into its characteristic fine-grained microstructure occurred. The temperature in the LPCVD furnace was lowered again to 570°C for deposition of the third layer. Even though the effective substrate for this layer was columnar polysilicon, deposition of an amorphous material ensued. Subsequent layers were grown by alternating the temperature in the furnace, and upon completion of the deposition, the entire film was subjected to a 2-h anneal at 615°C to ensure complete crystallization of every layer.

Technology Advances provides up-to-date reports of materials developments that show potential to bridge the gap between research innovation and application of advanced materials technologies. If you encounter or are involved with materials research that shows potential for commercialization and would like to present these developments, contact Renée G. Ford, Renford Communications, Ltd., P.O. Box 72, Harrison, NY 10528-0072; tel. 914-967-0955; fax 914-967-7927; or e-mail renford@compuserve.com.

The layer thicknesses in the film shown in Figure 1a were designed to achieve a near-zero overall film stress and stress gradient. However, the overall stress gradient in the film was slightly negative, and upon release, cantilever beams displayed a slight downward deflection. This departure from the targeted overall zero-stress, zero-stress-gradient condition occurred because the stress profiles of the individual layers were not known with sufficient accuracy (furthermore, better control of the silane flow rates in the LPCVD reactor used are also needed). However, the stress gradients within MultiPoly films can be trimmed to the desired specifications by depositing an additional ultrathin layer. Accordingly, a 0.10-μm-thick polysilicon layer was deposited at 570°C to create a 10-layer MultiPoly film with no detectable curvature, as shown in Figure 1b.

While the MultiPoly process has been demonstrated for polysilicon films with near-zero stresses and stress gradients, it is also the case that films can be designed with any value of stress ranging from ~300 MPa compressive (the value for films deposited at 615°C) to ~300 MPa tensile (the value for films deposited at 570°C). Also, a wide range of stress gradients can be achieved if a specific radius of curvature is desired for MEMS components upon release. In addition, while polysilicon was used in this demonstration, any material in which residual stress depends on deposition conditions could be employed.

Opportunities

This process can be used for any MEMS application where integration with electronics is desired. In addition, applications that are contingent on very flat large-area surfaces or predetermined curvatures, such as found in some optical devices, would benefit from this technique. One patent has been issued; a second is expected shortly. The developers welcome inquiries about joint research and development (R&D) projects. Licensing is also available.

Source: For R&D projects: Arthur H. Heuer, Department of Materials Science and Engineering, Case Western Reserve University, 10900 Euclid Blvd., Cleveland, OH 44106-7204, USA; tel. 216-368-3868, fax 216-368-8932, and e-mail heuer@cwru.edu; or Hal Kahn, tel. 216-368-6384 and e-mail kahn@cwru.edu. For licensing: Nick Frollini, tel. 216-368-6124 and e-mail nrf3@cwru.edu.

---

*The MultiPoly process was presented at the Materials Research Society MEMS Materials Issues workshop in April in San Francisco and was covered in a workshop report that appeared in the June 2002 issue of *MRS Bulletin*, p. 466.

Electron-Beam Physical Vapor Deposition Produces Coatings and Net-Shape Refractory Components Using a Single-Step Process

Figure 1. A uniform rhenium coating was applied to 18 graphite balls by placing them into a cylindrical molybdenum wire cage and then rotating the cage in the rhenium vapor above the rhenium melt pool. The diameters of the rhenium-coated balls are ~18 mm.

Alternative cost-effective and robust processes for applying coatings or fabricating net-shape components of refractory materials such as Re, W, HfN (hafnium nitride), HfCN (hafnium carbonitride), and BCN (boron carbonitride) for high-temperature applications are highly sought after, particularly for aerospace uses. At The Pennsylvania State University, researchers have used an industrial-prototype ion-beam-assisted electron-beam physical vapor deposition (EB-PVD) process to produce high-purity rhenium components including rhenium-coated graphite balls (Figure 1), plates, and tubes. They have also fabricated net-shape thrusters by evaporating titanium instead of rhenium. Due to the high flexibility of the EB-PVD process, two mirror-image thrusters were produced simultaneously. It is estimated that the cost of refractory components (such as rhenium) manufactured by EB-PVD would be as much as 50% lower than those currently produced by chemical vapor deposition (CVD) and powder hot isostatic pressing (HIP). The process is robust, with a high degree of flexibility in controlling the composition and microstructure of the components. Unlike CVD, no intermediate machining is required to eliminate a columnar microstructure because a much finer-grained microstructure is formed. Unlike powder metallurgy processes that generally require HIP, no surface machining is necessary to develop the required surface finish.

EB-PVD technology is used by the aerospace industry to apply ceramic and metallic coatings on components; however, such a large coating system has not been available for exploring net-shape components. The main challenge now faced by the researchers is to monitor the *in situ* coating thickness on components at elevated temperatures.

The Penn State EB-PVD model has six electron-beam guns with an average power capacity of 45 kW. The chamber accommodates up to three ingots in a continuous-feed system, with a chamber size of approximately 90 cm × 90 cm × 90 cm. Parts can be manipulated in three dimensions on a computer-controlled rack at up to 14 rpm, with a maximum load of ~20 kg. The maximum component size that can be accommodated is ~40 cm. The main advantage of using an ionized beam during the deposition process is the ability to obtain dense nano- and submicron microstructures.

Opportunities

The Penn State researchers are seeking collaboration on research and development to extend the applications of this technology.

Source: Jogender Singh, Applied Research Laboratory, The Pennsylvania State University, University Park, PA 16804, USA; tel. 814-863-9898, fax 814-863-2986, e-mail jxs46@psu.edu, and Web site www.psu.edu.

Devices that Change Color with an Applied Voltage Developed for Camouflage and Thermal Control

Advanced materials with controllable and highly variable thermal signatures, that is, infrared electrochromism (color change with applied voltage), have recently been sought for two specific applications: military camouflage against both IR sensors and cameras, where there is no current technology; and thermal control for microspacecraft, where technologies currently used for large spacecraft, such as mechanical louvers and heat pipes, do not work. While IR electrochromism is used for large spacecraft, microspacecraft are still new and, as yet, not in orbit. Currently, microspacecraft missions have low heat output and do not require thermal control, but this is expected to change in future missions. Electrochromic devices that satisfy needs in both military camouflage and microspacecraft have been developed by researchers at Ashwin-Ushas Corp. of Lakewood, N.J.

For the military IR-camouflage requirement, protection is sought for armed-forces equipment and personnel (e.g., ships, land vehicles, soldiers) against final-homing IR sensors such as those found on most missiles, and against IR cameras, which operate in the 3–5-μm and 8–12-μm regions. These sensors are different from night-vision goggles and cameras, which are image intensifiers in the near-IR and cannot "see" in complete darkness. In the case of the spacecraft application, when a spacecraft is not exposed to sunlight, it needs to emit less heat to conserve battery power used in its internal heater. It also may need to emit excess heat, for example, when heat from dense internal electronics overheats the spacecraft.

The electrochromic devices comprise a solid electrolyte sandwiched between two electrodes. Each electrode is made up of a gold-plated microporous membrane on which a conducting polymer, an aniline diphenyl amine copolymer, is electrochemically deposited. In the device's IR/visible "light state" at an applied dc voltage of approximately -1.0 V, the conducting polymer is in its reduced state and is highly transparent to IR and visible light, which is thus reflected off of the underlying gold layer. At ~0.0 V, the "IR-dark" state of the device, the conducting polymer is in its partially oxidized state and is nearly opaque to IR light but is partially transmissive to visible light. At ~0.85 V, the conducting polymer is in its fully oxidized state, which is again transparent to IR light but nearly opaque to visible light. This allows for quasi-independent control of visible and IR electrochromism. The performance parameters of interest are the variations in specular and diffuse reflectance across the 2.5–45-µm (IR) and 0.4–0.7-µm (visible) regions, the emittance (integrated emissivity across this IR region), and the solar absorptance. At Ashwin-Ushas, the researchers have demonstrated IR specular- and diffuse-reflectance variations in the 3–5-µm and 8–12-µm regions of >50% (with a range of 5–90%) and emittance variations of >0.53 (range 0.18–0.83), compared with emittance variations of 0.45 for extant mechanical louvers and less than 0.3 for solar absorptance.

The electrochromic device is thin (<0.5 mm), flexible, lightweight (0.16 g/cm^2), of variable area (from 0.5 cm^2 to 0.5 m^2), entirely solid-state, and physically durable. Other properties include light/dark switching times of <2 s, cyclabilities to 10^4 cycles (cycling from light to dark and back one time without degradation), power requirements of 40 µW/cm^2 in continuous operation (4 mW/cm^2 peak transients of <3 s), and low cost. Figure 1 shows an IR-camouflage device, a spacecraft device, and typical IR specular-reflectance electrochromic behavior as a function of applied potential.

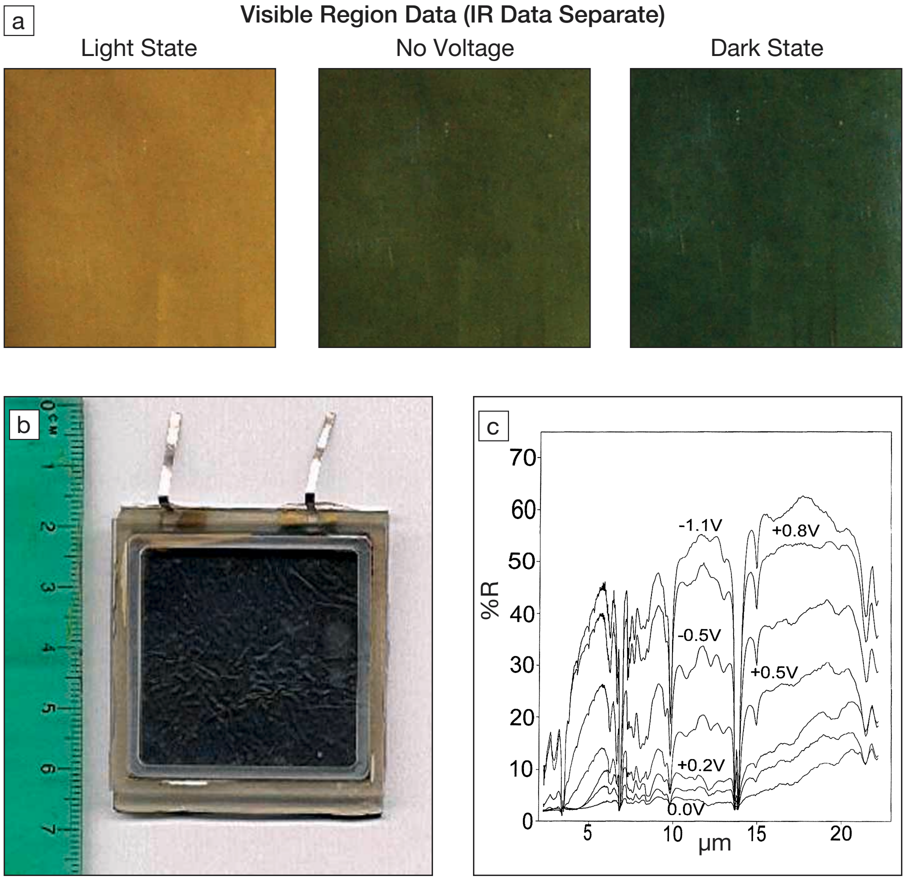

Figure 1. (a) IR-camouflage device, (b) spacecraft device, and (c) typical IR specular-reflectance electrochromic behavior as a function of applied potential.

In the military IR-camouflage application, electrochromic panels are placed on a target that must blend in with the background (i.e., have the same apparent temperature) under various scenarios (e.g., heated/cooled from behind, heated from the front, in total darkness) and against a variety of backgrounds (e.g., foliage, sand, water, sky). Figure 2 shows images taken with a 3–5-µm-range IR camera, demonstrating that the electrochromic device heated from behind can be made to match both the clothing and the background of the person in the photo. In the spacecraft application, the researchers have been working closely with NASA's Goddard Space Flight Center, the Jet Propulsion Laboratory, and the Air Force Research Laboratories at Kirtland and Wright-Patterson Air Force Bases. Devices for spacecraft use have passed space-durability tests: extended exposure in 10^{-6} Torr vacuum to temperatures from -70 to +85°C, solar wind, atomic-O, UV, gamma radiation, electromagnetic impulses, and micrometeoroids.

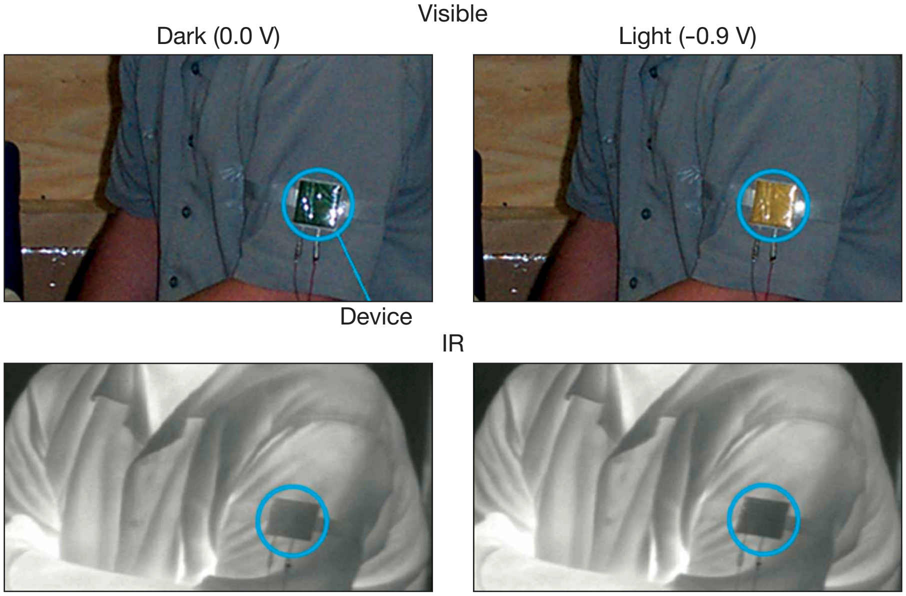

Figure 2. (Top) Visible-region photo, (bottom) IR photo (taken with 3–5-µm range IR camera), of electrochromic device (circled) in IR-light and IR-dark states. Shows excellent match with both background and clothing, suitable for IR camouflage.

Opportunities

Ashwin-Ushas Corp. is interested in licensing or co-production of their patented technology for military IR camouflage as well as for spacecraft applications, where it fulfills an immediate need.

Source: Prasanna Chandrasekhar, President, Ashwin-Ushas Corp., 500 James Street, Unit 7, Lakewood, NJ 08701, USA; tel. 732-462-1270, fax 732-431-2496, and e-mail ashwinusha@aol.com.