

FABRICATION STRATEGIES FOR PRODUCING GRADIENT ROUGHNESS LIBRARIES*

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Introduction

New technologies, such as flexible electronics and functional coatings systems, consist of hierarchical structures fabricated out of combinations of both soft and hard materials, with each material type lending unique properties and performance characteristics to the final integrated system. As these structures and devices become smaller and more complex, materials' interfaces will transition from contributing to overall performance and reliability measures to dominating them. In particular, interfacial properties, such as adhesion, of these hierarchical structures are paramount, as the overall performance of such systems depends on the integrity of the materials interfaces within them. Surface topography plays an important role on controlling interfacial interactions. The critical length scale of roughness depends on the application area and can span from the micro- to nano-scale.

We present here fabrication strategies for creating gradients in roughness having continuous changes in both the dominant period and amplitude of the surface topology. A versatile method for creating roughness gradients is through surface wrinkling. Here, a metal film is evaporated onto the surface of a thin polymer film supported on a substrate. Heating the films causes the surface to wrinkle due to the mismatch in thermal expansion of the polymer and metal layer.¹ The length scale of roughness is controlled by the film thickness of metal and polymer layers, the heating temperature, and annealing time.

Experimental²

Polystyrene ($M_n=600,000$ g/mol, polydispersity 1.09, Polymer Source) (PS) was used as the underlying polymer film. A PS solution in toluene was spin-coated onto a clean silicon wafer. The PS film thickness as measured by interferometry was $292 \text{ nm} \pm 3 \text{ nm}$.³ Approximately 30 nm of aluminum were thermally evaporated onto the surface of the PS-coated wafer. The surface of the wafer reached a maximum temperature of $46 \text{ }^\circ\text{C}$ during the evaporation process, as indicated by a Therman indicator.

To induce surface wrinkling, the Si/PS/Al specimen was placed on a temperature gradient stage.^{3,4} The surface temperature of the sample ranged from $130 \text{ }^\circ\text{C}$ to $93 \text{ }^\circ\text{C}$, as measured by a surface thermocouple (Omega Engineering, Inc) held in contact with the sample surface using a spring-

loaded lever. The sample was annealed on the temperature gradient stage for 1 min and then quenched down to room temperature. The wrinkled surface morphology was imaged using atomic force microscopy (DI Nanoscope 3100, Veeco Instruments) (AFM) operating in tapping mode.

Results and Discussion

Figure 1 shows AFM micrographs of the wrinkled surface. Visually, the surface of the sample immediately becomes hazy for the portion of the sample above the glass transition temperature (T_g) of PS ($T_g \approx 100 \text{ }^\circ\text{C}$), indicative of the onset of a wrinkled morphology on the surface of the sample. However, the AFM images show that wrinkling occurs even at temperatures below the T_g of PS, indicating that the critical strain for wrinkling can be reached even at temperatures below T_g . Further studies are being conducted to determine the effects of film thickness, annealing time, and type of polymer used. Combining this technique and others for creating gradient roughness over a range of length scales presents a valuable tool for screening interfacial properties, including adhesion, surface energetics, and defect identification and mitigation.

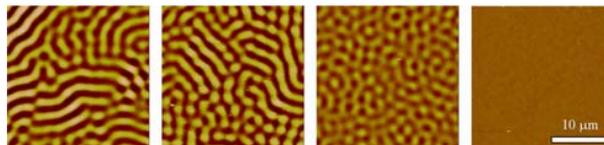


Figure 1. AFM micrographs of a wrinkled PS/Al film generated by annealing the film on a temperature gradient. Temperature decreases left to right from $124 \text{ }^\circ\text{C}$ to $74 \text{ }^\circ\text{C}$.

References

1. P. Yoo and H.H. Lee, *Macromolecules*, **2005**, *38*, pp. 2820-2831.
2. Equipment and instruments or materials are identified in the paper in order to adequately specify the experimental details. Such identification does not imply recommendation by NIST, nor does it imply the materials are necessarily the best available for the purpose.
3. The error bars represent one standard deviation of the data, which is taken as the experimental uncertainty of the measurement.
4. J.C. Meredith, A.P. Smith, A. Karim, E.J. Amis, *Macromolecules*, **2000**, *33*, pp. 9747-9756.
5. A. Sehgal, A. Karim, C. Stafford, M. Fasolka, *Microscopy Today*, **2003**, *Sept-Oct*, pp. 26-29.

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