

Towards Femtogram-Scale Materials Discovery using Scanning Probe Lithography

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This work explores recent progress in transforming scanning probe systems into platforms for materials discovery studying samples approaching the femtogram scale. There is a need to discover advanced materials to address the pressing challenges facing humanity, however there are far too many combinations of material composition and processing conditions to explore using conventional experimentation. One powerful approach for accelerating the rate at which materials are explored is by miniaturizing the scale at which experiments take place. Scanning probe lithography has the dual benefits of being capable of patterning nanoscale features and providing a system for measuring their properties at a commensurate scale (Figure 1).¹ However, before this potential can be realized, methods must be developed for controllably patterning and mixing samples at the nanoscale. Thus, the focus of this talk is recent progress in developing new methods for ultra-miniaturized combinatorial materials research using scanning probes. To begin, we describe the use of inertial sensing as a path to quantifying the amount of fluid on the probe and thus the amount of fluid transferred when patterning. This method allows us to study the fluid transport between a tipless scanning probe and a surface to develop relationships that allow for predictable control over feature size. This process is used to realize closed-loop patterning of fluids with better than 1% mass accuracy.² This level of control also allows us to attach metal-organic framework (MOF) crystallites to probes and study MOF-polymer interactions with high throughput.³ Following these innovations, we explore the use of ultrafast probes together with spherical tips that allow for patterning of fluids down to the femtogram scale. Finally, we show that these methods allow one to mix fluids on a surface to realize compositional gradients of materials. We close with a demonstration of a combinatorial experiment in which a scanning probe is used to prepare a gradient of polymer composition and then perform mechanical testing to functionally read out these materials. Taken together, these advances set the stage for scanning probes to function as a single system for materials discovery.

1. K. A. Brown, *Matter* **5** (10), 3112–3123 (2022).
2. V. Saygin, B. Xu, S. B. Andersson and K. A. Brown, *ACS Appl. Mater. Interfaces* **13** (12), 14710–14714 (2021).
3. J. M. Palomba, V. Saygin and K. A. Brown, *Chem. Commun.* **59**, 290-293 (2023).

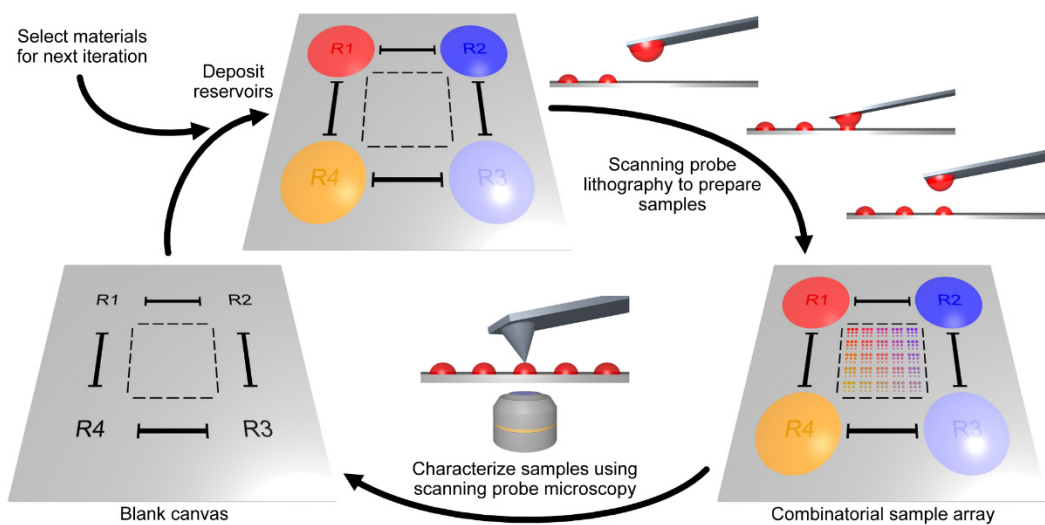


Figure 1. Schematic showing closed-loop materials discovery using a scanning probe. In this example, a scanning probe is used to produce an array of samples that are mixtures of four reservoirs. Subsequently, the same system can be used to functionally interrogate these samples. This process can be iterative with feedback from a round of experiments informing the selection of materials and compositions in the next round.