

# Electro-liquefaction of Cr Thin Films for Application in Scanning Probe Lithography

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On electrical stressing of nanometer-thick Cr films using a pair of sharp electrodes, a liquid material appears to flow out of the cathode probe, which spreads to over 100  $\mu\text{m}$  in diameter within a few seconds<sup>1</sup> (Fig. 1(a)). Various tests have confirmed that the liquid material is composed of chromium trioxide ( $\text{CrO}_3$ )<sup>2</sup>. The liquefaction process of Cr films being an electric field-induced process, a study of the electrical characteristics of the oxide domain was warranted. Impedance spectroscopy revealed a frequency-dependent capacitive nature of the said domain<sup>2</sup>. The charging time constant of the equivalent circuit helped to estimate the minimum pulse width ( $\sim 0.3$  ms) required for an expansive oxidation process<sup>2</sup> (Fig. 1(b)). This in turn helped us to reason that alternating electric fields could play a very dominant role in the said process. While investigating the effect of various AC signal parameters: frequency, DC shift and duty cycle, it is revealed that all the parameters play a massive role and can be tuned to confine the electro-oxidation process<sup>3,4</sup> (Fig. 1(c-e)). A detailed investigation into the effects of the individual half cycles revealed that the negative half is dominant, and the entire process is limited by the transport of oxyanions (hydroxide ions) to the periphery of the oxide domain<sup>4</sup> (Fig. 1(f)).

Ambient parameters also have a significant effect on the process. If the temperature is kept constant, a 50% increase in RH can result in the extent of the oxide spread increasing by almost 5 times<sup>5</sup>. On the other hand, a 20  $^\circ\text{C}$  increase in temperature can reduce the extent of oxidation by almost 3 times<sup>6</sup>. Thus, the combined effect of RH and temperature on the oxidation process is noteworthy (Fig. 2). However, if the cathode is traversed along a predetermined path, necessary patterns can be obtained on the Cr layer. Since  $\text{CrO}_3$  has a high solubility in water, the formed oxide can be easily developed/removed, and the pattern transfer process is also quite easy. This has led to the development of the scanning probe-based electrolithography (ELG) technique<sup>7</sup> (Fig. 3(a)). By introducing a polymer sacrificial layer, patterns created on the Cr layer can be easily transferred onto a material of choice. On implementing ELG using nanoprobe, a line width of 9 nm has been obtained on the polymer layer (Fig. 3(b)), while parallel lines of width 100 nm have been successfully transferred (Fig. 3(c)), demonstrating the suitability of ELG as a nanolithography process<sup>7</sup>. Using micron-sized probes, a 45% increase in RH can result in the resulting line width increasing from 3.9  $\mu\text{m}$  to 20.5  $\mu\text{m}$ <sup>5</sup> (Fig. 3(d)).

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<sup>1</sup> S. Talukder, P. Kumar, and R. Pratap, *IEEE Trans. Electron Devices* **60**, 2877 (2013).

<sup>2</sup> S.N. Ghosh and S. Talukder, *IEEE Trans. Nanotechnol.* **22**, 584 (2023).

<sup>3</sup> S.N. Ghosh, *et al.*, *Nanotechnology* **32**, 315304 (2021).

<sup>4</sup> S.N. Ghosh and S. Talukder, *IEEE Trans. Nanotechnol.* **22**, 606 (2023).

<sup>5</sup> S.N. Ghosh and S. Talukder, *Nanotechnology* **34**, 95302 (2023).

<sup>6</sup> S.N. Ghosh and S. Talukder, *Phys. Scr.* **99**, 15011 (2024).

<sup>7</sup> S. Talukder, P. Kumar, and R. Pratap, *Sci. Rep.* **5**, 17753 (2015).

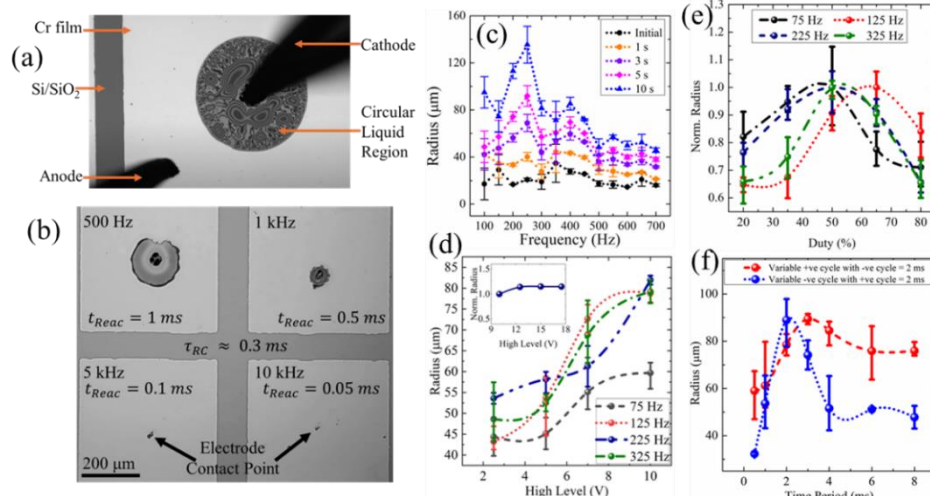


Figure 1: (a) Optical micrograph of a liquid region forming around the cathode; Effect of varying the (b) pulse width, (c) frequency, (d) DC shift, (e) duty cycle, and (f) half time cycles of an electrical signal on the spread of the oxide region.

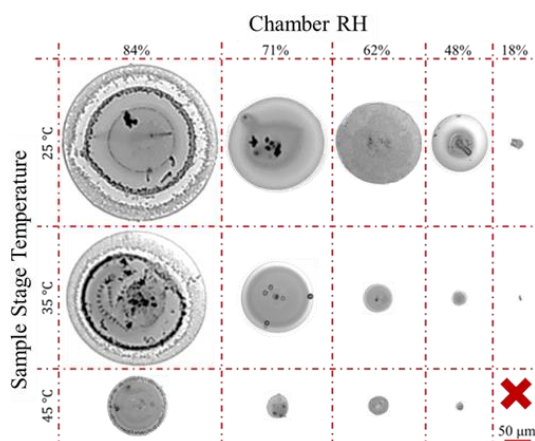


Figure 2: Combined effect of varying the relative humidity and temperature on the extent of oxide spread.

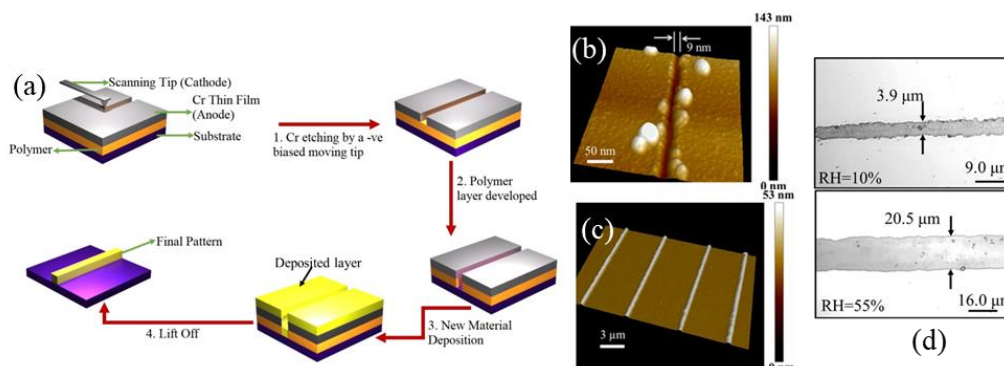


Figure 3: (a) Schematic workflow of the electroplating (ELG) technique for pattern transfer; (b) a 9 nm wide line formed on the polymer layer; (c) parallel lines 100 nm wide transferred to titanium; (d) effect of RH on the formed line patterns keeping other factors constant.