Process condition dependence of liquid-phase focused electron beam induced etching of copper

S. K. Lami, G. Smith, E. Cao, and J. T. Hastings
Electrical and Computer Engineering, Univ. of Kentucky, Lexington, KY 40506
todd.hastings@uky.edu

Focused ion beam induced etching of copper is routinely used for integrated circuit edit and debugging. However, this approach is limited by factors such grain orientation dependence, redeposition, ion implantation, low selectivity to dielectrics, and sample damage. Liquid-phase electron beam induced etching (LP-EBIE) of copper may overcome many of these drawbacks, and we previously demonstrated LP-EBIE copper films in aqueous sulfuric acid.[1] However, the effects of liquid thickness, beam energy, and beam current have not been quantified. Here we show that these parameters strongly influence the etch process and that their dependencies can be understood from a radiation-chemical model of beam-induced etching in liquids.

Samples representative of an upper-level copper interconnect layer ($1\mu m$ thick Cu on SiO_2 with a standard barrier layer) were etched in an FEI Quanta 250 FEG environmental SEM. Pressure and temperature were controlled to maintain a stable drop of aqueous sulfuric acid with a concentration of ~5M. An *in-situ* optical interferometry system enabled the measurement of liquid thickness vs. position as shown in Fig. 1a. Etching was accomplished by allowing the electron-beam to dwell on single spots using beam energies from 10 to 30keV and beam currents from 0.1 to 6 nA. All etches selectively terminated on the barrier layer as shown in Fig. 1b. The average etch rate, plotted in Fig. 2, was calculated from the measured volume of the features and the dwell time.

Electron energy loss verses position in the liquid was simulated with NIST's JMONSEL Monte Carlo code.[2] The output was used in COMSOL Multiphysics to model radiolysis processes, subsequent reactions, mass transport, and etch geometry. This hybrid model predicts a sublinear increase in etch rate with beam current as observed experimentally (Fig. 2a). This is not the result of depletion of the reactants, but rather from the changing flux of oxidizing species as the etched features grow. The model also predicts a complex interaction between beam energy and liquid thickness, and this is observed experimentally as well (Fig. 2b). This relationship would be expected as the beam energy alters both the penetration depth of electrons in the liquid and the total energy transferred to the liquid. The results suggest that copper is etched by oxidizing species from the radiolysis of sulfuric acid (SO_4^- and $S_2O_8^{2-}$). As a result, there will be a tradeoff between the resolution and rate of etching as one changes beam energy and liquid thickness.

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[1] L. Boehme et al. Nanotechnology **49**, 26 (2015).
[2] J.S. Villarrubia et al., Ultramicroscopy **154**, 15 (2015)

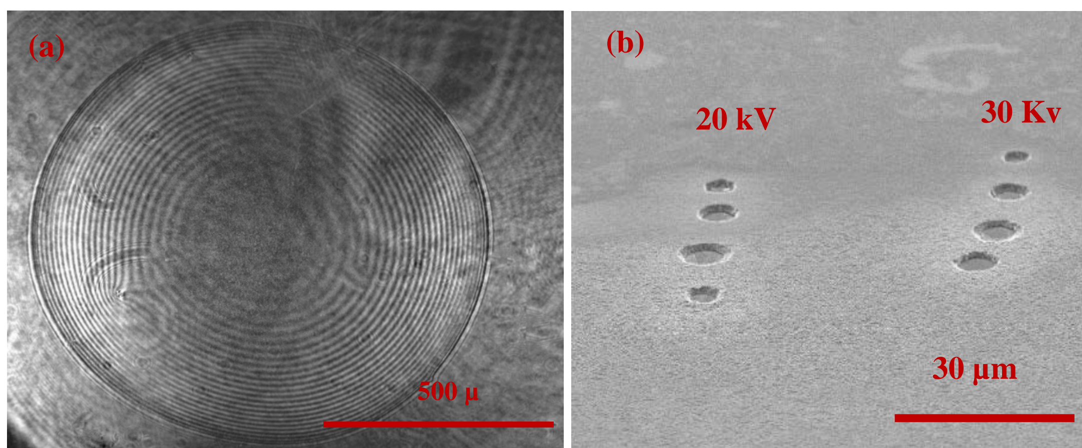

Figure 1: (a) *In situ* interferogram of 5M H_2SO_4 (aq) droplet on Cu substrate at λ= 660 nm. (b) An *ex situ* micrograph of 360 sec. spots scanned etched into ~1µm electroplated Cu at 30 and 20 kV at a different liquid thickness (0.2-1µm).

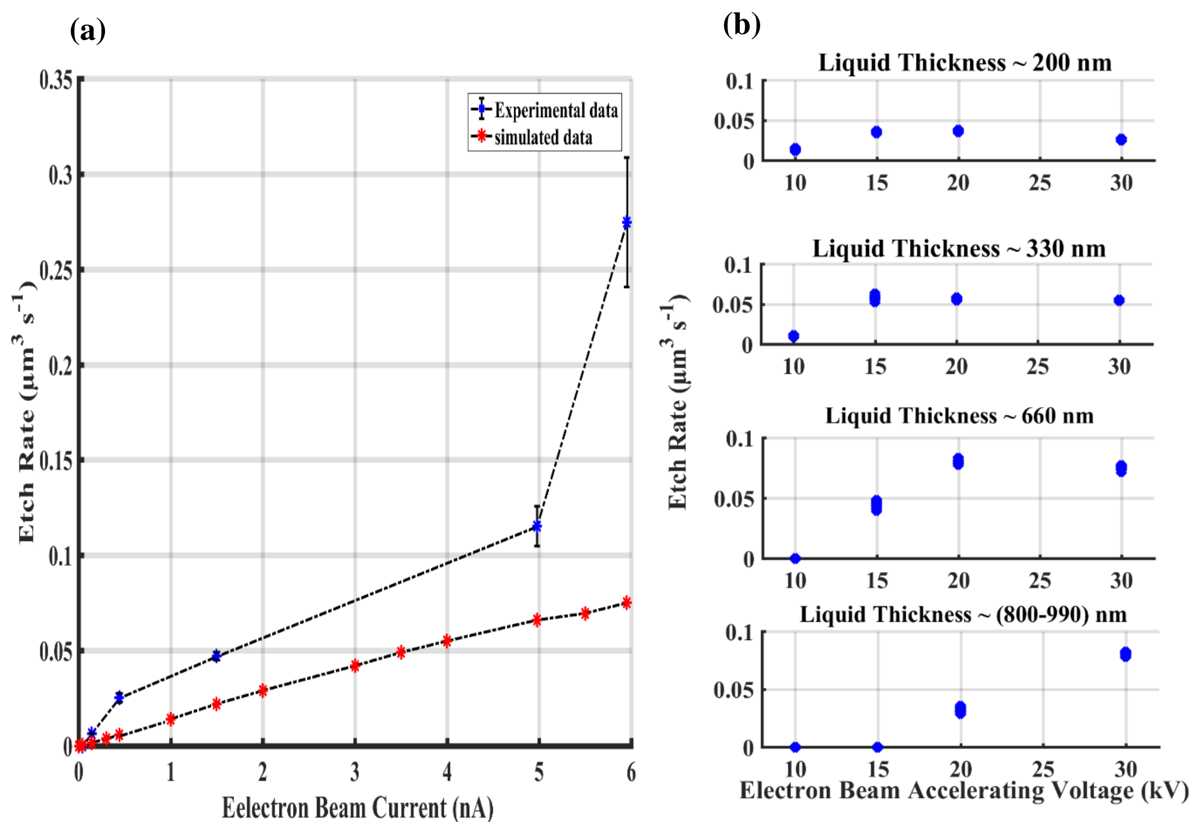

Figure 2: Volumetric etch rate of 1µm-thick electrodeposited Cu versus (a) beam current and (b) both liquid thickness and beam energy. Experiments and simulations in (a) considered a 330-nm thick H2SO4 (aq) layer, a beam energy of 30 kV, and a dwell time of 180 s. Experiments in (b) used a 360 s dwell time and beam currents ranging from 0.40 to 0.44 nA.