Fabrication and Characterization of the Capillary Performance of Superhydrophilic Cu Micropost Arrays

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Abstract—We report the fabrication of dense arrays of superhydrophilic Cu microposts at solid fractions as high as 58% and aspect ratios as high as four using electrochemical deposition and chemical oxidation techniques. Oxygen surface plasma treatments of photoresist molds and a precise control of the initial electrodeposition current are found to be critical in creating arrays of nearly defect-free Cu posts. The capillary performance of the micropost arrays is characterized using capillary rate of rise experiments and numerical simulations that account for the finite curvatures of liquid menisci. For the given wick morphology, the capillary performance generally decreases with increasing solid fraction and is enhanced by almost an order of magnitude when thin nanostructured copper oxide layers are formed on the post surface. The present work provides a useful starting point to achieve optimal balance between the capillary performance and the effective thermal conductivity of advanced wicks for micro heat pipes.

Index Terms—Cooling, electrochemical deposition, micro heat pipe.

I. INTRODUCTION

THE THERMAL management of high-power-density semiconductor devices, such as microprocessors, power amplifiers, power conditioning/switching devices, and lasers, has been a subject of intense research over the past decades. Heat pipes have proven to be one of the most attractive passive cooling devices as they have the potential to handle very high heat fluxes and offer effective thermal conductivities exceeding even that of diamond.

A number of previous studies reported micro heat pipes [2], [3]. Compared with conventional heat pipes made of round Cu tubes, planar micro heat pipes can be readily integrated into compact electronic systems. The majority of past micro heat pipes, however, incorporated simple linear grooves, channels, or meshes that offer limited capillary performance as wicks. This led to low heat flux capability, very often below 100 W/cm², and strong sensitivity to orientation with respect to gravity.

Some previous micro heat pipes used sintered metal powders to achieve much higher critical heat fluxes [4]. Direct sintering of metal powders, however, usually requires high and tightly controlled process temperatures and ambient conditions. As a result, it is often suited only for heat pipes whose envelopes are made of select ceramics or metals. In particular, sintering is not compatible with polymer-based heat pipes that can be directly integrated into printed circuit boards for highly volume- and cost-efficient thermal management solutions. The effective wick thermal conductivity of sintered powders is also often limited due to small contact areas between spherical powders and heating surfaces. Alternative low-temperature fabrication approaches for advanced wicks are therefore desired.

Another important consideration is wick materials. Recent studies [5] reported microfabricated Ti/TiO₂ micropost arrays for possible applications in micro heat pipes. The heat transfer performance of Ti micropost wicks, however, is expected to be limited by the low thermal conductivity of Ti.

Copper has been one of the most attractive materials for heat transfer applications due to its high thermal conductivity and favorable processing properties. The thermal conductivity of Cu is almost twice that of pure Al and an order of magnitude higher than that of Ti. Poor wettability of water on copper surfaces, however, may compromise their phase change heat transfer performance [6], [7]. The static contact angle of air-exposed Cu is often greater than 70°.

The maximum heat flux capability of micro heat pipes is most often determined by the capillary limit [2], [3], [8]

\[
Q = 2M(K/R_{eff})A_w/L_{eff}.
\] (1)

Here, \( R_{eff} \) is the effective pore radius \( (= r_p/\cos\theta_s) \), \( K \) is the permeability, \( L_{eff} \) is the effective heat pipe length, \( A_w \) is the cross-sectional area of the wick, and \( M \) is the merit number of the heat transfer fluid. Maximizing the ratio \( K/R_{eff} \) is a key design target for advanced wicks. A small pore radius leads to high capillary pressure, but it also decreases the permeability. The capillary performance parameter \( K/R_{eff} \) captures a tradeoff between these two competing effects. Improving the wettability and, hence, reducing the contact angle \( \theta_s \) are one important approach to increasing the capillary performance parameter and, hence, the critical heat flux.

In this paper, we report the fabrication and characterization of the capillary performance of superhydrophilic (contact angle < 10°) wicks that consist of dense arrays of nanostructured...
Cu microposts with solid fractions as high as 58% and aspect ratios as high as four. We develop and optimize electroplating and related microfabrication methods to synthesize uniform and nearly defect-free micropost arrays over areas as large as 3 cm². Defects such as residual plating molds and missing or partially formed posts can be detrimental as they can create local hot spots and initiate catastrophic dry-out. A controlled chemical oxidation scheme is used to nanostructure the surfaces of the Cu posts to enhance their wettability.

We perform capillary rate of rise experiments in conjunction with numerical simulations to characterize the capillary performance of micropost wicks of various feature sizes. The shape of liquid menisci is predicted using the energy minimization algorithm, which is then used to predict the wick permeability. The capillary pressure and, hence, the effective pore radius are then calculated from the measured wick capillary performance parameter $K/R_{eff}$.

Although we focus on heat pipes as our primary target applications in this paper, details of the microfabrication processes presented here can be applied to the fabrication of other devices and applications where dense uniform arrays of metallic microposts are necessary.

II. FABRICATION OF Cu MICROPOROUS ARRAYS

A. Microfabrication Processes

We fabricate dense hexagonal arrays of copper microposts with diameters of 30–100 μm, pitches of 62.5–115 μm, and heights of 50–120 μm using electrochemical deposition processes. Fig. 1 summarizes the process flow. We note that a recent study [9] reported Cu micropillar structures to serve as liquid feeds for micro-heat-pipe wicks. The aspect ratio and solid fraction of these Cu micropillars, however, were very low as they were fabricated using isotropic wet etching.

**Seed Layer Deposition:** We first deposit a seed layer (Ti/Cu/Ti) on a silicon substrate using e-beam thermal evaporation (CHA Mark 40). The bottom Ti layer (30 nm) serves as an adhesion layer. The Cu layer (350 nm) is the main seed layer. The top Ti layer (30 nm) protects the seed layer from oxidation during soft/post exposure bake and oxygen plasma treatments of the photoresist (PR) layer. The top Ti layer also improves adhesion with the PR layer.

**PR Spin Coating and Patterning:** We use lithographically patterned negative PR layers as molds [10]. PR-based molds are advantageous as they can be readily applied to most common substrates. Unlike interconnects [11]–[13], however, micropost wicks require PR molds to be completely removed after plating. Past efforts on the fabrication of dense electrode arrays for microbatteries [14] used expensive silicon molds that are chemically dissolved away after electroplating.

We choose KMPR (Microchem Inc.) rather than more widely used SU-8 as our PR. KMPR (Microchem Inc.) is a chemically amplified negative-tone epoxy-based PR, recently developed to address process challenges of SU-8, including poor adhesion [15] and lack of a chemical dissolver.

To create a mold of thickness approximately 140 μm, we double coat KMPR 1050 at 2000 r/min for 30 s. Two-step soft bake (65 °C for 5 min followed by 100 °C for 20 min) is conducted after each spin-coating step to reduce thermal stress. The PR layer is then exposed to UV illumination (Karl Suss’ MA6) at a dose of 8 mJ/cm² for ~170 s and baked again at 65 °C for 5 min and at 100 °C for 5 min. The PR layer is developed inside a sonication bath to define dense through-hole array patterns.

**Wettability Enhancement of Plating Molds:** When the electrolytes do not wet even a fraction of through holes in a mold, the resulting nonuniform current spikes can lead to the formation of metal particulates [Fig. 2(a)] and significant spatial nonuniformity in electrodeposition rates. Posts formed under current spikes also have poor mechanical integrity and readily detach from the substrate during PR mold removal processes [Fig. 2(b)].

The contact angle of a commercially available Cu plating solution (Technic Inc.) on as-baked KMPR layers is quite high (~80°) as shown in Fig. 3(a). We subject our molds to oxygen plasma in a commercial reactive-ion-etching tool (Technics Fluorine RIE 800, 150 W, 200 mtorr) for 2–10 min. After the plasma treatment, the contact angle is reduced below 10°.
Fig. 3. Optical images of droplets (4 μL) of the electroplating solution on the PR layers: (a) Before plasma treatment, (b) 24 h after 2-min plasma treatment, and (c) 10-min treatment.

Similar plasma treatments have been extensively used for polydimethylsiloxane (PDMS) surfaces [16], [17]. Unlike PDMS, the enhanced hydrophilicity is maintained for over two days [Fig. 3(b) and (c)]. The slow recovery may be explained by the dense molecular structure of cured KMPR layers, which limits the migration of molecules from the bulk phase.

Electrochemical Deposition: We first remove the protective Ti layer under the though holes using a diluted (1%) solution of hydrofluoric acid. The sample is then dipped in a 2-M hydrochloric acid solution to remove the native oxide layer formed on the Cu seed layer. The sample is sonicated in a plating bath for approximately 30 s to facilitate wetting of through holes.

To minimize void formation, which degrades thermal and mechanical properties, a bottom-up plating scheme is applied [12], [18]. We find it important to initiate electrochemical deposition using a very low current density (~2 mA/cm²). Such a low current density helps mitigate undesired effects of nonuniform current density distributions, which may be caused by residual oxide layers or contamination. A low initial current density also facilitates the formation of good electrical and mechanical contacts between the deposited copper and the seed layer by minimizing void formation. Once electrodeposition is activated in all holes, the current density is increased (~7 mA/cm²) to achieve higher deposition rates. Fig. 4 shows SEM images of final copper micropost arrays after the mold is stripped away.

B. Nanostructuring of Cu Surfaces

Controlled chemical oxidation is a convenient way to modify the wettability of Cu surfaces. Among various possible CuO nanostructures, sharp needle-like CuO nanostructures are chosen based on our previous comparative study [19]. The unique morphologies and high surface energy of CuO nanostructures allow us to achieve extreme wettability without introducing large parasitic thermal resistance. Since the oxidation process is quasi-self-limiting, uniform oxide nanostructures can be formed over complex microstructures. The static contact angle of water on flat Cu films incorporating these nanostructures was measured to be below 10°. The contact angle of water on a nominally smooth CuO surface was estimated to be approximately 46°, much higher than that on the nanostructured Cu surface. Nanostructuring is therefore essential to achieve the level of enhancement in the wettability we observe.

The nanostructures are formed by immersing the micropost samples in an alkali solution composed of NaClO₂, NaOH, Na₃PO₄ · 12H₂O, and deionized (DI) water. A thin (< 200 nm) Cu₂O layer first covers the entire surface of the microposts, and then, sharp needle-like CuO nanostructures are grown on this layer. The thermal resistance of the thin underlayer is estimated to be only on the order of 10⁻⁷ m²·K/W. Fig. 5(a) shows CuO nanostructures uniformly formed over the entire surface of the Cu microposts.

Fig. 5 also shows the images of water droplets placed before (b) and after (c) the chemical oxidation. Due to the extreme wettability of the CuO nanostructures, the poorly wetting copper post wicks are turned into superhydrophilic wicks. The water spreads in a hexagonal shape because the closest neighboring posts are located 60° from the main axes of the hexagonal array.

III. CHARACTERIZATION OF THE CAPILLARY PERFORMANCE OF NANOSTRUCTURED Cu MICROPPOST ARRAYS

A. Capillary Rate of Rise Experiment

The critical heat flux of micro heat pipes is most often determined by the capillarity performance of their wicks. Many
If the gravity effect is negligible, (2) reduces to Washburn’s equation [22]

$$x^2 = \frac{2\sigma}{\varepsilon \mu} \left( \frac{K}{R_{\text{eff}}} \right) t.$$  

We note that the capillary rise phenomena occurring in our micropost wicks are rather complex on microscale, involving a set of distinct liquid fronts moving vertically in a discontinuous manner while interacting/merging with each other in the horizontal direction. Equation (2) is meant to describe averaged macroscale behavior of the liquid front rather than these microscopic details.

Fig. 6(a) shows selected video frames from one of the capillary rates of rise experiments. When a wick sample touches the liquid reservoir, a macroscopic side meniscus is first formed [Fig. 6(b)]. The maximum height of such a meniscus in our samples is measured to be approximately 4 mm for water. These values agree well with the predicted values based on the balance between the gravity and the capillary force [23]

$$h_0 = \sqrt{\frac{2\sigma}{\rho g} \left(1 - \sin \theta_\alpha\right)^{1/2}}$$  

(4)

where $\theta_\alpha$ represents the apparent contact angle of the wick surface. The height of the macroscopic meniscus ($h_0$) is subtracted from the liquid rise height ($h$) and the associated transient time ($t_0$) from the original measurement time ($t$) in our analysis as shown in Fig. 6(b).

The measured liquid rise height is fitted using (2) to determine the parameters $K/R_{\text{eff}}$ and $K$. The goodness of fit is evaluated by calculating the mean absolute deviation (MAD) defined as the average value of \(|(h - h_0)/t_0|\), where $h$ is the measured value and $t_0$ is the calculated value. Fig. 6(c) shows the rise height as a function of time with the best fit.

**B. Prediction of the Permeability**

When the gravity effect is relatively weak, which is the case for our 3-cm-long samples, the liquid rise height at a given time is a strong function of the ratio $K/R_{\text{eff}}$ but only a weak function of the individual values of $K$ or $R_{\text{eff}}$ [see (3)]. Even when we change the value of $K$ by $\pm 40\%$ from its best fit value, for example, the MAD changes less than $1\%$ if we fix $K/R_{\text{eff}}$ by changing $R_{\text{eff}}$ by the same factor. Such large uncertainty was overlooked in many previous studies. While the accuracy of $K$ (or $R_{\text{eff}}$) can be improved by using much longer samples, evaporation makes it difficult to track liquid menisci over a length $> 10$ cm.

We instead predict the permeability $K$ using numerical simulations and combine it with the experimentally measured ratio $K/R_{\text{eff}}$ to determine $R_{\text{eff}}$. We first predict the shape of liquid menisci in each micropost wick sample using Surface Evolver [24], which is based on an energy minimization algorithm. In this algorithm, the shape of a meniscus, as defined by a set of vertices, is iterated down the surface energy gradient by calculating the virtual force on each vertex as the negative gradient of the energy with respect to position. The iteration is continued until the vertex positions converge to the values that
result in the minimum total surface energy. Fig. 7 shows the predicted shapes of liquid menisci for the present post wicks. The experimentally measured contact angle on the nanostructured Cu is used as an input parameter.

The shape of each predicted liquid meniscus is next imported into a commercial finite-element simulation package. Fig. 8 shows the unit cell and the boundary conditions for the Navier–Stokes equations. The microposts are assumed to be smooth cylinders. The no-slip boundary condition is applied on all the solid surfaces, and the symmetry condition is applied on the side walls. Since the viscosity of air is much smaller than that of water, we assume that the shear stress on the liquid meniscus is negligible. The deflection of the liquid meniscus from its equilibrium shape is also ignored. The normal velocity and the gradients of the tangential velocity in the normal direction are therefore both set to be zero on the meniscus surface. A periodic boundary condition is applied at the inlet and outlet. The pressure difference between the inlet and outlet is set to match the average flow rates observed in the experiments.

We solve the steady incompressible Navier–Stokes equations to predict full 3-D velocity profiles and then compute the average velocity across the inlet. From the predicted average velocity ($\bar{v}$), the permeability is then determined using Darcy’s law

$$\bar{v} = -\frac{K}{\mu} \frac{dP}{dx}. \quad (5)$$

IV. RESULTS AND DISCUSSION

A. Effect of Nanostructuring

Nanostructuring Cu microposts using controlled oxidation leads to enhanced wettability and, hence, improved effective capillary pressure. Substantial increase in the surface area, however, may lead to increase in the surface friction and, thereby, degradation in the permeability of nanostructured wicks. The capillary pressure and, hence, the pore radius $r_p$ are determined primarily by the diameter and spacing (tens of micrometers for the present wicks) and, therefore, are much less affected by submicrometer-scale roughness.

To quantify the net benefit of nanostructuring, we perform capillary rate of rise experiments before and after nanostructuring. Since the contact angle of water on smooth Cu is too high to observe any spontaneous capillary rise, we use methanol as the working fluid for wick samples with smooth Cu microposts (i.e., before nanostructuring). The contact angle of methanol on smooth Cu surfaces ($\sim 5^\circ$) is comparable to that of water on nanostructured Cu surfaces. Fig. 9 shows the rising height data for a wick sample before and after nanostructuring. The rising speed of methanol on the smooth wick is smaller than that of water on the nanostructured wick because methanol has a smaller value of the surface tension to viscosity ratio than water.

The capillary performance parameter ($K/R_{eff}$) extracted from the best fits is shown in Fig. 10. To facilitate comparison, we convert the results obtained for methanol into equivalent results for water (labeled as “prediction” in Fig. 10) using the measured contact angles ($R_{eff} = r_p / \cos \theta_s$). We first note that the capillary performance (for water) of the nanostructured wick is indeed enhanced by almost an order of magnitude compared with the nominally smooth wick. For the nanostructured wick, where we use water to perform direct measurements, the
predicted result matches well with the experimental data. This shows that degradation in permeability due to nanostructuring is relatively minor. This is consistent with the results of our recent experimental work [25] where we noted that the terminal velocity of falling Cu spheres under Stokes flow conditions changed little before and after nanostructuring.

B. Effect of Micropost Height

We next determine the permeability and capillary pressure of hexagonal arrays of microposts with a diameter of 50 μm, a pitch of 100 μm, and heights varying from 50 to 120 μm. Fig. 11(a) shows the permeability values at different heights calculated by numerical simulations. The permeability initially increases rapidly as the post height increases and then approaches a plateau corresponding to the 2-D limit. Such height dependence (or 3-D effect) originates mainly from the viscous force exerted on the liquid by the substrate. Since the effective pore radius is relatively insensitive to the post height, the ratio $K/R_{\text{eff}}$ increases with increasing post height [Fig. 11(b)].

To clarify the effect of finite meniscus curvatures on the wick permeability, we perform another set of simulations where we assume that the liquid menisci are flat [Fig. 11(a)]. For the 100-μm-tall post array, the finite curvature leads to a decrease in the permeability by approximately 15%. The effect becomes more pronounced for shorter posts.

Although increasing $K/R_{\text{eff}}$ is beneficial for achieving high capillary limits, thick liquid layers may increase the overall thermal resistance of the wick. All subsequent characterization in the present study is performed for posts with height of approximately 100 μm based on our preliminary heat transfer modeling study.

C. Effect of Post Diameter and Pitch

To investigate the effects of post diameter and pitch on the capillary performance, two sets of samples are fabricated as summarized in Table I. In case I, the diameter of the post $D_p$ is fixed at 50 μm, and the pitch $P$ is varied by changing the distance between the nearest neighboring posts ($D_{cc}$). In case II, the distance between the nearest neighboring posts is fixed at 50 μm, while the post diameter is varied from 30 to 100 μm.

Fig. 12 shows the experimentally measured capillary performance parameter $K/R_{\text{eff}}$ of the micropost wicks listed in Table I as a function of solid fraction $f_s$. For the present hexagonal arrays

$$f_s = \frac{\pi}{2\sqrt{3}} \left( \frac{D_p}{P} \right)^2.$$  

Fig. 13 shows the permeability and effective pore radius obtained for the same set of wicks.

Fig. 11. Effect of the micropost height on the permeability and the capillary performance parameter $K/R_{\text{eff}}$ (50-μm diameter, 100-μm pitch, and 50–120-μm height). (a) Predicted permeability $K$ (solid line) with and (dashed line) without considering the finite curvatures of the meniscus. (b) $K/R_{\text{eff}}$ and $R_{\text{eff}}$ as a function of the post height.

**TABLE I**

<table>
<thead>
<tr>
<th>Case</th>
<th>$D_p$ [μm]</th>
<th>$D_{cc}$ [μm]</th>
<th>$P$ [μm]</th>
<th>$f_s$</th>
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<tbody>
<tr>
<td>I</td>
<td>12.5</td>
<td>62.5</td>
<td>0.580</td>
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<td></td>
<td>25</td>
<td>75</td>
<td>0.403</td>
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<td></td>
<td>37.5</td>
<td>87.5</td>
<td>0.296</td>
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<td></td>
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</tr>
<tr>
<td></td>
<td>100</td>
<td>150</td>
<td>0.403</td>
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Fig. 12. Experimentally measured $K/R_{eff}$ of the micropost wicks as a function of solid fraction.

In case I, the solid fraction increases as the distance between posts ($D_{cc}$) is decreased. As a result, both the permeability and the effective pore radius decrease as the solid fraction ($f_s$) increases. The ratio $K/R_{eff}$ decreases approximately linearly with increasing solid fraction as the flow passage area blocked by the posts increases. The effective pore radius ($R_{eff}$), in contrast, is not strongly affected by the solid fraction ($f_s$) as the distance between neighboring posts ($D_{cc}$) remains fixed. As a result, the capillary performance parameter $K/R_{eff}$ decreases once again with $f_s$.

Fig. 14 compares the capillary pressure obtained from the experiments and from the analysis of the equilibrium meniscus shapes obtained through surface energy minimization. The two results agree reasonably well with each other. Difference between the two results is due in part to the fact that the experimentally obtained values are affected strongly by nonuniformity in the post geometry and in part to the complex microscale wetting phenomena not captured by (2). Cross-sectional SEM images of the present wicks show that microposts tend to be slightly tapered, having larger diameters near the substrate. The predicted capillary pressure, in contrast, is determined primarily by the geometric parameters near the free meniscus surface.

V. SUMMARY AND CONCLUSION

We have demonstrated superhydrophilic Cu micropost arrays fabricated using electrochemical deposition and controlled chemical oxidation techniques. To prevent the formation of defects and nonuniformity, PR-based plating molds are oxygen plasma treated, and the electroplating current density is carefully controlled during deposition. Uniform and nearly defect-free arrays of micro Cu posts of solid fractions as high as 58% and aspect ratios as high as four are achieved over large areas ($\sim 3$ cm$^2$). Following the electrochemical deposition, controlled chemical oxidation is used to nanostructure the micro Cu posts. The unique morphology and the high surface energy of the CuO nanostructures turn the poorly water-wetting copper post wicks into superhydrophilic wicks.

The capillary performance of the micropost arrays is characterized using capillary rate of rise experiments. The shape of the liquid menisci is predicted using the surface energy minimization algorithm and is found to have appreciable effects
on the permeability of the present micropost wicks. Despite substantial increase in the surface area, nanostructuring has relatively minor effects on the permeability and significantly enhances the capillary performance parameter.

Our work provides a useful starting point to achieve optimal balance between the capillary performance and the effective wick thermal conductivity. The fabrication process suggested in this study can also be applied to other devices requiring dense arrays of metallic microposts.

REFERENCES


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