

Growth and aggregation of Cu nanocrystals on ionic liquid surfaces

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Growth and aggregation of Cu nanocrystals on ionic liquid surfaces*

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We report a catalyst-free growth of Cu nanocrystals on ionic liquid surfaces by thermal evaporation method at room temperature. After deposition of Cu on ionic liquid surfaces, ramified Cu aggregates form. It is found that the aggregates are composed of both granules and nanocrystals with triangular or hexagonal appearances. The sizes of the nanocrystals are in the range of tens to hundreds of nanometers and increase with the nominal deposition thickness. The growth mechanism of the Cu aggregates and nanocrystals is presented.

Keywords: ramified aggregates, Cu nanocrystals, liquid substrates, critical state

PACS: 68.55.–a, 81.10.Aj, 61.46.Df, 68.37.–d **DOI:** 10.1088/1674-1056/ab8896

1. Introduction

During the last two decades, researches on various nanostructures have received great interests due to their wide range of applications, such as quantum resistors, catalysts, optics, and electronics.[1–6] As a result, more and more kinds of nanostructures, including nanowires, nanotubes, nanoplates, etc., have been successfully synthesized.[7–10] In theoretical research, a number of studies about the nucleation and aggregation of atoms and molecules on liquid surfaces have been performed so far and several interesting results have been obtained.[4,11,12]

In order to fabricate various nanostructures, several growth methods can be adopted, such as sputter deposition,[13] thermal evaporation,[4] chemical etching,[9,14,15] laser ablation method,[16,17] inverse micelle method,[18] cold-wall physical vapor deposition (CWPVD),[19] etc. In most cases, the addition of stabilizing agents or catalysis is generally needed for preparing the nanostructures in different solutions.[15,20,21] As a result, the prepared samples may adhere to byproducts, stabilizing agents, and other chemical impurities, which are unfavorable for subsequent measurements and applications. Generally, vapor-deposition method is regarded as a clean way for preparing of nanostructures.[4,22]

To date, liquid surfaces can be considered as quasi-free sustained substrates, which possess isotropic characteristics and quasi-atomical surface smoothness. In this case, the deposition atoms may exhibit large coefficient and diffuse randomly on the substrates.[4,23] Recently, many studies focus on the formation mechanism of different nanostructures on liquid surfaces, such as atomic aggregates,[24,25] metallic nanoparticles,[13,22] graphene,[26] etc.

In 1998, Ye *et al.* generated ramified silver aggregates on silicone oil surfaces and presented the two-stage growth model.[4] Subsequently, gold (Au)[27] and copper (Cu)[28] ramified atomic aggregates were fabricated on liquid surfaces successfully. It was found that, in most cases, the ramified aggregates formed on the liquid surfaces are composed of granules and therefore exhibit polycrystalline structure.[12,24] What excites people was that, in 2016, one-dimensional zinc (Zn) nanocrystals on silicone oil surfaces were firstly generated,[29] which demonstrates the possibility that various metallic nanocrystals can be prepared on liquid surfaces. It was proposed that the growth of the one-dimensional Zn nanocrystals is contributed to its hexagonal close-packed (hcp) structure, which possesses the preferential growth directions.[29–31] This proposal was supported partially by Bao and co-workers' experimental result.[32] However, the experimental results about the growth of metallic nanocrystals with face centered cubic (fcc) or body centered cubic (bcc) crystalline phase structures on liquid substrates have been rarely reported until now.

It has been realized that, in principle, generating metallic nanocrystals on the liquid surfaces is possible if the experiment conditions are appropriate. Since the liquid surfaces may be considered as a quasi-free sustained substrate, nanocrystals may grow on the liquid surfaces without the obstacle of lattice mismatch,[29,30] which is quite helpful for the growth of nanocrystals. Unfortunately, the quantitative growth mechanism of metallic nanocrystals on the liquid surfaces has been rarely reported so far.

In this paper, we report the catalyst-free growth and aggregation of Cu nanocrystals, which possess the fcc structure, on ionic liquid surfaces by thermal evaporation at room temperature. The experiment shows that the ramified Cu aggregates grown on the ionic liquid surfaces are composed of both granules and nanocrystals with triangular or hexagonal appear-

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ances. Moreover, the mean size of the triangular Cu nanocrystals increases with the nominal deposition thickness and remains nearly unchanged with the deposition rate. It is found that the average size of the hexagonal Cu nanocrystals is much larger than that of the triangular Cu nanocrystals. Based on the experimental results, the growth and aggregation mechanism of the Cu nanocrystals is proposed.

2. Experimental method

The samples were fabricated by thermal evaporation method in a vacuum of about 3×10^{-4} Pa at room temperature ($T = 25 \pm 2$ ℃). As a liquid substrate, an ionic liquid (1-butyl-3-methylimidazolium tetrafluoroborate, purity, 99%+, Adamas) with low vapor pressure close to zero at room temperature was painted onto a frosted glass surface. The resulting liquid substrate with an area about 10 mm×10 mm exhibited a uniform thickness of ∼ 0.5 mm and was fixed 130 mm below the filament in the vacuum chamber. The Cu wires (purity, 99.99%+) were uniformly wrapped around a portion of the filament. The quartz-crystal balance (ULVAC CRTM) was located near the liquid substrate, which monitored the deposition rate f and the nominal deposition thickness d. In our experiment, the deposition rate was in the range of $f = 0.001$–0.200 nm/s and the nominal deposition thickness was in the range of $d = 0.1$–6.0 nm.

After deposition, the sample was kept in the vacuum chamber for a time interval of $\Delta t = 30$ min, providing the aggregation time for the ramified Cu aggregates on the liquid surfaces[4,12,24] and then removed from the vacuum chamber. Then the Cu aggregates were transferred from the ionic liquid surface to a polished single crystalline silicon wafer: a silicon wafer cleaned by ultrasonic was used to cover on the sample surface for 10 minutes, so that the Cu aggregates could adhere firmly to the silicon wafer surface, then it was washed carefully by acetone and ethanol step by step.[12,29] The morphology of the ramified Cu aggregates was measured by a scanning electron microscopy (SEM, Zeiss Supra 55) equipped with an energy-dispersive x-ray spectrometer (EDS). A transmission electron microscopy (TEM, FEI Tecnai G^2 F20 S-TWIN) was used to measure the microstructure of the Cu nanocrystals.

3. Results and discussion

Figure 1 shows the morphologies of the ramified Cu aggregates, granules, and nanocrystals grown on the ionic liquid substrates. The whole morphology of the Cu aggregates can be seen in Fig. 1(a), which is similar to those of other metallic aggregates on liquid substrates observed before.[4] If we take a closer look at the Cu aggregates, however, it is quite unexpected that the aggregates are composed of both Cu granules and nanocrystals with triangular or hexagonal appearances.[28] It is noted that the average size of the Cu nanocrystals is generally larger than that of the Cu granules, as shown in Fig. 1.

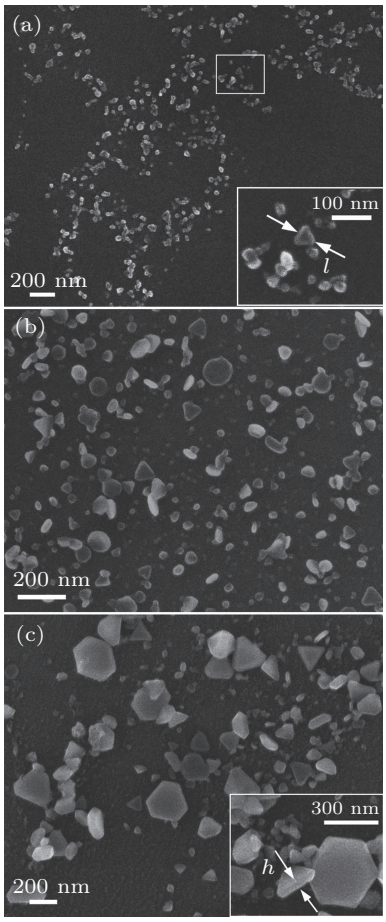


Fig. 1. SEM images of the ramified Cu aggregates and nanocrystals on ionic liquid surfaces, $f = 0.005$ nm/s. (a) The morphology of the ramified Cu aggregate, and the inset is the high-resolution SEM image of the rectangle region, $d = 0.3$ nm. (b) The morphologies of the Cu granules and nanocrystals, $d = 3.0$ nm. (c) The morphologies of the Cu granules and nanocrystals, and the inset shows the side view of a nanocrystal with triangular appearance, $d = 4.0$ nm.

From Fig. 1, one can see that, as the nominal deposition thickness d increases, more nanocrystals with regular morphologies appear on the ionic liquid substrates. Comparing Figs. 1(a)–1(c), we find that the size of the triangular nanocrystals, namely l shown in Fig. 1(a), increases with the thickness d. On the other hand, from the side view of the nanocrystal pointed by the white arrow in the inset of Fig. 1(c), we obtain that the nanocrystals exhibit a sheet morphology. Our measurement shows that the average thickness h of the nanocrystals grown on the ionic liquid substrates is around the order of 30 nm.

In order to understand the microstructure of the Cu nanocrystals, high-resolution TEM (HRTEM) measurements are carried out and the result is shown in Fig. 2(a). As we all know, Cu crystals exhibit the face centered cubic (fcc) crystalline phase structure. The TEM image in Fig. 2(a) shows that the lattice spacing of the atomic plane is 0.21 m, corresponding to the [111] direction of the Cu crystals.[33] Besides, the EDS

measurement was also performed for a triangular nanocrystal shown in Fig. 2(b), confirming that the nanocrystals are composed of Cu atoms. The signals of Si and C in the spectrum are from the single crystalline silicon wafer and a small amount of liquid remains, respectively.

The statistical measurements of the Cu nanocrystal size l are performed in the experiment, as shown in Fig. 3. At a fixed deposition rate f, the size distribution of the triangular Cu nanocrystals changes obviously with the nominal thickness d. More specifically, all the four size distributions are in conformation with the lognormal distribution and, as the thickness d changes from $d = 0.5$ nm to $d = 4.0$ nm, the most probable size of the distributions increases from 40.3 ± 0.5 nm to 72.2 ± 1.5 nm. Another noteworthy phenomenon shown in Fig. 3 is that the distribution range also increases with d.

In order to investigate the growth mechanism of the Cu nanocrystals on the ionic liquid surfaces, we have systematically measured the mean size l of the triangular Cu nanocrystals with d and f, and the results are shown in Fig. 4. Within the error bar of our measurement, as the deposition rate f is fixed, the mean size l increases with the thickness d, as shown in Fig. 4(a), which is consistent with the result in Fig. 3. When $d = 3.0$ nm, the average size of the triangular Cu nanocrystals is about $l \approx 70$ nm and it remains nearly unchanged with f, as shown in Fig. 4(b).

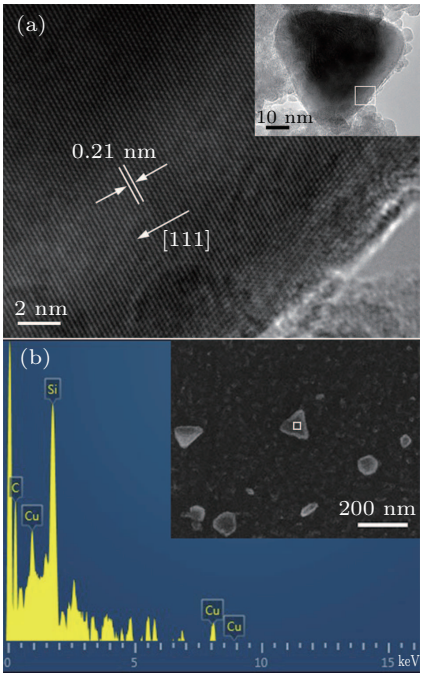


Fig. 2. TEM images and EDS spectrum of the triangular nanocrystals, $f = 0.005$ nm/s. (a) The high-resolution TEM image of the area marked in the inset, $d = 3.0$ nm. (b) EDS spectrum of the square area of the triangular Cu nanocrystal shown in the inset, $d = 4.0$ nm. The spectrum is presented with log scale on the y-axis.

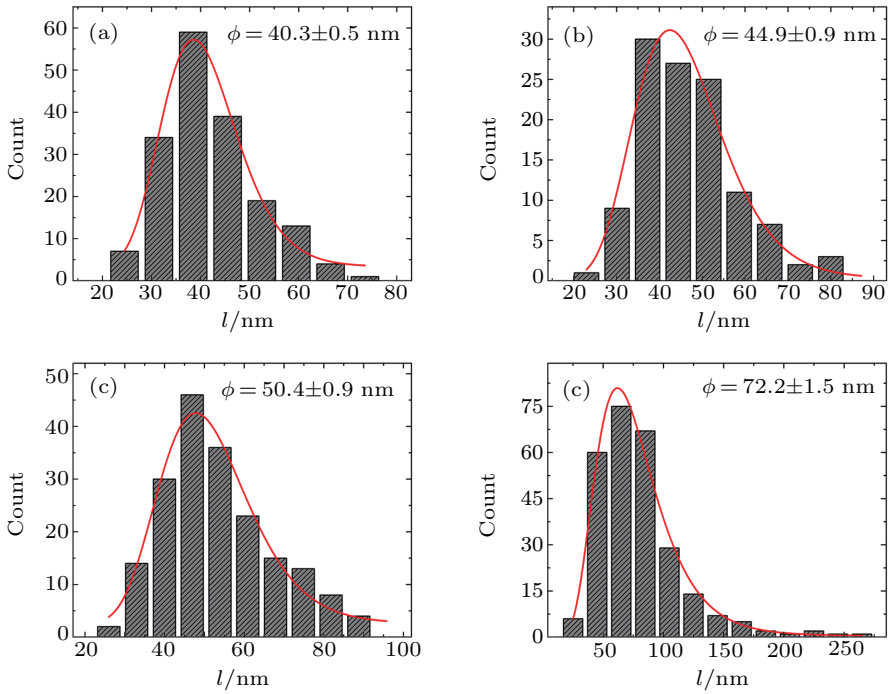


Fig. 3. The statistical distribution histograms of the size l, $f = 0.005$ nm/s. (a) $d = 0.5$ nm. (b) $d = 1.0$ nm. (c) $d = 3.0$ nm. (d) $d = 4.0$ nm. The solid curves are the lognormal fittings with the experimental data.

An important fact in our experiment is that, for higher deposition rate, e.g., $f = 0.200$ nm/s, various Cu nanocrystals can still be observed on the ionic liquid surfaces. This phenomenon is quite unexpected since, in the solution-phase method, low reaction rate is the key factor to synthesize plate-like nanocrystals.[9,34]

Based on the experimental results above, we propose that the formation mechanism of the ramified Cu aggregates can be described basically by the two-stage growth model.[4] In the first stage, the deposition Cu atoms diffuse on the liquid

surface randomly, then they nucleate to form Cu atomic clusters. The growth and crystallization of the clusters results in the formation of both the granules and nanocrystals with regular appearances simultaneously. In the subsequent stage after deposition, the granules and nanocrystals continue to diffuse on the liquid surface by Brownian motion and finally ramified aggregates form, as show in Fig. 1(a).

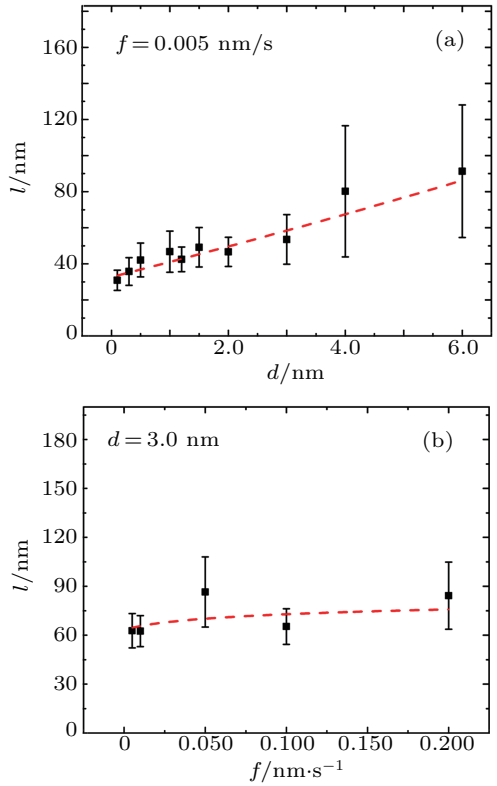


Fig. 4. The evolution of the average size l of the triangular Cu nanocrystals with the nominal deposition thickness d and deposition rate f. (a) The dependence between l and d. (b) The dependence between l and f. The dashed lines in (a) and (b) are the guides to the eye. Each data point represents an average value of over 13 nanocrystals and the error bars represent the standard errors of the mean values.

The phenomenon that both the Cu granules and nanocrystals may form on the liquid surfaces simultaneously is quite interesting, which has never been observed before.[4,12,28] The growth mechanism may be proposed as follows.

After the Cu atomic clusters form on the ionic liquid substrate, they continue to diffuse and aggregate with other Cu atoms and clusters randomly. In this case, the clusters with different sizes and morphologies grow and their average size gradually increases. If the size of a cluster reaches a critical size value ϕ_c, then the structural phase transition and crystallization process take place.[35] Since the ionic liquid substrate can be considered as an isotropic and free-standing liquid surface, the morphologies of the granules and nanocrystals are determined by the free energy minimization principle of the clusters.[24,36,37] In this case, both the compact granules and nanocrystals with regular shapes may form simultaneously on the liquid substrate.

Let us assume that the granules on the liquid surfaces exhibit a hemisphere structure with radius r,[24] then the total free energy of a granule can be written as

$$E_g = 2\pi r^2\sigma_1 + \pi r^2\sigma_2 + \frac{2}{3}\pi r^3\sigma_3, \tag{1}$$

and the total free energy of a triangular nanocrystal reads

$$E_t = \left(\frac{l^2}{\sqrt{3}} + 2\sqrt{3}lh\right)\sigma_1 + \frac{l^2}{\sqrt{3}}\sigma_2 + \frac{l^2}{\sqrt{3}}h\sigma_3, \tag{2}$$

where σ_1, σ_2, and σ_3 correspond to the surface energy between the Cu aggregates and vacuum (granules or nanocrystals), interface energy between the Cu aggregates and liquid substrate, and the bulk energy, respectively, as shown in Fig. 1(c).

Since the compact granules and nanocrystals with regular shapes may form simultaneously on the liquid substrate, we propose that the structural phase transition and crystallization process occur when it is very close to the critical size value ϕ_c. In this case, the volume of the granule is equal to that of the triangular nanocrystal, i.e., $\frac{2}{3}\pi r^3\sigma_3 = \frac{l^2}{\sqrt{3}}h\sigma_3$, and the total free energies of them are the same, i.e., $E_g = E_t$. Therefore, we have

$$2\pi r^2\sigma_1 + \pi r^2\sigma_2 = \left(\frac{l^2}{\sqrt{3}} + 2\sqrt{3}lh\right)\sigma_1 + \frac{l^2}{\sqrt{3}}\sigma_2. \tag{3}$$

In order to understand the nature of Eq. (3) approximately, let us suppose that, near the critical size value ϕ_c, the r, h, and l are on the same order of magnitude. Therefore, under the first order approximation, we may let $r \approx h \approx l$. In this case, equation (3) gives $\sigma_1/\sigma_2 \approx -1.1$, indicating that σ_1 and σ_2 are on the same order of magnitude and their signs are opposite, which is a reasonable result compared with those of other similar systems.[38] Therefore, we propose that equation (3) gives an approximate criterion for the experimental result shown in Fig. 1.

Another interesting phenomenon is that the formation of the hexagonal Cu nanocrystals strongly depends on the thickness d. For the samples with $d = 0.3$–1.2 nm, hexagonal Cu nanocrystals cannot be clearly observed. After that, as d increases, hexagonal Cu nanocrystals gradually appear. However, the triangular Cu nanocrystals appear in all the samples if $d \geqslant 0.3$ nm, as shown in Fig. 1(a). Furthermore, it is found in our experiment that the mean size of the hexagonal Cu nanocrystals (namely L, the distance between two opposite parallel edges of the hexagonal Cu nanocrystals) is generally larger than the average size l of the triangular Cu nanocrystals. For the samples prepared with $f = 0.005$ nm/s and $d = 4.0$ nm, for instance, the average sizes of L and l equal 202.8 nm and 80.2 nm, respectively. Therefore, we propose that the hexagonal Cu nanocrystals may grow from the triangular Cu nanocrystals during deposition by the aggregation with the Cu atoms and other atomic clusters.[39]

In 2006, Chen *et al.* prepared ramified Cu aggregates on silicone oil surfaces with $f = 0.080$ nm/s and $d = 1.0$ nm,[28]

the morphology of which is quite similar to that observed in our experiment. Unfortunately, nanocrystals with regular shapes were not observed in their experiments. The result is obvious since the conditions of the two experiments are very different. On the other hand, the insufficient resolution of their optical microscope images is probably another reason for this regret.

Finally, it should be mentioned that, except for the granules and the nanocrystals with triangular or hexagonal appearances, one-dimensional nanocrystals, such as nanowires, nanorods, nanobands, etc., have not been observed in all the Cu samples prepared with different experimental conditions. This result is in good agreement with the previous prediction that the nanocrystals with the face centered cubic (fcc) and body centered cubic (bcc) crystalline phase structures do not exhibit preferential growth characteristics on the isotropic liquid surfaces.[30]

4. Conclusions

In summary, we have successfully developed an energy-saving, environmentally friendly, and inexpensive method to prepare ramified Cu aggregates and nanocrystals on ionic liquid surfaces. The Cu aggregates are composed of both granules and nanocrystals with triangular or hexagonal appearances. The average size of the nanocrystals is about 10^1 to 10^2 orders of magnitude and it increases with the nominal deposition thickness.

Basically, the formation process of the ramified Cu aggregates still follows the traditional two-stage model.[4] It is proposed that, after the Cu atomic clusters form on the ionic liquid substrate, they continue to diffuse and aggregate with other Cu atoms and clusters randomly. If the size of a cluster reaches the critical size value, then the structural phase transition and crystallization process occur.[24] Therefore, both the granules and nanocrystals may form simultaneously and the morphologies of them are determined by the energy minimization principle of the clusters.

The experimental phenomena shown above indicate that, as long as the experimental conditions are suitable, more nanocrystals may be prepared on some specific liquid substrates. Since the liquid substrate can be considered as an isotropic and free-standing surface, the preferential growth characteristics of various crystals may stand out obviously. Therefore, it is expected that more kinds of crystals with larger size and various morphologies may be fabricated massively on different liquid substrates in the near future.

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