Hypergravity-assisted chemical liquid deposition of nano-granular film on the inner surface of a quartz tube

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1. Introduction

To investigate the flow-pattern dependent heat transfer and pressure drop of the two-phase flow, one needs direct observations of the flow patterns of nucleation and boiling in real time [1–3]. Transparent pressure-resistant quartz tubes are generally used as the windows for visualization experiment.
intentionally with heating elements and temperature sensors mount on the outer wall of the tubes [4].
However, the thermal resistance between the inner surface and outer wall of the quartz tube blocks
the accurate measurement of the temperature on the inner surface (the interface between the fluid and
the tube) and results in a non-negligible error of the heat transfer coefficient, because of the heat leak
of the heat source outside the tube. Heater on the inner surface of the tube can heat the fluid and
measure the temperature of the heating wall more directly. This helps to reduce the heat leak and
improve the accuracy of the local measurement of the temperature, and thus provide reliable
parameters for the analysis. Therefore, it is necessary to develop a visual experimental tube with an
effective heating surface that contacts with the fluid and measures the surface's temperature as well.

At present, the technologies of coating an inner surface of the tube generally adopt physical vapour
deposition (PVD) [5–7], chemical vapour deposition (CVD) [8–11], electroplating [12,13], electrical
explosion [14,15] and dip-coating [16–18]. They are summarized in table 1 for the comparisons of
processes and sizes of the films. However, conventional coating methods have difficulties in
implementing and fabricating uniform films inside a small tube, especially those with large aspect
ratios. Methods of vapour deposition [19] face the challenge of concentration gradient of vapour;
while electroplating [20] faces the challenges of the uniformity requirements of electric field and that
of concentration of the solution. For an instant process of electrical explosion, there is not enough
time to develop a uniform film structure. Dip-coating, one of the chemical liquid deposition, on the other
hand, faces the challenges of ultra-thin liquid film development on a curved surface and in gravity,
and the film surface management especially during the evaporation of the solvent. Spin-coating and
ink-jet printing methods were applied to prepare conductive silver films on the glass surface [21].
However, the prepared silver films could not be thin enough to favour light transmission. Rotating
the tube around its axis is helpful to improve the liquid distribution on the inner surface of the tube.
When the rotation speed is high enough to produce centrifugal force much higher than the gravity
(i.e. hypergravity to the rotating tube) on the inner surface of the tube, more advantages should
appear for forming a uniform liquid film: (i) spreading out the liquid by overcoming the liquid
surface tension; (ii) promoting the escape of the evaporated vapour (possibly bubbles) from the film,
and (iii) accelerating the deposition of the once-produced solid particles onto the inner surface of the
tube in the heating process. Therefore, one could expect to obtain thin films with increased particle
density and improved particle adhesion on the inner surface of the tube.

In this paper, we developed a method of hypergravity-assisted chemical liquid deposition (HACLD)
to prepare the required transparent heating tubes and characterized the structures and properties of the
thus prepared films.

2. Experimental

Organometallic Ag precursor solution was the key precursor to prepare the Ag films. Five organometallic
Ag precursor solutions were synthesized by using a modified complex reaction process [22] (see table 2

| methods          | year, author and reference | tube size and material | film material and thickness |
|------------------|----------------------------|------------------------|-----------------------------|
| PVD              | 1992, J. A. Sheward [5]    | Ø120 mm, steel         | Cr-Nb, 7 – 17 μm            |
|                  | 2016, D. Kottner [7]       | Ø200 – 300 mm, steel   | Ti, 1.3 – 3.9 μm            |
| CVD              | 1986, H. Itoh [10]         | Ø10 mm, steel          | TiN, 12 μm                  |
|                  | 2013, H. Kousaka [9]       | Ø4.4 mm, steel         | DLC, 529 nm                 |
|                  | 2014, R. Hatake [11]       | Ø25 mm, steel          | Ag-DLC, 150 nm              |
|                  | 2017, Y. Xu [8]            | Ø0.9 mm, glass and lucalox | DLC, –                     |
| Electroplating   | 2014, L. D. Sun [13]       | Ø9 mm, Ti              | TiO₂, 5–15 μm               |
|                  | 2017, C. J. Xiang [12]     | Ø10 mm, Ti             | TiO₂, 0.1–1 μm              |
| Electrical explosion | 2000, O. Demokan [15]     | Ø13 mm, Ta             | Al, 7.1–8.1 μm              |
| Dip-coating      | 2007, W. C. Gu [17]        | Ø50 mm, steel          | ceramic, 300 μm             |
|                  | 2018, S. Ayata [18]        | Ø16 mm, Al-Ga-Mg       | ZrO₂-DLC, 182 nm            |
for the nominal Ag mass fraction and labelling). In the process of the Ag precursor solution preparation, Ag citrate, 1,2-diaminopropane and 2-methoxyethanol were used as the precursor compound, organic complexing agent and protective solvent, respectively. Taking the silver precursor solution SC as an example, 0.0078 M Ag citrate (C₁₈H₁₄Ag₂O₇, 4.0 g) and 0.0234 M 1,2-diaminopropane (C₆H₁₀N₂, 1.7 g) were dissolved in 0.2933 M 2-methoxyethanol (C₃H₈O₂, 22.3 g), stirred at 5°C for 10 min to ensure complete complexing, and then stirred at room temperature for 1 h to ensure a full chemical reaction. The ratio of the Ag to amine in the precursor solution was 1:3. At last, the transparent organometallic Ag precursor solution was obtained (figure 2a).

A set-up was designed and built for the HACLD inside a tube (figure 1a). The quartz tube, with inner diameter of 8 mm and length of 200 mm, can be rotated by a DC servo motor at an adjustable speed (from 120 to 3000 r.p.m.) to provide different levels of gravity (from 1.6 g to 40 g, respectively) on the inner surface of the tube, and can be heated with a transparent heating tube (D = 23 mm, L = 110 mm) outside the rotating tube to allow in-site observation of the heat-treatment. The rotational speed of the quartz tube can be adjusted according to the colour of the film in the quartz tube which is observed through the transparent heating tube in real time. Hanging in the air, one end of a K-type thermocouple was installed between the quartz tube and the heating tube to indirectly measure the temperature of the rotating tube during the heat treatment. The temperature it took must be higher than that of the inner surface of the quartz tube (figure 1b). Calibration shows 7°C higher at the temperature of 290°C for static tube. The temperature distribution inside the heating tube with the static quartz tube shows that only the central part of 5 cm is uniform within 2% difference (figure 1c). Due to the limitation of the effective and uniform heating length of about 50 mm for the hypergravity set-up, all the results and discussions of the Ag films in this paper will be confined to this area. Figure 2 schematically illustrates the key procedure for the preparation of the uniform and transparent Ag films in the tube by HACLD.

The fabrication process consists of four phases (figure 2b–e). First, fix the quartz tube on the set-up, and inject the needed amount of Ag precursor solution into the tube; and then rotate the tube at the speed of 120 r.p.m. (ω₁) for 5 min to ensure that the Ag precursor solution has spread out uniformly (figure 2b). Second, increase the rotation speed to 3000 r.p.m. (ω₂) to create a hypergravity of 40 g to make the liquid film even thinner (figure 2c). Third, preheat the quartz tube at the nominal temperature of 150°C (T₁, taken by the K-type thermocouples) by using step-type AC voltage (20–40 V, shown in figure 3), which promotes the volatilization of the solvent and the decomposition of the solute in the liquid film (figure 2d). Assisted by the hypergravity, the Ag nanoparticles (NPs) once decomposed from the solution are accelerated to sink onto the inner surface of the quartz tube. Fourth, heat the quartz tube at the rotation speed to 400 r.p.m. (ω₂) at the nominal temperature of 290°C or 340°C (T₂) with step-type AC voltage (50–60 V, shown in figure 3) for 20 min (figure 2e). Ten films were prepared from the five Ag precursor solutions (table 2).

Differential scanning calorimetry–thermal gravimetric analyser (DSC-TGA) was used to study the thermal decomposition of the organometallic Ag precursor solutions, so as to determine the heating temperature on the film formation and determine the mass fraction of Ag in the solutions. The simultaneous thermal analyser (STA) 449F3 (Netzsch, Germany), with the mass measurement accuracy of ±1.5%, was employed for the DSC-TGA, with the heating rate of 10 K min⁻¹ in flowing air. X-ray diffractometer D8 ADVANCE equipped with Cu Ka radiation (Bruker, Germany), was used to perform X-ray diffraction (XRD), to characterize the crystal structures and the composition of the prepared films. For better comparison among different films, the fragments of cracked quartz tubes were pasted onto the test bench by using the Plasticine in the way that the quartz tube axis is parallel to the test bench and the bottom surface (on which X-ray sweeps) of the tube is at the same level of the surface of the bench with maximal error of ±0.2 mm. In this way, the error caused by the curvature of the inner surface of the quartz tube could be minimized to 0.31°. Scanning electron microscopy (SEM) images were performed on the field emission SEM Gemini500 (Zeiss/Bruker, Germany), to study the surface morphology of the prepared films. To characterize the relationship between the morphology and the optical and electrical

| solution | SA | SB | SC | SD | SF |
|----------|----|----|----|----|----|
| nominal Ag mass fraction in solution (wt%) | 1.0 | 2.0 | 3.0 | 4.0 | 5.0 |
| heating at nominal 290°C, 3000 r.p.m. | FA | FB | FC | FD | FE |
| heating at nominal 340°C, 3000 r.p.m. | FA' | FB' | FC' | FD' | FE' |
properties of the films, a UV-3600 (Shimadzu, Japan) was used to measure the optical transmittances for
nano-granular films on the pieces of broken tubes at room temperature; and a KDB-1 (KunDe, China)
was used to measure the sheet resistance by a four-point probing system. At last, after a semi-transparent
Ag film heater was prepared inside the quartz tube, a thermal camera A600 (FLIR Systems AB, Sweden)
was used to take the infrared images of the surface of the tube heated by its conductive film. The voltage
was applied by a DC power supply through copper wires at both ends of the film heater.

3. Results and discussion

Hypergravity was found essential to the preparation of thin films on the inner surface of the quartz tubes.
In the attempt to obtain a suitable rotation speed for the film formation process, we heated the rotating
tube with spread Ag precursor solution film at the nominal temperature of 290°C for 20 min and inspected the colour of the film simply with the naked eye. Take the solution SC as an example (see table 2 for the labelling), at low rotation speed of 120 r.p.m. during heating (ω2 in figure 3), the as-prepared sample looks like randomly distributed drops rather than film (figure 4a); while at 3000 r.p.m., the film was spread out (figure 4b), and formed uniformly and continuously as shown by the SEM image at the cross-section of the Ag film (FC in figure 7c). The digital photos in figure 4c show the films from SB to SE heated at 290°C and 340°C for 20 min, respectively. The black line drawn on the scotch tape behind the quartz tubes is for better comparison of the optical transmittance among the different quartz tubes. With the increase of Ag content in the precursor solution, the film colour changes from light blue to dark black, which may indicate the increases of the film thickness. The exception yellow colour of the film FB will be discussed later.

Correspondingly, with the increase of the Ag content in the precursor solution, the sheet resistance of the Ag film decreases dramatically (table 3). The decrease of the resistance could not be simply attributed to the increase of the thickness. The thickness of the film is 73 nm with sheet resistance of 2.1 Ω/□, while the thickness is 42 nm with 625.6 Ω/□.

By applying a DC voltage of 6.0 V to the conductive film FC on the quartz tube, Ø8 mm × 50 mm in size, the surface temperature of the tube rises over 100°C, as shown by the infrared camera (FLIR, figure 5).

Heating at suitable temperature can assist the evaporation of the solvent and promote the complete decomposition of the organic matter in the film. The TGA curves show that the five prepared organometallic Ag precursor solutions can completely evaporate at the temperature of 290°C (figure 6a). For comparison, the pure solvent completely evaporates at 111°C (as shown in the inset TGA curve of figure 6a). Take solution SC as an example, there are three stages of the reaction found in the DSC-TGA curves (figure 6b): (i) 70–120°C with a huge endothermic reaction accompanied with a large corresponding weight loss of about 93%; (ii) 140–160°C with an endothermic reaction appears and a small weight loss of about 4%, and (iii) 300–340°C with a gentle exothermic peak and almost negligible weight loss.

The first stage is mainly related to the evaporation of the solvent, which is similar to Nie et al.’s result [23]. The second stage must be crucial to the formation of the Ag films. It may be related to the silver precipitation from the solution to form Ag NPs. After subtracting the error (0.16%) caused by organic residues of the solvent evaporation (see the inset TGA curve of figure 6a), the final mass residue of Ag is 3.41% in this stage, more than the theoretical content (2.43%), which is probably due to the carbon retained. The formation reaction could be expressed by the following formula:

$$4[Ag(C_3H_{10}N_2)]_3C_6H_5O_7 + 15O_2 \xrightarrow{\Delta} 12Ag \uparrow + 12C_3H_{10}N_2 + 24CO_2 + 10H_2O.$$  

(3.1)

The temperature of the endothermic reaction (150°C) is much lower than the decomposition temperature of silver citrate powder (190°C), but agrees with the thermal decomposition behaviour of the silver citrate in
solution shown by Nie et al. [23]. The gentle exothermic peak in the third stage (300–340°C) could be attributed to the fusion of the Ag NPs into much bigger drops. This is supported by the corresponding SEM images (shown in figure 7c,f), which show that the Ag film turns from the thin and dense granular structure at nominal 290°C, into a thick and drop-like structure at nominal 340°C.

SEM was performed to characterize the surface morphology at the stage of the growth of Ag films. The morphologies of films (FA to FE, FB' and FC') are shown by the SEM images in figure 7a–g. They are selected from 80 images of the representative samples. Isolated particle clusters are found for film FA, and evenly dispersed on the quartz substrate (figure 7a). For film FB, much more dense and better-connected particles are found with size of about 40–50 nm, comparable to those of film FA (figure 7b). Particles are fully connected with each other in film FC (figure 7c), with mean size that looks even smaller than those of FB. Particles seem to start to fuse into each other for film FD, supported by the merged boundaries and loose interspace in the image (figure 7d), as compared to film FC. The average particle size of film FD is comparable to that of film FC. Film FE shows the film thickness of about 70 nm with the apparent particle size of 40–50 nm (see figure 7e and its inset).

Higher Ag content in the solution leads to the higher density of the Ag particles and larger thickness of the film, but has little effect on the size of the Ag particles (40–70 nm). All the films from FB to FE are of single-layer particles, as shown in the inset SEM cross-sectional image in figure 7c,e. By binarizing the SEM images with the threshold level of 110, the coverage of the silver particles in the film was obtained for each film and listed in table 3. By increasing the heating temperature to nominal 340°C, the particles turn into much bigger isolated drops (figure 7f).

X-ray diffraction (XRD) was performed for the films to characterize their crystal structure. Figure 8a shows the XRD patterns of the Ag films, heated at nominal 290°C for 20 min. No obvious diffraction peak is observed for the film FA, and only a weak diffraction peak appears at the position of the main peak in the film FB. Twenty accumulation scans for both film FA and FB show only a weak diffraction peak of...
(111) plane, but no trace of any of the silver oxide (figure 8b). With the increase of Ag content (FC and FD), the diffraction peaks of Ag appear gradually, and the intensity increases. All of the diffraction peaks of film FE match those of Ag (111), (200), (220) and (311) crystal planes (Ag-PDF # 04-0783). There is no other diffraction peak in the patterns. The small offsets of the diffraction peaks (2θ = +0.25° for film FB, +0.15° for film FC, −0.29° for film FD, and +0.01° for film FE, respectively) in the XRD patterns (figure 8) could be attributed to the curvature of the inner surface of the quartz tube, leading to the X-ray scanned surface being different from that of the test bench surface. They were within the error range of 0.31°. However, the intensity of the diffraction peak is not proportional to the thickness of the film, nor to the Ag content in the precursor solution that forms the film.

One can conclude that all the particles in the films (FC to FE) are in the same crystal structure, and with the same lattice constant, but might not be fully crystallized or there might be mass loss as suggested by the SEM, especially for that with low Ag content. The full width of the diffraction peak at half-maximum (FWHM 0.756) is almost the same for all the three films (figure 8), also suggesting that the particles are comparable in size (58 nm). This is in agreement with the SEM observation (figure 7).

Optical transmittance of a single layer of the Ag film was measured to evaluate the application availability of the tubes. The transmittance decreases as the Ag content in the solution increases (figure 9a), and basically, is a little higher at shorter wavelength. This explains why the tubes with thin film are light blue in colour. The film FA shows the highest transmittance of 74%, and the film FE shows the lowest of 6%. The transmittance and the absorption spectra of film FB' (figure 9b) tell why the tube is yellow in colour (see the photo in the inset). The length of the yellow region is about the same as that of the uniform area of the heating tube of the set-up (figure 1). The absorption spectrum
is in close agreement with the absorption of the localized surface plasmon resonance (LSPR) ($\lambda_{\text{LSPR}} \approx 458$ nm) on the Ag NPs sizing 40 nm in the previous study [24]. This particle size is comparable to that of FB shown in figure 7g.

After analysing the films’ properties, we may understand more on the role the hypergravity plays in assisting the formation of the nano-granular thin film. First, competitive with the surface tension, the hypergravity makes it possible to control the thickness of the liquid film of the Ag precursor solution, which determines the thickness of Ag film. Second, during the heat treatment process the surface tension becomes weaker at the beginning as the temperature increases, the hypergravity compresses

**Figure 7.** Electronic microscopy images of the Ag films. (a–e) SEM images of the Ag films (FA–FE) deposited on the inner surface of the quartz tube, heated at nominal 290°C. The inset SEM images in (c,e) show the cross-section of the Ag films. (f) SEM image of the Ag film FC, heated at nominal 340°C. (g) SEM image of the Ag FB, heated at nominal 340°C.
the liquid film to become even thinner and more uniform, before the solvent evaporation that may
increase the surface tension and viscosity of the liquid. Then, by offering much higher buoyancy, it is
beneficial to the escapes of smaller bubbles of the volatilized solvent vapour and the gas produced by
the decomposition of the organic matter (Ag citrate) in the liquid film, as those found by Zhang
et al. [25]. Finally, it makes the once-formed Ag NPs (with much higher density than that of the liquid) in
liquid film to sink more quickly to the bottom of the liquid film. This is also more conducive for the
sinking particles to collect more Ag from the remaining organic compounds in liquid film, which
assists faster growth of the NPs. In short, it helps to form the thin film and improve its surface
roughness and uniformity, making possible a heating window of quartz tube.

HACLD provides alternative film preparation technology from that of spin coating. First, spin coating
can only apply to a flat surface, and it requires weak surface tension solution, even though the uneven
tangential force that applied along the substrate surface may tears the liquid film apart. HACLD, on the
other hand, offers the centrifugal force \( \omega^2 r \) perpendicular to the substrate surface; it fits the liquid film

Figure 8. X-ray diffraction to study the Ag films with different mass fraction of Ag. (a) XRD patterns for Ag films (FA–FE); (b) magnification of the XRD intensity for Ag films (FA and FB) with 20 accumulative scans.

Figure 9. Transmittance spectra and absorption spectrum of the Ag films. (a) Changes in the UV/vis transmittance spectra of the five types of Ag film heated at nominal 290°C; (b) transmittance and absorption spectra of Ag FB’. Inset: digital photo of the Ag FB’ on the inner surface of the quartz tube, heated at nominal 340°C.
to the circular surface, thins the liquid film by pressure that competes with the surface tension, and prevents the liquid film from tearing apart. Then, heat treatment is only in normal gravity for the spin coating, but in hypergravity plus normal gravity for HACLD where the normal gravity becomes a periodical disturbance ($g \sin(\omega t)$) on the hypergravity. Taking into account the ineluctable vibration (noise) of the set-up, the force added on the film is given by:

$$G_\perp = \omega^2 r + g \sin(\omega t) + \sum_{k=0}^{n} a_k \sin(k\omega t)$$

(3.2)

and

$$G_{||} = \sum_{k=0}^{n} a_k \sin(k\omega t),$$

(3.3)

where $\omega$ is the angular frequency, $r$ is the inner radius of the tube, $g$ is the normal gravity acceleration, $t$ is the time of process and $k$ is an integer. In other words, the vibration disturbance always accompanies the hypergravity. It could help to organize the granular structure of the micro- and even the nanoparticles. More investigation is needed to understand the mechanism of the combined effects of periodical disturbance and hypergravity on the formation process of the nano-granular structure of the growing particles.

4. Conclusion

With the self-designed and home-made set-up, we developed a technique of hypergravity-assisted chemical liquid deposition for thin film preparation on the inner surface of quartz tubes. Semi-transparent and uniform silver films were thus obtained, with suitable electric resistance for the application of heating windows for experiments of fluid mechanics. Micro-structure characterization showed that the films were of nano-granular structure with particle size of 40–70 nm. More researches are expected to understand the combined effects of periodical disturbance and hypergravity, and even the chemical reaction on the motion of vapour bubbles, the growth of the particles, and the organization of the granular structure. Optimization among the film properties is needed for specific applications. It also paves a way to expand the technology of HACLD to the preparations of other films.

Ethics. This work is about inorganic film preparation, with no animal samples involved, so this section is not relevant to this work.

Data accessibility. The datasets supporting this article have been uploaded as part of the electronic supplementary material. Authors’ contributions. Y.K.s. was responsible for the detail design, the manufacture, and the improvement of the hypergravity set-up, performing all the HACLD preparation and the characterization of the films, and the analysis of the data; X.Q.H. and X.G. offered help with the preparation; and Z.L. was responsible for the silver precursor solution; Z.H.H. was responsible for the concept design of the experiment and set-up, supervising the experiment, providing guidance to the analysis of the data, and the writing of the paper. All the authors approved the publication of the paper.

Competing interests. We have no competing interests.

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