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# Quasi-two-dimensional equilibrium solid/liquid interface of colloids at low osmotic pressure



Zhijun Wang<sup>a,b</sup>, Jincheng Wang<sup>a,\*</sup>, Lilin Wang<sup>a</sup>, Junjie Li<sup>a</sup>, Yaohe Zhou<sup>a</sup>

<sup>a</sup> State Key Laboratory of Solidification Processing, Northwestern Polytechnical University, Xi'an 710072, PR China

<sup>b</sup> Center of Advanced Structural Materials, Department of Mechanical and Biomedical Engineering, City University of Hong Kong, Kowloon, Hong Kong, PR China

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## ABSTRACT

Solid/liquid interface is very essential for nucleation and the crystal growth process. In the present paper, we present a new method to prepare the crystal–fluid interface of colloidal hard spheres by evaporation in microchannel, which is a good candidate as a model system for the investigation of solid/liquid interface. In the system, the quasi-equilibrium colloidal solid/liquid interface is obtained at low osmotic pressure controllable by evaporation. The results show a dynamical broadening amorphous region near the solid/liquid interface. The crystal growth process in the dynamical broadening region depends on the competition between the attaching kinetics and the supersaturation related to the volume fraction. The results agree with previous investigations.

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## 1. Introduction

Solid/liquid interface is an important issue in nucleation and the crystal growth process where the solid/liquid interface status plays an important role in determining the critical nucleation nucleus and the interface morphologies [1,2]. Progress on the investigation of solid/liquid interface has greatly promoted the understanding of crystal growth [3–6]. The criterion for the rough/facet interface was firstly proposed by Jackson about 50 years ago [6], where the derivation was based on the single atomic layer interface model. Subsequently, several multi-layer interface models were developed [6]. These theoretical investigations reveal the interface property from atomic statistical view. Unfortunately, over the years the details of the atomic movement across the solid/liquid interface were still difficult for theoretical investigation and thus the related investigation has stagnated for decades. The development of computers provides powerful tools for the investigation of solid/liquid interface. The Monte Carlo and Molecular Dynamics methods raise prosperity for the solid/liquid interface investigation once again [7]. The perspective of solid/liquid interface in simulation presents the relationship between the interface configuration, the interface energy and atomic arrangement across the interface, which gives further understanding on the solid/liquid interface. However, that is not the end of exploration on the

solid/liquid interface. The experimental investigation on the solid/liquid interface is necessary and still insufficient.

So far, it is very difficult to directly observe the equilibrium solid/liquid interface at atomic scale and at diffusion time scale simultaneously in the real materials. In contrast to theoretical and simulational investigations, experimental studies are cumbersome referring to the atomic structural dynamics across the solid/liquid interface. As a good model of the hard sphere system, the colloidal system provides a chance to investigate the solid/liquid interface by considering the dynamic length scale analogy to atomic scale in real materials [8]. The colloidal crystallization system has been well applied into the nucleation research, which reveals the structural transformation and the two-step nucleation phenomena [9–11]. The related investigations extend the classical nucleation theory into a more specific case considering the atomic structure evolution. For the equilibrium solid/liquid interface, many works have been carried out to reveal the intrinsic properties of the colloidal solid/liquid interface [12–16]. The intrinsic properties of the solid/liquid interface in atomic scale have been revealed. It has been observed to be rough with the capillary wave. Accordingly, these experiments provide a useful test for future models of the solid/liquid interface.

In most previous experiments, the gravity sedimentation [12–14] and the thermal gradient [15,16] were used to set up the solid/liquid interface. For the gravity sedimentation method, the mass density difference between the solvent and the colloidal particle determines the driving force of the system. Accordingly, the system needs solvents with different densities to obtain a different interface status. Furthermore, the system must be vertically placed, which takes

\* Corresponding author. Tel.: +86 29 88460650; fax: +86 29 88491484.  
E-mail address: [jchwang@nwpu.edu.cn](mailto:jchwang@nwpu.edu.cn) (J. Wang).

much long time for the system to be in equilibrium. For the thermal gradient method, it is only appropriate for the thermosensitive colloidal systems. Here we introduce a much simpler and more efficient method to set up the equilibrium colloidal solid/liquid interface based on the low osmotic pressure from evaporation. Properties of the solid/liquid interface obtained here agree with those in previous investigations by the gravity sedimentation method. The most remarkable advantage of this method is that it is controllable and applicable to all colloidal systems.

## 2. Experimental details

The method proposed here is based on the variation of osmotic pressure by evaporation. In a microchannel filled with dilute colloidal suspension, the evaporation induces particles accumulation at the meniscus between the gas and the suspension. The evaporation will decrease the osmotic pressure in the colloidal solid adjacent to the meniscus. The variation of osmotic pressure makes the water flow from the dilute liquid to the solid, bringing the particles in the suspension to attach onto the colloidal solid. The driving force in this system can be controlled by adjusting the evaporation process. Compared with the gravity sedimentation method and the thermal gradient method, this method is easier to be implemented and universal for almost all kinds of colloidal particles.

Here, the monodisperse polystyrene spheres (diameter  $1.73\ \mu\text{m}$ , polydispersity  $< 5\%$ , Bangs Laboratories) were dispersed uniformly in deionized water. The colloidal suspension is then injected into a very thin microchannel with  $20\ \mu\text{m}$  thickness. The microchannel illustration is shown in Fig. 1. The gas/colloidal suspension interface exhibits a meniscus, which results in the small pressure in the channel. The rapid evaporation at the two entrances of the channel results in the colloidal crystallization at both ends. The evaporation will decrease the osmotic pressure in

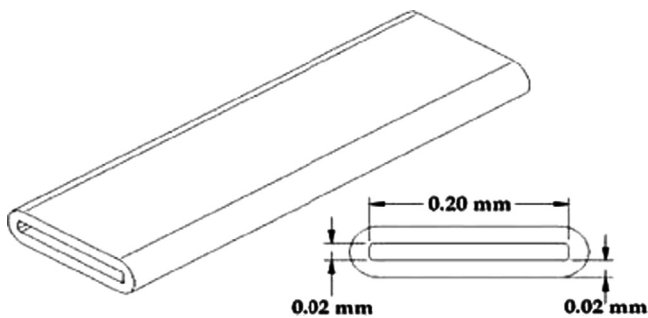


Fig. 1. The microchannel 5002S from VitroCom.

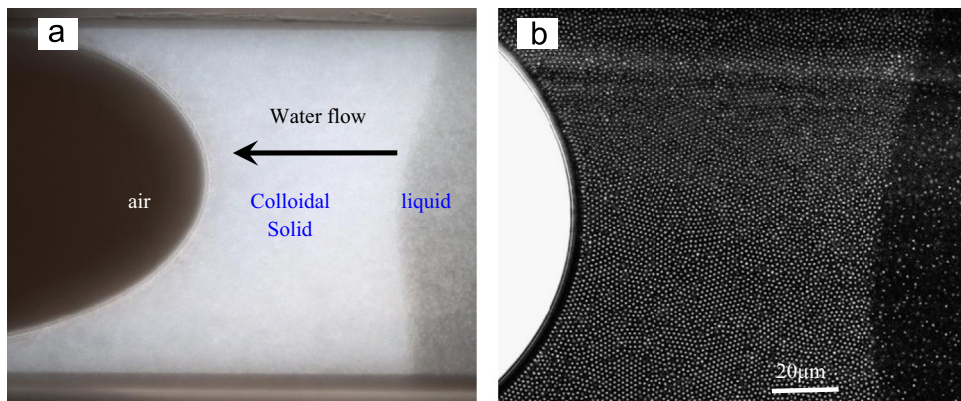


Fig. 2. The solid/liquid interface and the solid/air meniscus, where the meniscus presents the low osmotic pressure in the microtube. (a) The optical image and (b) the laser confocal image of the local area.

crystallization, which introduces water flow in the channel. Due to the water flow from evaporation, the solid/liquid interface propagates into the channel. The images are obtained by laser confocal microscopy. Fig. 2 shows the solid/air meniscus and the solid/liquid interface. After a period of time, the evaporation is prevented by sealing both entrances of the channel. The air is still unsaturated and the system will be equilibrated by water redistribution from the dilute liquid to the solid and then to the air. Twelve hours later, the system will be in a quasi-equilibrium state.

## 3. Results and discussions

Fig. 3 shows the solid/liquid interface morphologies and the particles configuration through the dilute fluid to the crystalline solid. In the crystalline solid phase, there are several large grains with different orientations. The FFT transformation indicates that the particles exhibit obvious hexagonal structure. In the dilute fluid phase, the particles have the Brownian movement. Between the crystalline solid and the dilute fluid phase, there is a dense amorphous region, where the particles vibrate locally. Although ordering appears locally in the amorphous region, the FFT transformation indicates the holistic disorder status. Accordingly, the interface between the crystalline solid and the amorphous is analogized to the quasi-equilibrium solid/liquid interface. Fig. 3 shows that the whole transition region broadens to about 15 particle diameters. This transition region is similar to that in the gravity sedimentation experiments, where the interface was thought to be a dynamic broadening region [12].

To reveal the evolution of transition region, we further present the dynamic movement of the quasi-equilibrium solid/liquid interface in Fig. 4, where the slow water flow drives the particles to approach the interface with a velocity of about  $0.01\ \mu\text{m/s}$ . In Fig. 4, the dashed circles illustrate the onset of the hexagonal structure and the line in the corner marks a grain as reference of the initial crystalline position. The particles first condense in front of the crystalline solid at the interface region and then vibrate to attach on the grains or nucleate into a new grain. Initially, the concentrated region near the dilute fluid is liquid-like, where most of the particles are randomly distributed. At 0 s, several particles at the bottom of the circle present the rudiment of the crystal nucleus with hexagonal structures. In the subsequent evolution stages, several particles become ordered and small nucleus appears in the amorphous region. However, the ordered region in the circle does not propagate before 700 s when the circle is near the dilute fluid. At 1260 s, in the hexagonal structure region

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