



# Exploration of Direct-Ink-Write 3D Printing in Space: Droplet Dynamics and Patterns Formation in Microgravity

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## Abstract

As a simple, fast and effective 3D printing method, direct-ink-writing (DIW) has potential applications in repairing the circuit board in orbit, printing the wearable devices for the astronaut, and producing the solar cells for the energy supply in space. To expand the DIW technology to space, we designed the colloidal material box (CMB) as the prototype printer of DIW and verified its applicability in the Chinese SJ-10 satellite. The colloidal suspensions was adopted as a diluted ink model to investigate two key processes of DIW under microgravity environment: manipulation of the droplet and formation of the patterns. We have showed the dynamics of the droplet, which would determine the size of the features, could be controlled through tuning the wettability of the needles and the solid surface. Compared to the ground, the “coffee ring” effect was weakened for the drying patterns because of strong interfacial effect under weightless conditions. We have found that fast evaporation could assist for fabricating more uniform and ordered structures.

**Keywords** 3D printing · Direct-ink-write · Colloidal self-assembly · Droplet evaporation · Deposition pattern

## Introduction

There are strong application requirements of the micro-nano structures in space, such as printed circuits (Jiang et al. 2018), sensors (Huang et al. 2011; Yang et al. 2020), solar cells (Bag et al. 2016), and tissue engineering (Zhu et al. 2016), which can be used for spacecraft electronics (Werkheiser 2014), astronaut health monitoring (Hill 2019) and energy supply (Leake et al. 2016), respectively. Suppose that these structures and components can be fabricated directly in space, it will save a huge cost of space transportation and bring great convenience of replacement and maintenance in-orbit (Cowley et al. 2019; Fateri et al. 2018, 2019; Sacco and Moon 2019; Wang et al. 2020). However, the traditional “top-down” fabrication methods such as photoetching for chips are difficult to expand

in space for its expensive costs, complicated process and enormous consumption (Xiao 2001).

Compared to traditional manufacturing techniques, the three-dimensional (3D) printing has been proved to be an effective manufacturing technology that employ digitally controlled layer-by-layer deposition schemes to realize controllable composition and produce complex components and architectures (Gebhardt 2011; Guo and Leu 2013; Lipson and Kurman 2013; Wong and Hernandez 2012), therefore, it has been rapidly extended in the field of space manufacturing. Multiple 3D printing technologies on the basis of various materials (polymer, metal, ceramic, composite, etc.) was tested in space microgravity environment (Prater et al. 2017, 2019; Zocca et al. 2019). As a simple, fast and effective 3D printing method, the direct ink writing (DIW) technology can print a multitude of materials and construct arbitrary 3D structures through programmatically depositing fluid droplets on substrate surfaces (Lewis 2006; Lewis and Gratson 2004). Based on extensive utilizations for electronics, optics, and displays under terrestrial condition (Cox et al. 2001; Gao et al. 2012; Hon et al. 2008; Park et al. 2007; Zhang et al. 2012), DIW has tremendous potential application prospects in space if it can accommodate the extreme environments of space especially weightlessness.

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Both the fluid dynamics and the evaporation kinetics of ink droplets play important roles in defining the printing structures deposited by DIW (Lewis and Gratson 2004), therefore, it is necessary to clarify the gravitational effects on these two physical processes under the condition of weightlessness. Firstly, the inks should be injected or squeezed through a needle successfully. In microgravity environment, gravitational effects are negligibly small and surface tension dominates liquid behavior, thus the liquid tends to climb along the wetting surface or float away without the downward force of gravity, which increases the difficulties in droplet injection and manipulation (Dreyer et al. 1994; Meseguer et al. 2014; Stange et al. 2003). Secondly, the inks will solidify on solid substrate either by liquid evaporation or temperature-induced phase change. When the inks are injected and form a sessile droplet, it always forms specific patterns such as the common “coffee ring” (Deegan et al. 1997) under the coupling physical effects after evaporation in normal gravity. However, the printing patterns will be altered for vanishing of gravitational sedimentation and buoyancy convection in microgravity. The difficulty in controlling evaporation kinetics limited their applicability to forming uniform and ordered structures in space. Therefore, studying the droplet manipulation and patterns formation in microgravity is the essential step of achieving DIW in space.

In this work, we have adopted the colloidal suspension as a diluted ink model to study the manipulation of the droplet and the formation of the drying patterns in space. We have studied the fluid dynamics of droplet injection, spreading and wetting on solid surface, and further revealed the self-assembly behavior of colloidal particles and formation mechanisms of the printed patterns under microgravity. All experiments were conducted in the colloidal material box (CMB) aboard the Chinese SJ-10 satellite (Hu et al. 2014).

## Experimental Payload

The CMB is a subsystem and payload of Chinese SJ-10 satellite, as shown in Fig. 1. It is composed of four units: (i) a liquid injection and management unit for uniform dispersion and injection of colloidal suspensions; (ii) a displacement stage for motion and heating of the colloidal droplet; (iii) an optical observation unit for observation of droplet profiles and drying patterns; (iv) a drive control unit for automatic control of the experimental procedures in space. The detailed structures and functions of the CMB can be referred to our previous works (Li et al. 2016a, b). This paper mainly introduces the experimental principle and operation processes as follows: Before the experiment, the polystyrene (PS) colloidal suspension (particle size: 3  $\mu\text{m}$ , concentration: 0.1 ~ 0.15 wt%, Duke (5200)) in the reservoir was mixed thoroughly about 15 min under ultrasonic stirring, which ensures the uniform

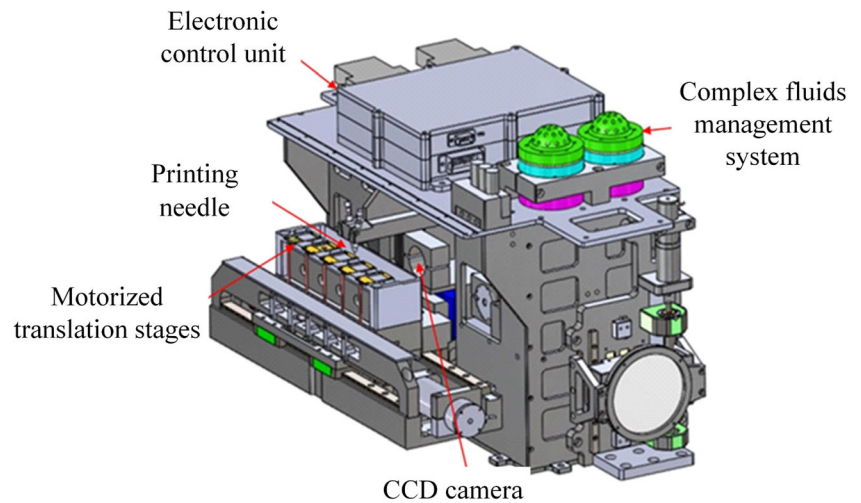
concentration of the colloidal suspensions and prevents the needle blocking. Subsequently, the quartz substrate placed on the displacement stage was precisely positioned under the needle, and the substrate was preheated for above 40–50  $^{\circ}\text{C}$  to accelerate the evaporation. When the experiments began, the colloidal suspension was injected from the printing needle onto the heated substrate and formed sessile droplets, then the colloidal particles self-assembled relying on the interfaces as with droplet evaporation. The injection and wetting process of the droplet could be observed by a macroscopic CCD camera, and particles self-assembling and final depositions could be recorded by a microscope, as shown in Fig. 2. The specific patterns could be obtained theoretically through changing the positions of the displacement stage and repeating the processes of injection and evaporation. The internal structure and functional units of the CMB can be seen in Fig. 1.

To verify the applicability of the DIW technology in space, we had conducted several experiments of droplet manipulation and patterns formation under microgravity conditions. The real-time images and videos of droplet injection and evaporation from a macro camera, and colloidal particles assembly and final patterns from a microscope were simultaneously filmed. Manipulation and evaporation of different initial volumes of colloidal droplets (20–160  $\mu\text{L}$ ) were tested and investigated. In addition, deposition of the colloidal particles relying on interfaces of solid surface was studied in terrestrial gravity and microgravity, respectively.

## Droplet Dynamics of Injection and Wetting

The flow behavior during printing is relied on the rheological properties of the colloidal materials, especially for the extrusion-based systems. For droplet-based printing system, most inks are Newtonian fluids. Therefore, the surface tension of inks are of vital importance to the printing. In fact, injection and spreading of the diluted ink droplet on solid surface were dominated by the coupling effects of surface tension and gravity. The interrelation between gravity and surface tension can be described by the dimensionless Bond number,  $Bo = \rho g L^2 / \gamma$ , where  $L$  is the characteristic length scale,  $\gamma$  is the surface tension,  $\rho$  is the liquid density, and  $g$  is the gravity constant (Diana et al. 2012). In microgravity environment,  $g = 10^{-3} g_0$ , thus  $Bo \ll 1$ . Therefore, the effects due to gravity are negligible, and the liquid shapes from injection is controlled by surface tension. Thus the liquid remains more unstable without the downward restriction of gravity in space, which increases the difficulty of droplet control and management in orbit. We found the injecting liquid was easily to climb along

**Fig. 1** Simulated assembly drawing of the CMB



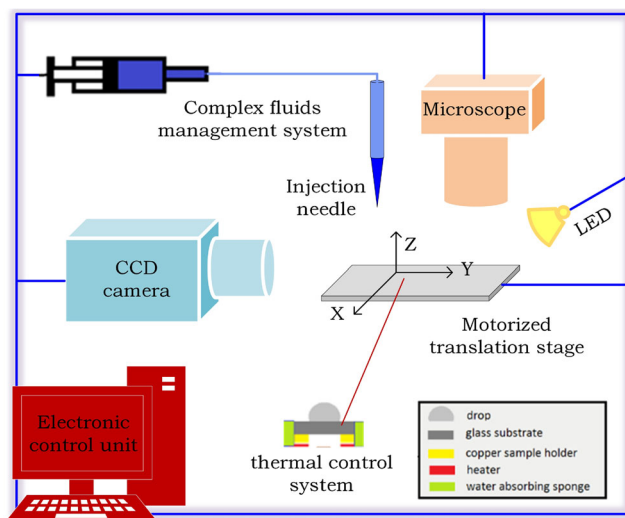
the needles even for the large volume of droplet, which might result in the failure of injection in microgravity, as shown in Fig. 3b. Because of the dominant interfacial tensions, the printing process of injection and spreading can be manipulated through tuning the wettability of the needles and the solid surface, and the surface tension of the colloidal inks.

The water-based PS colloidal suspensions were injected from a miniature electromagnetic pump (LPVA, Lee Co.) at a rate of  $11.42 \mu\text{L/s}$ . The needles (062 MINSTAC, Lee Co.), whose inner and outer diameters were 0.1 and 1.3 mm, respectively, were treated as hydrophobic to repel the water solution from climbing. Two hydrophobic needles were adopted as the printing head, and the hydrophilic quartz glass was chosen as the solid substrate. In the injection process, the droplet first stayed at the pinpoint of the needle. As the liquid volume increased, the droplet would contact the hydrophilic surface and then spread out rapidly, it finally formed the sessile droplet with balanced contact angle and specific contact area. Sizes

of the final pattern is probably fixed already at this spreading stage. It is worth mentioning that the spreading process can be controlled by the roughness and heterogeneity of the solid surface, which implies that the printing patterns can be regulated through modifying the surface properties. In the experiments, a specific hydrophobic circle (diameter:  $\sim 5 \text{ mm}$ ) was designed to restrain the spreading of the colloidal droplet because the aqueous suspension tended to wet the hydrophilic region but dewet the hydrophobic region, as shown in Fig. 3a. The pinned droplet would evaporate as constant contact radius (CCR) mode (Fig. 3c), therefore, we finally obtained patterns with fixed areas and sizes.

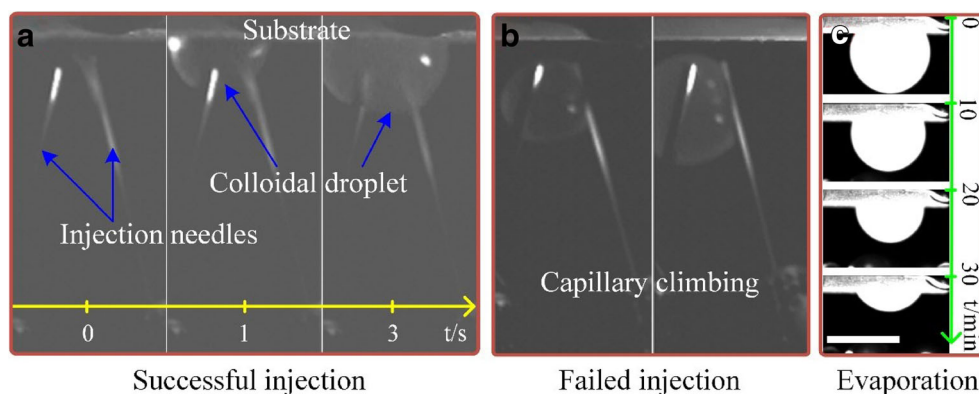
## Colloidal Self-assembly and Patterns Formation

The morphology of the dried patterns that directly reflects the printing quality is highly related to the evaporation process. So it is necessary to investigate the kinetics of evaporation and the dynamics of particles deposition. Previous studies have shown that the deposition patterns are determined by complex interactions of the micro-flow (including capillary flow and Marangoni flow) (Deegan et al. 1997, 2000; Hu and Larson 2005, 2006), contact line behavior (pinning and de-pinning) (Li et al. 2013; Orejon et al. 2011), interfacial effects (such as liquid-air interface capture, liquid-solid interaction) (Bhardwaj et al. 2010; Li et al. 2016a, b; Nguyen et al. 2017), and solutes properties (sizes, shapes, etc.) (Li et al. 2019; Yunker et al. 2011). Therefore, the patterns can be regulated through controlling these multiple physical effects. Despite great process, the gravitational effects on the evaporative convection and deposition still lacks full understanding. Here, we have further investigated the formation mechanism of the drying patterns in microgravity to verify the feasibility of DIW in space.



**Fig. 2** Working principle diagram of the CMB

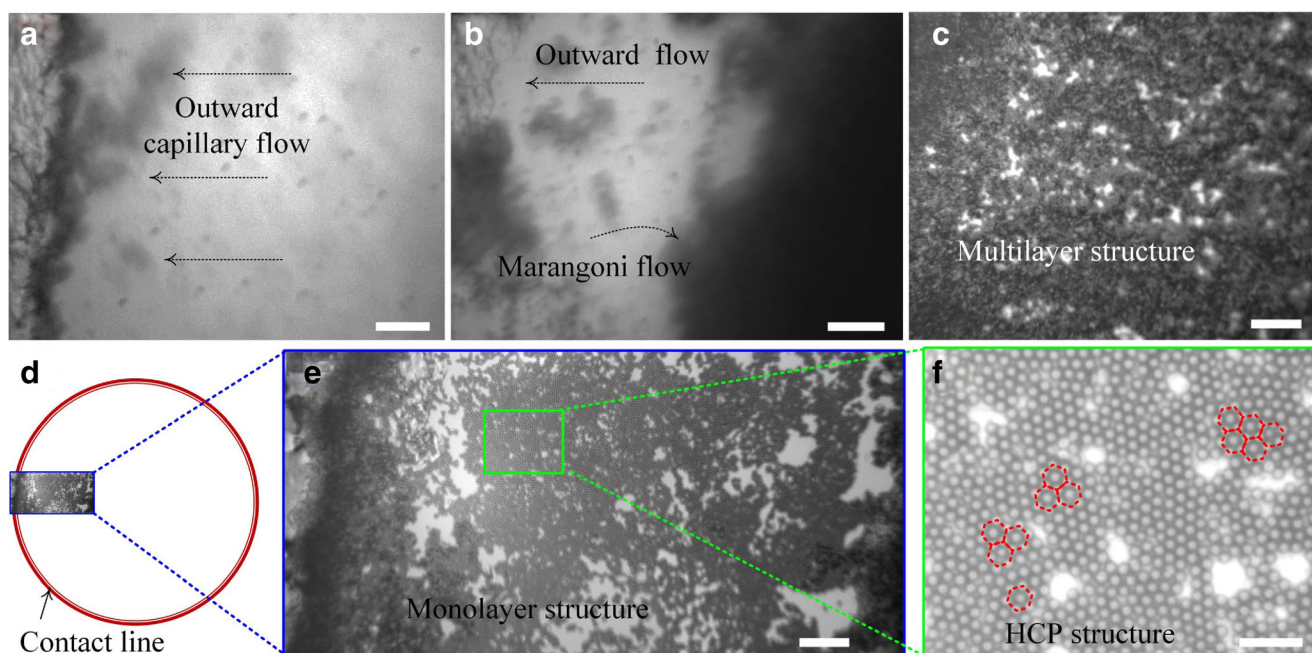
**Fig. 3** **a** Injection of the colloidal droplet. **b** Failed injection due to the droplet climbing along the needles. **(c)** Evolution of the droplet shape as with liquid evaporation, the scale bar is 5 mm



Experimental results show the interfacial effect dominates the deposition of colloidal particles under microgravity environment for the absence of gravity sedimentation and buoyancy convection. Therefore, we focus on the micro-flow and self-assembly dynamics relying on the interfaces of a droplet, including the liquid-air interface, the liquid-solid interface, and the triple interfaces of liquid, solid and air. Figure 4 shows the dynamic deposition of the colloidal particles and the eventual patterns under microgravity condition. When the contact angle was large, most particles were easily captured by the shrinking liquid-air interface and formed islands and pieces of monolayer structures because the gravitational sedimentation was negligible. When the contact angle was small, few colloidal particles were pushed towards the contact line under the outward capillary flow and finally formed the weakened ring-

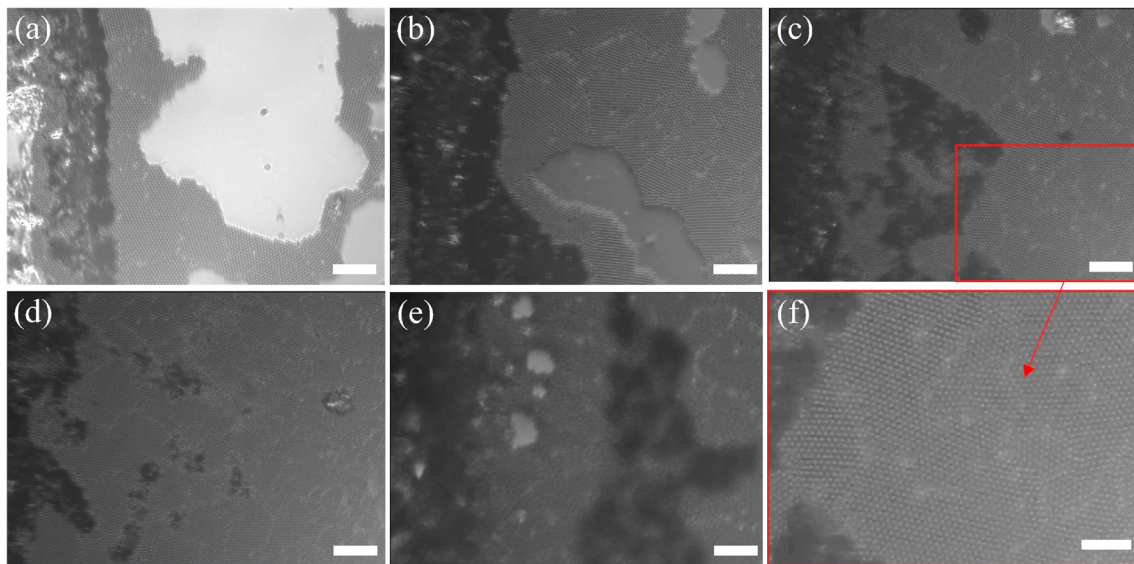
like structure, as shown in Fig. 4a. The capillary flow was resulted from the non-uniform evaporation flux between the edge and the center, which was not influenced by gravity (Deegan et al. 1997). These structures on the liquid-air interface, strongly affected by the Marangoni flow (as seen in the black region in Fig. 4b), would eventually transfer to the solid surface after evaporation and rearranged into monolayer and multilayer structures under the action of liquid film dewetting, as shown in Fig. 4c & e. Figure 4f shows that the monolayers have a stable hexagonal close-packed (hcp) structure containing voids. Therefore, we can obtain more uniform patterns in microgravity because of the “weakened coffee ring”.

To further understand the effects of gravity and heating on the patterns, we had conducted experiments of droplet evaporation with an increased temperature (50 °C) under terrestrial



**Fig. 4** Patterns formation from evaporating droplets on a heating substrate (40 °C) under microgravity. **a** Particles were carried toward the contact line under the outward capillary flow. **b** The self-assembled monolayers at the liquid-air interface moved under the combined action of the Marangoni flow and the capillary flow. **c** Multilayer structures in

the center of the ring. **d-e** The outer ring-like structure and inner monolayer structure with particle-free region. **f** The local region of **e** has hcp structure. The bars of (a), (b), (c), (e), (f) are 30 μm, 50 μm, 60 μm, 60 μm, and 15 μm, respectively



**Fig. 5** Final deposition patterns for colloidal droplets with different initial volumes (a) 30  $\mu\text{L}$  (b) 40  $\mu\text{L}$  (c) 50  $\mu\text{L}$  (d) 60  $\mu\text{L}$  (e) 70  $\mu\text{L}$  on a heating substrate (50  $^{\circ}\text{C}$ ) under normal gravity, the concentration is 0.14 wt%; (f)

is an enlarged image in red square of (c). The scale bar for (a)–(e) is 30  $\mu\text{m}$  and for (f) is 15  $\mu\text{m}$

gravity. The droplet evaporated as CCR mode. Figure 5 shows the deposition patterns for colloidal droplets with different initial volumes (30, 40, 50, 60, 70  $\mu\text{L}$ ; 0.14 wt%). It formed the common ring-like pattern with multilayer structures at the edge of the droplet. This inner structures of the ring show ordered arrays of sub-monolayer, monolayer and bilayer by increasing the initial volumes, as shown in Fig. 5a–e. Figure 5f is an enlarged image in red square of Fig. 5c, which shows that the colloidal particles has a hcp structure. Unlike the ring-like structure caused by capillary flow, the ordered hcp structures in the center could be attributed to interface capture effect. Compared with microgravity environment, there was a stronger “coffee ring” effect in normal gravity because of more particles remaining in the droplet at the last stage of evaporation due to the particles sedimentation and the weak interfacial effects. Liquid-air interface can promote the formation of monolayer ordered structures, because the particles captured by the liquid-air interface always experience the capillary attraction initiated by interfacial deformation. Therefore, substrate heating not only shortened the drying time, but also induced formation of the ordered structures through fast evaporation. This provides an excellent opportunity to print periodic structures.

## Conclusions

In this work, we have designed the CMB as the prototype printer to investigate the applicability of DIW technology in space. Both the fluid dynamics and the patterns formation are the deciding factors in the success of DIW. Therefore, the colloidal suspensions was adopted as a diluted ink model

system to investigate the droplet manipulation and evaporation-induced deposition of colloidal particles in microgravity environment. The dynamics of droplet injection and spreading was firstly analyzed, which shows that the droplet could be manipulated through tuning the wettability of the needle and the solid surface. The self-assembly behavior of the colloidal particles and formation mechanisms of the printed patterns was further revealed, which indicated that the “coffee ring” effect was weakened because of the strong interfacial effect in microgravity. Combined with fast evaporation through substrate heating, it could be able to fabricate more uniform and ordered structures.

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