

## Structural color generation based on grating/colloidal crystal microsphere structure

Li Xiu\*, Zhang Biqian, Lu Qi, Ma Minyue, Duan Shujie, Xu Zhanshun

(School of Printing and Packaging Engineering, Beijing Institute of Graphic Communication, Beijing 102600, China)

**Abstract:** A thorough research of the optical properties of structural color generation based on grating/colloidal crystal was reported in terms of structural design, experimental preparation and spectral color rendering performance testing. Around realizing wide color gamut, narrow band gap, high brightness and anisotropic structure photonic crystal color control problems, the color mechanism of grating/colloidal crystal microsphere composite photonic crystals was explored by gravity deposition and vertical deposition. Taking the gravity sedimentation method, when the concentration of the dispersion was 1%, the colloidal microspheres were uniformly attached to the grating. When the concentration of the solution was 5%, the PS beads can be closely arranged, but cannot form a single layer or double. The layer structure would precipitate multiple layers and the grating structure was not observed. With the vertical settlement assembly method, when the concentration of the dispersion liquid was 1%, the assembly effect was not good and a dense structure couldn't be formed. When the solution concentration was 5%, the composite structure was assembled well, and the colloidal crystal was assembled in the grating groove. The spectral performance was tested and its color properties were evaluated, which laid a theoretical and practical foundation for its effective application in the field of anti-counterfeiting printing.

**Key words:** photonic crystal; polystyrene; grating; self-assembly

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## 基于光栅/胶体晶体微球结构的结构色制备研究

李 修\*, 张碧芊, 卢 奇, 马敏月, 段舒洁, 许展顺

(北京印刷学院 印刷与包装工程学院, 北京 102600)

**摘 要:** 从结构设计、实验制备和光谱呈色性能检测等方面,对基于光栅/胶体晶体结构色的光学性质进行了深入研究。围绕实现宽色域、窄禁带、高亮度和各向异性结构的光子晶体色彩控制问题,通过重力沉降和垂直沉积自组装的方法,探索了光栅/胶体晶体微球复合光子晶体的呈色机理。采用重力沉降法,当分散液浓度为 1%时,胶体微球均匀地附着在光栅上。当分散液浓度为 5%时,胶体微球可以紧密排列,但不能形成单层或双层密排结构,将会沉淀多层,已观察不到光栅结构。采用垂直沉降组合法,当分散液浓度在 1%时,组合法效果不好,不能形成致密结构,当溶液浓度为 5%时,复合结构组合法效果较好,胶体晶体组合法于光栅凹槽中。实验中还进行了光谱性能测试,对其颜色特性进行评价,为其在防伪印刷领域得到有效应用奠定理论与实践基础。

**关键词:** 光子晶体; 聚苯乙烯; 光栅; 自组装

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作者简介:李修(1981-),女,讲师,博士,主要从事印刷电子相关光学后处理以及图案化光子晶体结构色方面的研究。Email:lixu@bigc.edu.cn

## 0 Introduction

In recent years, photonic crystals have made great progress as research hotspots at home and abroad<sup>[1-3]</sup>, and countries have successively produced materials related to photonic crystals, such as photonic crystal fibers, structural color materials<sup>[4-7]</sup>, etc. Photonic crystals can also be used as sensitive materials for various micro-sensors<sup>[8-14]</sup>. Similar to the recent development of semiconductor materials driving the electronics industry, photonic crystals will certainly promote the future development of photonics technology and IT technology industry.

In this paper, the research on composite structure photonic crystals is carried out in terms of structural design, experimental preparation and spectral color rendering performance testing. The coloration mechanism of grating/colloidal crystal microsphere composite photonic crystals is explored, a micro/nano structure with two distinct discoloration effects in two mutually perpendicular directions is designed, and the influence mechanism of materials and composite structures on the color rendering effect is analyzed. An effective color control method is proposed, which is processed and prepared to test its spectral color rendering performance, and its color characteristics are evaluated, which lays a theoretical and practical foundation for its effective application in the field of anti-counterfeiting printing.

## 1 Experiment

### 1.1 Preparation of grating template

The ultrasonically cleaned silicon grating is placed in a low temperature plasma processing apparatus for plasma treatment, the processing time is 5 minutes, the discharge power is 200 W, and the gas flow rate is 60 SCCM, so that it has good hydrophilicity and is easier to obtain better self-assembly effect.

### 1.2 Preparation of grating/colloidal microsphere composite structure

#### 1.2.1 Gravity sedimentation assembly

The gravity sedimentation method is that under the action of gravity, the PS microspheres are settled and arranged in order to form a colloidal crystal structure. In the experiment, conventional gravity sedimentation method was used, and 600 nm polystyrene microspheres and deionized water were taken and matched according to a certain ratio. The concentration selected for the experiment was 1% and 5%. The configured solution was placed in an ultrasonic device and sonicated for 2 min. The glassware with a diameter of 1 cm was cleaned and ultrasonically applied several times, and then placed on the surface of the water platform. The processed grating was cut to the corresponding size and placed horizontally therein, and the 600 nm polystyrene microsphere solution was dropped into the solution. In the container, the grating is completely immersed. The amount of drip should be controlled to ensure that the liquid instilled each time is the same. The experimental setup is shown in Fig.1.

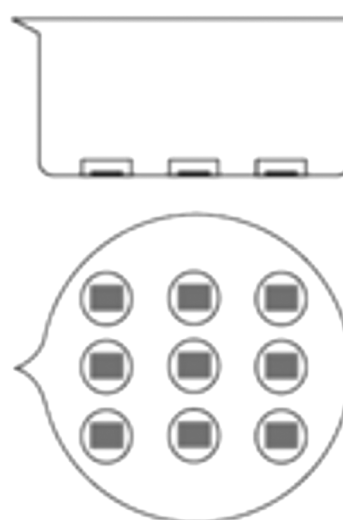
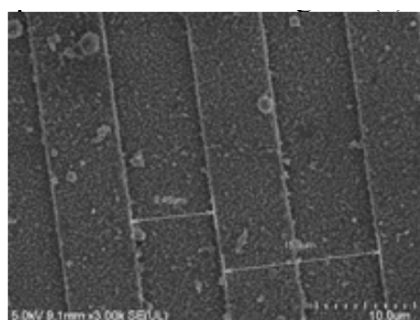


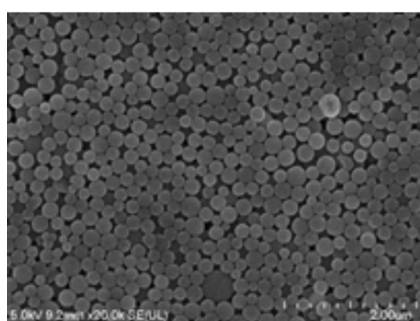
Fig.1 Schematic diagram of experimental apparatus for gravity sedimentation

After standing at room temperature for 48 hours, the sample was taken out. The results of the obtained

finished product observed under a scanning electron microscope are shown in Fig.2(a) and (b).



(a) Concentration is 1%



(b) Concentration is 5%

Fig.2 SEM image of gravity settlement of PS microsphere/grating composite structure at different concentrations

It can be seen from the figure that when the concentration of the dispersion is 1% , the colloidal microspheres are uniformly attached to the grating. When the concentration of the solution is 5%, the PS beads can be closely arranged, but cannot form a single layer or double. The layer structure will precipitate multiple layers and the grating structure has not been observed.

### 1.2.2 Vertical settlement assembly

The vertical sedimentation method is a substrate which has a smooth surface and good wettability, and is immersed in a dispersion of the prepared PS microspheres at a vertical angle, and the colloid is made by capillary force during evaporation of the solvent. The order of the particles is arranged. The experiment selected 600 nm polystyrene microspheres and deionized water, and matched them according to a certain ratio. The concentration selected for the experiment was 1%

and 5% . The configured solution was placed in an ultrasonic device and sonicated for 2 min.

The 10 cm diameter glassware was washed several times with deionized water and ultrasonically washed twice for approximately 10 min each. After washing the 1.5 mL centrifuge tube and ultrasonicing several times, it was placed in a horizontal tank, the grating was cut to the corresponding size and then placed vertically, and the 600 nm polystyrene microsphere solution was dropped into the centrifuge tube to all immersed. The experimental setup is shown in Fig.3.

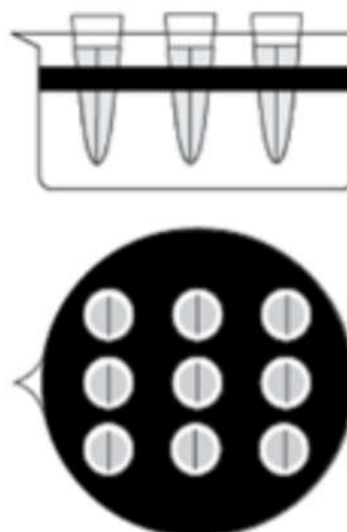
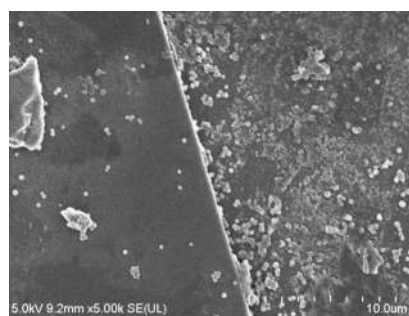
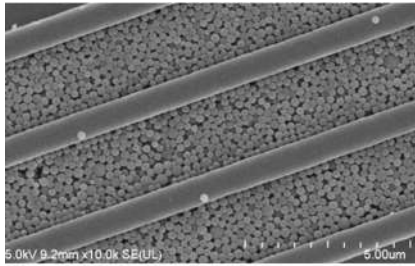


Fig.3 Schematic diagram of the experimental device for vertical sedimentation

After standing at room temperature for 48 hours, the sample was taken out. The results of the obtained finished product observed under an electron microscope are shown in Fig.4.



(a) Concentration is 1%



(b) Concentration is 5%

Fig.4 SEM image of vertical sedimentation assembly of PS microsphere/grating composite structure at different concentrations

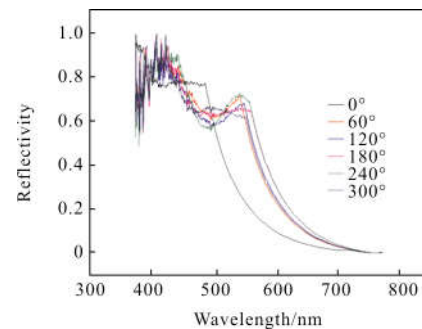
It can be seen from the figure that when the concentration of the dispersion liquid is 1% , the assembly effect is not good and a dense structure cannot be formed. When the solution concentration is 5% , the composite structure is assembled well, and the colloidal crystal is assembled in the grating groove.

Through experiments, it is found that the gravity sedimentation method requires stricter solution concentration. In general, the concentration of 1% PS pellet solution can obtain the best experimental results. The low dispersion concentration allows for a tight alignment on the substrate. And the low concentration can clearly see the grating structure under the scanning electron microscope, and the sample can see the more obvious rainbow color. When the concentration of the solution is more than 5% , the PS pellet will precipitate a plurality of layers, and a good experimental effect cannot be obtained. The vertical sedimentation method slightly expanded the concentration range of the required solution, and the silicon wafer grating obtained a better experimental sample at a concentration of 5% . The microspheres are well filled into the gaps of the grating plates, and the test samples have good coloring effects.

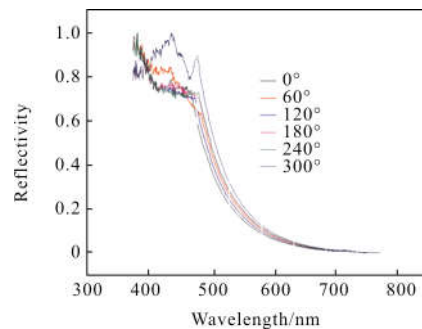
## 2 Spectral properties of grating/colloidal crystal structure

In the experiment, the reflectance spectra of the samples by gravity deposition method (concentration:

1%) and vertical sedimentation method (concentration: 5%) were measured by a built-in angular resolution spectrometer. The incident angle are 15° and 30° . During the experiment, the sample is rotated horizontally at different angles for measurement, that is, the measurement equipment is measured at six angles of 0°, 60°, 120°, 180°, 240°, and 300° in the horizontal direction, that is, every other measurement. The measurement sample data is taken once at 60°, and the measurement data is saved. The reflection spectrum of the silicon grating was measured experimentally, and an image was drawn, as shown in Fig.5 and Fig.6.

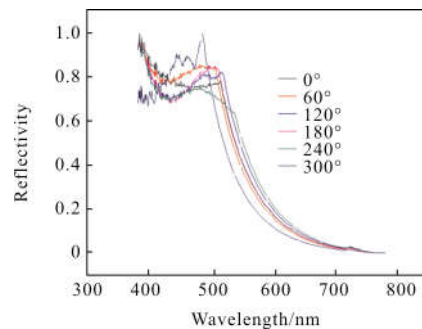


(a) Incident light is 15°

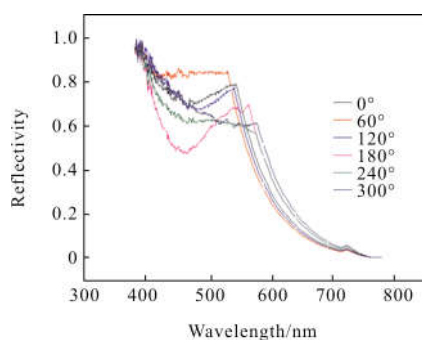


(b) Incident light is 30°

Fig.5 Sample reflection spectrum of 1% concentration of gravity sedimentation assembled



(a) Incident light is 15°



(b) Incident light is 30°

Fig.6 Sample reflection spectrum of 5% concentration of vertical sedimentation assembled

As can be seen from Fig.5, when the detection angle is 15°, the reflection spectrum of the 1% concentration gravity sedimentation assembly sample, the reflection peaks around 450 nm and 550 nm, and the position of the reflection peak does not change greatly with the change of the horizontal rotation angle of the sample. After changing the detection angle, the position of the reflection peak is blue-shifted obviously, but the effect of the horizontal rotation angle is still small.

When the detection angle is 15°, the reflection spectrum of the 5% concentration vertical sedimentation assembly sample, the main peak position is basically located substantially at 500 nm. With the change of the horizontal rotation angle, the reflection peak position is abnormal at individual angles, but with little change in the overall; When the detection angle is 30°, the position of the reflection peak is red-shifted and changes around 550 nm.

### 3 Conclusion

In our work, the composite structure of grating/colloidal crystal is fabricated by gravity deposition and vertical deposition, and the optical effect is discussed. Gravity sedimentation self-assembly is suitable for preparing large-area samples and is easy to operate, but its most prominent deficiency is that it is difficult to control the surface morphology of the colloidal crystal and the number of layers of the crystal, and

the time of preparation is relatively long. Vertical sedimentation of self-assembly as a superior method of assembling, but it still needs to control the temperature and humidity during the assembly process, which brings certain inconvenience to the experimental process.

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