Deterministic and controllable photonic scattering media via direct laser writing

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Abstract

Photonic scattering materials, such as biological tissue and white paper, are made of randomly positioned nanoscale inhomogeneities in refractive index that lead to multiple scattering of light. Typically these materials, both naturally-occurring or man-made, are formed through self assembly of the scattering inhomogeneities, which imposes challenges in tailoring the disorder and hence the optical properties. Here, we report on the nanofabrication of photonic scattering media using direct laser writing with deterministic design. These deterministic scattering media consist of submicron thick polymer nanorods that are randomly oriented within a cubic volume. We study the total transmission of light as a function of the number density of rods and of the sample thickness to extract the scattering and transport mean free paths using radiative transfer theory. Such photonic scattering media with deterministic and controllable properties are model systems for fundamental light scattering in particular with strong anisotropy and offer new applications in solid-state lighting and photovoltaics.

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I. INTRODUCTION

The scattering of light is a familiar physical process that is abundant in everyday life and in nature; manifestations of scattering are the opacity of white paper, of clouds, and of biological tissue [1–16]. Light scattering occurs at any interface between different materials and causes a part of the light to deviate from its original path. A medium with a high density of such interfaces can be a strong, multiple-scattering material. The strength of the light transport through and interaction with the scattering medium is quantified through the following length scales. The scattering mean free path $\ell_{sc}$ quantifies the mean distance between subsequent scattering events. The mean distance that the incident light propagates before its direction is scrambled is the transport mean free path $\ell_{tr}$. The transport mean free path $\ell_{tr}$ sets the typical length scale for a medium to be diffusive ($L > \ell_{tr} > \lambda$, where $L$ is the thickness of the scattering medium $\lambda$ is the wavelength of light in the embedding medium), and is therefore relevant if such a scattering medium is applied in devices such as solar cells and white LEDs. In solar cells, a thin scattering layer on top of the cell increases the absorption of sunlight and hence the cell’s efficiency [14]. White LEDs employ a layer of scattering and phosphor particles to control the spectral and spatial distribution of the emission [13, 16].

Applications of scattering media often require a specific optical thickness, which is defined as the ratio of the physical thickness $L$ and transport mean free path $\ell_{tr}$. This ratio defines the scattering regime (ballistic, diffusive, localization [2, 5, 7]) and is controlled to meet the demands of an application. For example, if there is demand for high opacity diffusing medium, then the physical thickness should exceed the transport mean free path ($L/\ell_{tr} > 1$). Today LED devices have dimensions down to the few $\mu$m and thin film solar cells as well. Therefore, such devices benefit if the scattering mean free path is in the $\mu$m regime. One strategy to minimize the scattering mean free path is to increase the number of interfaces in the medium.

A large number of scattering media with well-defined scattering properties, covering a broad range of optical thicknesses have been presented in the literature [17–22]. However, the fabrication of those media rely on random processes and only the average values are controlled during fabrication. To know the detailed internal structure of these media requires careful dissection of the media and sub-micron inspection, destroying the medium in the
process. On the other hand, a scattering medium with a precisely known internal structure has become feasible as a result of the recent advances in 3D nanofabrication [24–26]. An a priori knowledge of the internal structure, without the need of sacrifice of the medium to extract the internal structure, makes it ideal for studies of light propagation that are sensitive to microscopic wave interference of light. In literature, several scattering media with deterministic geometry and scattering properties have been demonstrated. Matoba et al. [27] have created a large-volume deterministic scattering medium with laser micromachining [28, 29], but with undefined scattering strength. An alternative nanofabrication technique is direct laser writing (DLW) [25], offering about 1...10 nm material deposition precision and a feature size down to about 100 nm [24]. The physical principle of DLW is two-photon polymerization to initiate polymerization at a targeted location inside the volume of the photoresist. The advantage of DLW is the freedom to create deterministic and complex 3D geometries. An example of a DLW-nanofabricated photonic structure with disordered geometry is the hyperuniform disordered medium made by Haberko et al. [30–32]. In the current work, we report on the fabrication of deterministic scattering media that combine both a large anisotropy and a short scattering mean free path. We measure the transport mean free path of the structures and validate that light scattering in our structures is in the diffusive regime ($L/\ell_{tr} > 1$).

II. METHODS

A. Direct laser writing

The structures were fabricated with a direct laser writing system [25] (Nanoscribe Professional GT) in the University of Twente Nanolab. The structures were made using polymer photoresist (Nanoscribe IP-G) with a refractive index of 1.51 [33]. The photoresist is a gel and its high viscosity ensures that the features in the structures do not drift during the writing processes, minimizing possible deformation of the structures. The illumination dose was set to be 14% higher than the polymerization threshold (laser beam power 8.90 mW and piezomotors scan speed of 140 $\mu$m/s). This results in rods of an average thickness of 500 nm instead of 100 nm that one could reach with a smaller illumination dose. Although the choice of a dose closer to the polymerization threshold would provide finer features [34], the slightly
higher dose provides two advantages in the nanofabrication of those media. Firstly, thicker rods provide a more robust mechanical support of the structure; this is important because the design relies on the self support of the whole structure without any additional walls, unlike earlier demonstrations [35]. The second advantage of a higher dose is the reduction of fabrication disorder through the complete cross-linking of polymer chains across the entire volume that improves mechanical stability [36].

B. Designing a deterministic scattering medium

The scattering medium consists of rods that randomly fill a cubic volume. The randomly intersecting polymer rods run from one facet of the cube to another. An important parameter in our fabrication is the filling fraction of polymer, which is set by the number of rods written in the cubic volume. The desired filling fraction is the one that maximizes light scattering. Given the rod thickness, this is equivalent to maximizing the air-polymer interface. To create a free-standing structure, we require a minimum number of rods that intersect to form a rigid
skeleton that does not collapse under its own weight or under capillary forces. If we increase the number of rods above a certain critical value, we start to decrease the air-polymer surface, reducing the scattering strength of our structures. A design example is shown in Fig. 1b). We developed an algorithm based on Jaynes’ solution to Bertrand’s paradox \[37\] to ensure an on-average uniform filling of the volume and the random positioning of the rods in the cube. To keep fabrication times acceptable (7 minutes per structure), we created structures with a lateral size of 15 to 20 \(\mu\)m and a height up to 20 \(\mu\)m. For structures wider and taller than 20 \(\mu\)m, we suggest the model to be divided in cubes of \((20 \mu m)^3\) to prevent pyramidal distortions, shadowing effect and weight deformations \[38, 39\].

The DLW setup is ideally suited to write rods in a successive manner \[40\]. After we generate the coordinates of the rods, we sort them such that the rods attached to the substrate are written first, as illustrated in Fig. 1(a). This creates a stable scaffold to which the other rods are attached to. At the end of the writing process, a rigid structure is created to follow the design model as is shown in Fig. 1(b).

In Fig. 2 we plot the estimated filling fraction of the structures corresponding to increasing number of rods given the structure volume. The deterministic fabrication of our structures allows us to calculate the filling fraction of the structures, given the knowledge of their design. \[1\] The estimated filling fraction also allows us to estimate the effective refractive index, indicated in Fig. 2 by the second ordinate. In the same figure, one can see that for the series of 15 \(\mu\)m and 20 \(\mu\)m lateral dimensions there are filling fractions that match for a different number of rods.

In order to study the total transmission and extract the transport mean free path, a set of structures is needed with varying height at a given filling fraction \[7, 41\]. The uniform density of the scatterers certifies an on-average uniform filling fraction also in the structures with a reduced height. In addition, we keep the design of the structures constant but tune the height of our structures by writing deeper in the glass. In this way we avoid the transmission noise that may be introduced by different realizations. In order to study the scattering properties of this design, we fabricated two series of structures with lateral dimensions of 15 and 20 \(\mu\)m. The first series are made of 200 up to 2000 rods/(15\(\mu\)m)\(^3\) with a step size of

\[1\] The fill fraction is numerically estimated by first discretizing the design volume into cubic pixels (voxels) with dimensions of \((20 \text{ nm})^3\) and then binarizing the discretized volume into written and unwritten blocks. The infinitesimal lines in the design are converted to finite thickness by assuming a laser focus of ellipsoidal volume with a diameter of 500 nm and an elongation of 3.5 times the diameter.
Figure 2. Estimated filling fraction and effective refractive index versus the density of rods over volume. Red markers and line denote how the filling fraction increases with increasing density of rods per volume for structures with volume $(15 \, \mu m)^3$, while the black markers show the same trend for cubes of $(20 \, \mu m)^3$. The estimation was performed by numerically integrating the volume of the intersecting rods.

200 rods/$(15 \, \mu m)^3$, corresponding to a filling fraction from 32 vol% up to 97 vol%. For each filling fraction we also varied the height from $1.5 \, \mu m$ up to $15 \, \mu m$ with steps of $1.5 \, \mu m$. The second series is composed of 600 rods/$(20 \, \mu m)^3$ up to 2400 rods/$(20 \, \mu m)^3$ with a step size of 200 rods/$(20 \, \mu m)^3$, corresponding to a filling fraction from 48 vol% up to 93 vol%. For each filling fraction we also varied the height from $6.5 \, \mu m$ up to $20 \, \mu m$ with a step of $1.5 \, \mu m$. The series of structures with varying heights are used to extract the transport parameters and quantify the scattering strength of structures following this design.

C. Optical setup

The light scattering strength in our samples is quantified by extracting the transport mean free path $\ell_{tr}$ using total transmission measurements [7]. The experimental set-up is
Figure 3. Schematic of the set up for measuring the total transmission and ballistic light attenuation. a) A continuous wave He:Ne laser at 633 nm is expanded to a diameter of 3.1 cm and focused on a single structure with an aspheric lens F1 with a focal length of 100 mm. A microscope oil immersion (Fl.) objective with NA=1.4 collects the transmitted light from the glass surface. Illumination of a specific structure is controlled with the use of a 3-axis piezo positioning stage. We use a CCD camera to confirm correct positioning while the total transmission is recorded with a power meter (D1). The light that is reflected on BS2 is used to evaluate the ballistic light attenuation. An iris with a polarizer aligned parallel to the polarization of the laser beam filters out the scattered light. The filtered light intensity is measured with a power meter (D2).

shown in Fig. 3. The laser beam from a linearly-polarised He-Ne laser ($\lambda = 633$ nm, beam diameter of 3 mm) is expanded 10× with a beam expander and focused with a plano-convex lens (focal length = +100 mm) onto the sample. The focused beam has a full width half maximum of 4 $\mu$m, smaller than the lateral dimensions of 15 and 20 $\mu$m of our structures. An oil-immersion objective (numerical aperture NA=1.4) is used to collect the light transmitted light through the photonic scattering media. We position each structure with a 3-axis piezo positioning stage, while viewing with a CCD camera. The light collected by the objective
is split with two non-polarizing 50:50 beam splitters (BS1 and BS2). The transmitted part of BS3 is sent to power meter 1 (D1, Ophir Nova 2) to obtain the total transmission. The reflected part of BS2 is used to monitor the ballistic light. For this reason it is filtered with an adjustable iris. A polarizer filters out multiple-scattered light, allowing only polarization components parallel to the original beam polarization to be transmitted to power meter 2 (D2, Ophir Nova 2).

The total transmission $T_z$ for a sample of thickness $L = z$, was measured by recording the total power of the light $I_z$ that was transmitted through the structure with given thickness $z$. The reference power $I_0$ was measured on a bare glass substrate. The ratio of the total power for given thickness $z$ and the reference $I_0$ is the total transmission of the sample, $T_z = I_z/I_0$. In a similar manner, the ballistic light attenuation $B_z$ is measured as the ratio $B_z = P_z/P_0$ with $P_z$ the power of light that propagates through a sample of thickness $z$. To extract the transport parameters of $\ell_{tr}$ and $\ell_{sc}$, we model the measured data with the P3 approximation of radiative transfer theory [42, 43].

III. RESULTS

We have fabricated 693 structures with various thicknesses and densities of scatterers and employed scanning electron microscope (SEM) to determine their feature sizes. Figure 4 shows a qualitative comparison of the surface features in the designed (a, c) and the fabricated structures (b, d) that highlights a high degree of similarity. The rod thickness was measured to be $524 \pm 60$ nm. The elongated focus of the laser beam in the resist results in an elliptical cross-section of the rods with the semi-major axis along $z$. The resulting size asymmetry of the voxel is 3.5. We note that the elongation of the rods depends on its angle with respect to the $z$-axis [25]. Although the resemblance between the design and real structure is very good and features remain in the same relative positions as seen in Fig. 5, there are a few errors in the fabricated structure, for e.g. missing rods and shrinkage artifacts. Typically, the very short rods on the cube faces are washed away during development due to insufficient adhesion. The total number of missing rods is small (few dozen) and their contribution to the structure is very small, estimated to be less than 2 vol% and not easy to discriminate from the final structure as shown in Fig. 5. In addition, most fabrication errors repeat in different realization of the structure, making it feasible to compare their
Figure 4. Design of a scattering sample of polymer rods a) in bird’s eye view and c) from above. b) and d) show SEM images of the fabricated structures according to the designs a) and c). We have highlighted several nanorods to ease the comparison of the model and the fabricated sample.

optical interference properties. Moreover, the SEM images show no significant pyramidal distortions [30], a distortion that DLW structures frequently suffer from [39, 40]. Thus we conclude that the chosen height and laser dose were a good choice and proceed to the optical characterization of the structures.

Figure 6(a) shows the sample thickness dependence of the ballistic light transmission in the series of structures with lateral dimensions of 15 µm and 400 inscribed rods. The ballistic transmission decreases with increasing sample thickness, while the highest drop occurs for 400 rods/(15µm)^3, signifying the smallest value for \( \ell_{sc} \). We used non-linear least squares fitting of the experimental data with an exponential decay function \( P = P_0 \exp(-z/\ell_{sc}) \), finding a scattering mean free path \( \ell_{sc} = 2.6 \pm 1.5 \) µm where the error is the 95% confidence interval. This value confirms that our media are in the multiple-scattering regime since their thickness is at least 5 times the scattering mean free path.

In the color graph of Fig. 6(b) we plot the ballistic light measurements for the series with lateral dimensions of 15 µm. On the x-axis, the thickness of the structure increases, while
Figure 5. Top view of model and SEM picture of the fabricated structure. One can see that the features evolve continuously between model (shaded in green) and the fabrication (grey SEM image). Some of the smaller rods may not adhere to the main skeleton, but their final contribution is small (~2 vol%).

The y-axis shows the number of rods composing the full structures.

The total transmission measurements are shown in Fig. 7 for structures with edge lengths of 15 µm and 20 µm in (a) and (b), respectively. The total transmission decreases with increasing thickness of the structure. The decrease is stronger for structures with 400 rods in and 600 rods in (b). This is sensible since they correspond to the filling fractions that maximizes air-polymer interface [41, 44, 45], see Fig. 2.

To check the consistency of the transmission data for different linked dimensions at identical filling fractions (~93%), two total transmission series are made: for the 15 µm cubes with 1400 rods and for the 20 µm cubes with 2400 rods. The structures’ lateral dimensions and the number of rods differ, but since the filling fractions coincide, the light transport is expected to be similar. This consistency is validated from Fig. 8 and highlights that the design method is effective for the chosen finite lateral dimensions and scatterer geometry.
Figure 6. Ballistic light transmission versus thickness for samples with lateral dimension of 15 µm. a) The experimental data are presented with the black markers, while the red solid line shows the fitting to Lambert-Beer law with a scattering mean free path of \( \ell_{sc} = 2.6 \pm 1.5 \) µm. The fitting error is 95% confidence interval, shown with the black dashed line. b) The color graph depicts ballistic light transmission for the structures as a function of the rods/volume (x-axis) and thickness (y-axis). The ballistic light attenuates with thickness, as expected. The largest decay of the ballistic light with thickness appears for 400 rods.

### A. Transport mean free path

We obtain \( \ell_{sc} \) and \( \ell_{tr} \) from two of the methods making standard approximations. The transport properties of a scattering medium are derived from radiative transfer theory (RTT). Light transport Monte Carlo methods can solve complex geometries and finite boundary conditions [46]. Those quantities for a non-absorbing medium are connected through the anisotropy factor \( g \), as \((1-g)\ell_{tr} = \ell_{sc}\). In the case of our DLW structures that are composed from rods with a diameter on the scale of the wavelength, a substantial anisotropy factor is expected [47]. In such a case, there are several approaches that can be used to extract the transport parameters from experimental data [6, 48, 49]. We decided to make an analysis combining a MC parameter estimation [46, 50] and the so called P3 approximation to radiative transfer [16]. In the MC diffusion parameters estimation, one numerically solves radiative transfer theory by considering the geometry of the system for a range of transport...
Figure 7. Total transmission of the samples as a function of their thickness. In a) three series of samples are presented with different markers, corresponding to density of rods of 200, 400 and 1200 rods and with lateral dimensions of 15 $\mu m$. In b) three more cases presented, 600, 1200 and 2200 rods and with lateral dimensions of 20 $\mu m$. In both cases the media exhibit strong attenuation of the total transmission that can be tuned with the design parameters.

Parameters that match with experimental observations. The P3 approximation is an approximation to the radiative transport equations that expands the validity to a wider range of anisotropy values compared to the normal P1 approximation. P3 has been shown to be very accurate in transport parameters estimation \[16, 42\]. In the P3 approximation total transmission measurements are combined with the ballistic light measurements to extract the anisotropy factor $g$ and evaluate the transport mean free path $\ell_{tr}$. Although there is practically no absorption of the photoresist for 633 nm illumination, during the P3 modeling we allowed for an "effective absorption" $\ell_{a}$. This effective absorption takes into account the scattered light that leaks from the sides of the cubic structures.

In Fig. 10 we show two cases of total transmission as a function of sample thickness, for structures of 400 rods with lateral dimensions of 15 $\mu m$ and 600 rods with lateral dimensions of 20 $\mu m$. In the 400 rods series one can see that the P3 estimation matches well with a scattering mean free path of $\ell_{sc} = 2.64 \pm 0.60 \mu m$, anisotropy of $g = 0.90 \pm 0.02$ and "effective
Figure 8. The total transmission values for structures of different density of rods over volume but the same filling fraction. The red markers correspond to experimental measurements series with 1500 rods in a (15 $\mu$m)$^3$ cube while the blue markers correspond to series with 2400 rods in a (20 $\mu$m)$^3$ cube. The filling fraction for the two series is 93% and both trends match well within error bar, proving consistency in our fabrication and design.

absorption” length of $\ell_a=20\pm2\,\mu$m. The estimation using the P3 approximation tends to deviate from the data at small sample thicknesses. This is because the accuracy of the P3 approximation is limited at small sample thicknesses ($L < l_{tr}$). From the P3 calculation, we estimate a transport mean free path $\ell_{tr}=26\pm8\,\mu$m. The second series of 600 rods interprets reasonable well with $\ell_{sc}=2.54\pm0.15\,\mu$m, $g=0.95\pm0.01$ and $\ell_a=13.1\pm0.4\,\mu$m. With the above estimations, the transport mean free path estimation by the P3 appears for the given structure is $\ell_{tr}=56\pm11\,\mu$m. This rather high value is attributed to the high anisotropy value that is estimated. For comparison we also use a MC approach to extract the $\ell_{sc}$ and $g$ values from the total transmission. The code is scanning a range of possible values of $\ell_{sc}$ between 0.1 and 300$\mu$m and $g$ between -1 and 1. The estimations are then modeled with the least-square difference method to the experimental data for the total transmission, to find the best matching quantities \[50\]. For the two series in Fig. 10 the MC estimates for the 400 rods yield a scattering mean free path value $\ell_{sc}=1.8\pm0.5\,\mu$m and $g=0.85\pm0.04$, corresponding to a transport mean free path of $11.8\pm4.5\,\mu$m, while for the 600 rods it estimates a scattering
Figure 9. Total transmission of the samples as a function of the density of rods over volume for given thickness. At the plot a), the markers denote the sample size for given volume \((15\mu m)^3\) and increasing number of rods. At the plot b) the markers refer to the total transmission measurements versus increasing density of rods over volume for samples of volume \((20\mu m)^3\). Both graphs show agreement with Fig. 2, where the lower transmission appears for filling fraction of 50%.

Mean free path value \(\ell_{sc}=2.8\pm0.3\mu m\) and \(g=0.65\pm0.04\). These values correspond to a transport mean free path \(8.3\pm1.2\mu m\). Although both approaches yield similar results for \(\ell_{sc}\) and \(g\), the dependence of \(\ell_{tr}\) on \(g\) results in very different \(\ell_{tr}\). We listed the various results in Table II.

The MC approach is a numerical solution of RTT which has been validated by statistical iterations and careful parameter scanning for the exact geometry and boundaries effects [51], but the actual microscopic structure of the sample and in particular its anisotropy caused by the laser focus elongation is not taken into account. The P3 result is still an approximate solution for infinite slabs, where we introduced an effective absorption to model light leaking out of the finite structures, all in all, we trust the estimation of the MC better. In conclusion, our estimation for the transport mean free path is \(11.8\pm4.7\mu m\) for 400 rods and \(8.3\pm1.2\mu m\) for the 600 rods series. Thus the DLW structures are diffusive since their thickness surpasses the transport mean free path \(L > \ell_{tr}\).
Table I. Transport parameters

|         | 400 rods/(15µm)^3 | 600 rods/(20µm)^3 |
|---------|-------------------|-------------------|
| P3      |                   |                   |
| ℓ_{sc}  | 2.64 ± 0.60 µm    | 2.54 ± 0.15 µm    |
| g       | 0.90 ± 0.02       | 0.95 ± 0.01       |
| ℓ_{tr}  | 26 ± 8 µm         | 56 ± 11 µm        |
| Monte carlo |             |                   |
| ℓ_{sc}  | 1.8 ± 0.5 µm      | 2.8 ± 0.3 µm      |
| g       | 0.85 ± 0.04       | 0.65 ± 0.04       |
| ℓ_{tr}  | 11.8 ± 4.5 µm     | 8.3 ± 1.2 µm      |

Figure 10. Total transmission of the samples versus thickness. The symbols are the experimental measurements for two samples series, black squares depict the samples with lateral dimensions of 15 µm and 400 rods, while red circles represent samples with lateral dimensions 20 µm and 600 rods. The curves show the calculated P3 to match the measurements, the dashed black curve matches for scattering mean free path of ℓ_{sc}=2.64 µm, and the red solid curve corresponds to ℓ_{sc}=2.54 µm.

IV. CONCLUSION

We have implemented a DLW method to fabricate small deterministic optical multiple scattering media with optical thickness \( \frac{L}{\ell_{tr}} > 1 \). The fabrication process allows full control
over the position and shape of the scatterers. We show that one can tune the density of scatterers and accordingly vary the transport mean free path. We demonstrate that our best design has a scattering mean free path of $\ell_{sc} = 1.8 \pm 0.5 \mu m$. The deterministic nature of fabrication makes the scattering samples extremely interesting for light propagation studies. This permits validation of various fundamental and applied aspects of light scattering for a given disordered structure, something that was not possible until now for optical frequencies [52]. In the future we want to investigate the reproducibility of the method to study the clonability of multiple scattering media as optical physical unclonable functions [53].

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