OCT FOR DEPTH DETECTION OF BURIED PARTICLES IN POLYMERIC MATERIALS

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Introduction

Optical coherence tomography (OCT) is a non-invasive, tomographic and three-dimensional imaging technique capable of detecting sub-surface occlusions at a depth of up to 2 mm with micrometer-level resolution. Among its purposes, optical phantoms made from polymeric materials are used routinely for the validation of physical models and simulations, instrument performance testing and optimization, instrument calibration and testing of stability and reproducibility, and inter-laboratory comparison and standardization. Challenges, however, exist in optical phantom standards work due to the lack of uniformity and the lack of a "gold standard" for OCT device inter-comparison.[1] In this work, we demonstrate a method to produce novel optical phantoms usable for the characterization of OCT axial resolution and contrast. By varying the diameter of the microspheres and the thickness of the polymer layers, different spatial frequencies can be replicated in the axial dimension of the phantom. These frequencies provide a methodology to determine the axial contrast transfer function for an OCT system. Because the phantom dimensions can be determined and verified independently of the OCT measurement, no information about the system's spatial calibration is required. Such a test system will enable a more accurate and repeatable determination of buried occlusion/particle distribution within polymer coatings, adhesives and biological materials.

Experimental

The multilayered phantom was prepared by first dip-coating 3.5 bilayers of polyelectrolytes to adsorb a monolayer of spin-coated polystyrene (PS) microspheres on a glass slide.[2] Bilayers were formed using layer by layer assembly from aqueous solutions of poly(allylamine hydrochloride) (PAH) and poly(sodium 4-styrene sulfonate).[3] Due to the opposing charges on each polymer, alternating depositions built a polyelectrolyte multilayer with a specific charge at the surface. The negatively-charged microspheres were adsorbed the multilayer with positively-charged PAH at the surface. The particle layer attached to the layer by layer surface is shown in Figure 1.

A poly(ethylene glycol) dimethacrylate (PEGDMA) formulation was then coated on the PS monolayer and laminated with a fixed spacer height. The monomer was photopolymerized using a Novacure 2000 ultraviolet mercury arc lamp (EXFO, Mississauga, ON) at a light intensity of 10.0 mW/cm^2 for 600 s. Once polymerized, the polymer film had embedded PS microspheres at the glass surface. When the glass was delaminated from the film, the particles remained in the PEGDMA film and left a layer of PS particles at the exposed surface. Additional particle/polymer layers could be assembled on top of the first instance by repeating the procedure and increasing the spacer thickness.

A Fourier-domain OCT imaging system was implemented using a spectrometer that is read with a high-speed line scan camera. The interferometer functioned as a spectral filter with a periodic output spectrum depending on pathlength mismatch. Fourier transforming of the output interference spectrum yielded a measurement of the echo time delay of backscattered light. The axial resolution in OCT is determined by the coherence length of the light source, which varies dependent on the chosen light source. This work uses a 1310 nm center wavelength, 50 nm bandwidth light source for all measurements.

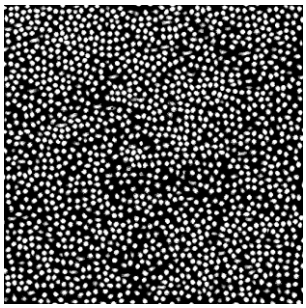

Figure 1. Darkfield image of a dense layer of 5 μm polystyrene microspheres . Image size is 200 μm square.

Results and Discussion

Particles of varying sizes (1 μm, 5μm, and 10 μm) were embedded into a single PEGDMA sample, then scanned using OCT to obtain an axial profile of the PS monolayers in Figure 2a. The axial profiles exhibited characteristic widening of the respective peaks with increasing particle sizes with the peak centered at the axial distance from the surface. Particle layers with large gaps in surface coverage have less than uniform intensity, and this effect is apparent in the inset OCT image for the 10 μm layer.

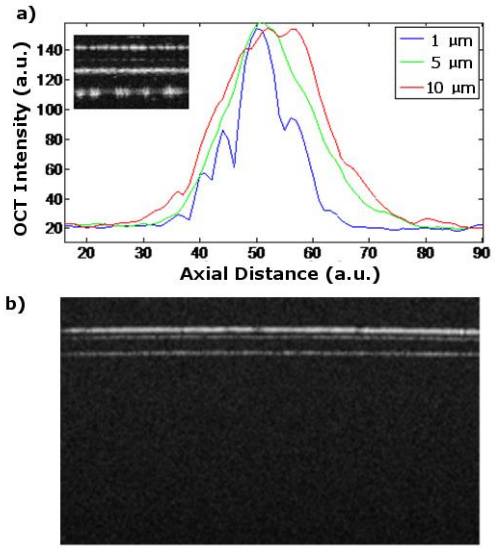

Figure 2. OCT intensity **(a)** measured for three different particle sizes, layered in a PEGDMA matrix. OCT scan imaged with two particle monolayers **(b)**, with the top-most line from the polymer-air interface and the lower two from buried particle layers.

A dual layer optical phantom was fabricated in which the successive monolayers of PS microspheres were measured by OCT (in Figure 2b) to be 17.5 µm and 72.0 µm subsurface. Both of these layers are well defined throughout the longitudinal scan length, and no particles are seen except at the expected locations. For validation for OCT thickness determinations and scattering interparticle distances, surface profilometry based on the principle of white light interferometry was used. Surface profilometry measured the step height between top surface and the first sub-surface PS monolayer at a 16.11 µm step height.

Conclusions

The layered assembly for fabricating an optical phanton as described here enables the reliable determination of OCT axial resolution, which is a critical performance metric with no current instrument-independent measurement method. We have established techniques to assemble multilayered samples consisting of alternating reflective layers of light-scattering microspheres and transparent layers of polyethylene glycol dimethacrylate (PEGDMA)-based polymer. These samples can calculate the separation length scale of the scattering layers for axial resolution measurements, which will be further confirmed by surface profilometry. With the ability to fully quantify the three dimensional structure of systems laden with particle or voids, OCT can provide a novel route to the study of polymeric coatings and biological materials.

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2. Equipment and instruments or materials are identified in the paper in order to adequately specify the experimental details. Such identification does not imply recommendation by NIST, nor does it imply the materials are necessarily the best available for the purpose.
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