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Scattering rings in optically anisotropic porous silicon

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We report the observation of strongly anisotropic scattering of laser light at oblique incidence on a (100)-oriented porous silicon layer. The scattered light forms cones tangent to the incident and reflected beams. The conical pattern is caused by scattering on the vertical walls of pores, which are straight along the layer thickness. The light cone defines structured light rings onto a screen normal to the cone axis. We explain the various structures by optical anisotropy of porous silicon. For the sample under analysis, we directly measure from the ring patterns a value of $\Delta n/n_{\mathrm{ord}}=8\%$ of positive birefringence. © *2002 American Institute of Physics.* [DOI: 10.1063/1.1531834]

Porous silicon (PS) has been actively investigated in the last decade because of the possibilities it offers to produce a new generation of both active and passive optoelectronic devices.[1,2] Since the size of the etched pores is much smaller than the wavelength of visible or infrared light, PS behaves as a homogeneous dielectric layer with an effective refractive index n.

Optical anisotropy is a well-known property of PS.[3–7] This effect is due to the anisotropic geometry of the pores. The small size of the pores allows application of an effective medium approximation. However, if the shape of the pores is very anisotropic, special care must be taken, as n can depend on light polarization. Anisotropy of PS depends on the crystalline orientation of the silicon substrate. Birefringence has been reported in (100)-,[3–5] (111)-,[6] and (110)-oriented PS.[7] In all these cases, the PS layer can be assumed to be uniaxial. The direction of the optical axis to be normal to the surface for the (100) and (111) cases, and parallel to the surface for the (110) case. (100)-oriented PS birefringence is reported to be positive.[3–5] Elastic light scattering or light diffusion in PS has also been studied and reported in previous work.[8,9] Light scattering can be produced either by the roughness of the PS/silicon interfaces, or by the walls of the pores present in PS. In heavily doped PS, the latter mechanism prevails because of the larger size of the pores and better flatness of the PS/Si interfaces. In this work, we report the observation of light-scattering cones in PS, which allows direct measure of birefringence.

We have made PS layers starting from heavily doped p^+-type (resistivity $=0.01\ \Omega\,\mathrm{cm}$) (100)-oriented Si substrates. The electrolyte was obtained by mixing a 30% volumetric fraction of aqueous HF (48 wt %) with ethanol. An anodic current density of 50 mA/cm^2 was applied for 23 min. We also made free-standing samples by applying a short high current (500 mA/cm^2) pulse at the end of the electrochemical attack. The samples were rinsed in ethanol and pentane, and dried in ambient air. Scanning electron microscopy shows a layer depth of 32 μm. Transmission electron micrographs show very straight pore walls along the layer growth direction, as expected in heavily doped p^+-type (100)-oriented PS.[10] Normal reflectance measurements give an ordinary refractive index (n_{ord}) of about 1.4.

The samples were obliquely illuminated with a He–Ne ($\lambda=633$ nm) laser beam, and a white screen was placed parallel to the surface of the sample to image the reflected scattered light. Images were recorded for several angles of incidence for both TE and TM polarizations. The patterns corresponding to 45° of incidence angle are shown in Fig. 1. Both polarizations gave a scattering ring tangent to the incident and reflected beams (zero-order ring). However, for incoming TM-polarized light, a smaller concentric ring (internal ring) was observed, whereas for incoming TE-polarized light, a larger concentric ring (external ring) was observed. The scattering rings were also polarized themselves. The zero-order ring always had the same polarization as the incident beam, whereas the internal or external rings had crossed polarization with respect to it. Thus, a TM incoming light gave a TM zero-order ring and a TE internal ring, and a TE incoming light gave a TE zero-order ring and a TM external ring. In free-standing samples, the scattering rings could be observed in transmission with the same radii and polarization. For a given angle of incidence, the radii of the three rings were independent of the thickness of the PS layer.

The ring patterns can be explained by scattering from the vertical walls of the pores. In fact, when an oblique light beam encounters a dielectric infinite column, the scattered light conserves the angle with respect to the axis of the

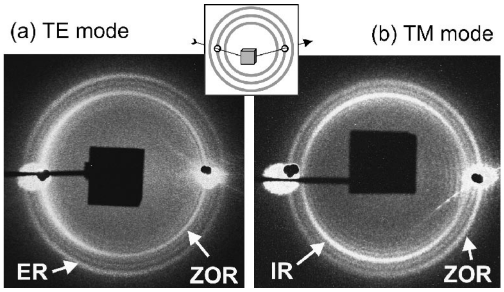

FIG. 1. Photographs of the scattered reflected pattern of a PS sample projected on a screen. The inset in the center sketches the experiment. The dark square is the sample. The angle of incidence is 45°, and the distance between the two holes in the screen is 8 cm. The ring tangent to the holes is the zero-order ring (ZOR) (a) incident TE polarized light with visible ZOR and external ring (ER); (b) incident TM polarized light with visible ZOR and internal ring (IR).

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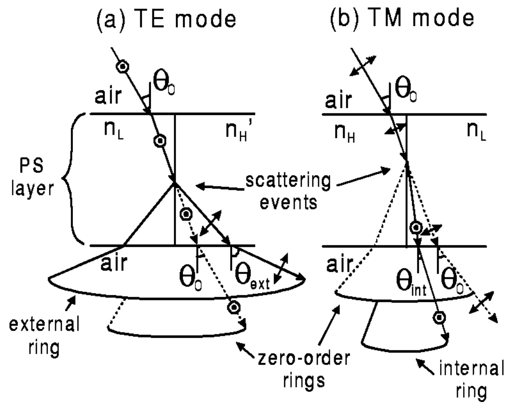

FIG. 2. Model of the formation of the three scattering rings. (a) Incident TE mode; (b) incident TM mode. The polarization of the dashed beams is conserved after the scattering event, so they form the zero-order rings. Only the transmitted rings are sketched for the sake of simplicity. The reflection on the bottom interface allows seeing the same rings in reflection.

column.[11] In a random arrangement of cylinders of different sizes and shapes, many scattering events occur, and scattering directions generate a cone. This cone is tangent to the transmitted light and its axis is parallel to the columns. On a screen normally oriented with respect to the cylinders, the scattered light forms a circumference. This ring can be easily observed with laser light on two-dimensionally structured media both in transmission and in reflection, and can be exploited to measure the orientation of the scattering centers, for example, wood fibers.[12] This allows modeling this type of PS as a network of densely packed air cylinders in a silicon matrix and explains the zero-order scattering ring.

The secondary rings, which to our knowledge have not been previously reported are due to PS optical anisotropy. The scattering process is described in Fig. 2. When a mode (TE or TM) enters the medium, it propagates with its correspondent refractive index (n_{ord} or n_H). When a scattering event occurs, the polarization can be partially lost, especially for the out-of-plane light. The light that conserves the polarization generates the zero-order ring, and the crossed-polarized light undergoes a refraction event because of the change of refractive index. If the change is from a smaller to a large index (TE→TM), an external ring will appear, while an internal ring is observed if the change is from large to small (TM→TE) (see Fig. 2). As the angle of refraction depends only on the refractive indices, this model explains the nondependence of the radii on layer thickness.

To study the problem quantitatively, we have assumed a plane wave incoming with an incident angle θ_0 on an anisotropic medium with indices n_{ord} and n_{ext}. The ordinary mode will propagate with index n_{ord}, and the extraordinary with an index that depends on the angle of propagation, verifying $n_{\mathrm{ord}} < n_H(\theta_0) < n_{\mathrm{ext}}$. Assuming $n_{\mathrm{air}} = 1$ and the Huygens wave-front method,[13]

$$n_H(\theta_0) = n_{\mathrm{ord}} \sqrt{1 + \left(\frac{1}{n_{\mathrm{ord}}^2} - \frac{1}{n_{\mathrm{ext}}^2} \right) \sin^2(\theta_0)}. \tag{1}$$

If we consider light initially to be ordinary (TE) but scattered into extraordinary (TM), the resultant index n'_H will be slightly different from n_H because the angle is different. After some algebra we find:

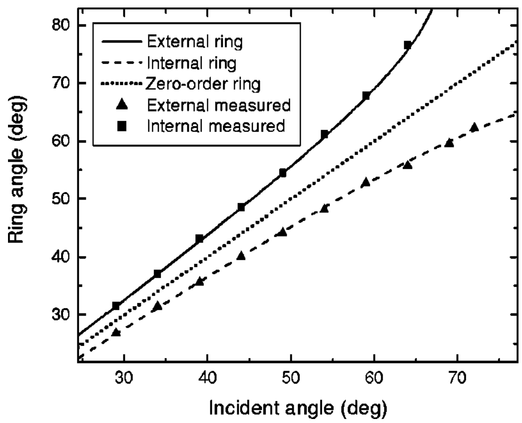

FIG. 3. Experimental and calculated aperture of the rings with respect to incident angle, assuming $n_{\mathrm{ext}}/n_{\mathrm{ord}} = 1.08$. The angles are measured with respect to the normal axis of the surface. Solid line: calculated external ring. Dashed line: calculated internal ring. Dotted line: zero-order ring. Solid squares: measured angle of external ring. Solid triangles: measured angle of internal ring.

$$n'_H(\theta_0) = \sqrt{n_{\mathrm{ord}}^2 + \left(\frac{n_{\mathrm{ext}}^2}{n_{\mathrm{ord}}^2} - 1 \right) \sin^2(\theta_0)}. \tag{2}$$

To obtain θ_{ext}, the angle of the external ring (TE→TM), we use the sequence of indices $\{1, n_{\mathrm{ord}}, n'_H, 1\}$ and to obtain θ_{int}, the angle of the internal ring (TM→TE), we use the sequence $\{1, n_H, n_{\mathrm{ord}}, 1\}$. After algebraic manipulations, we find very simple equations:

$$\sin(\theta_{\mathrm{ext}}) = \frac{n_{\mathrm{ext}}}{n_{\mathrm{ord}}} \sin(\theta_0), \tag{3a}$$

$$\sin(\theta_{\mathrm{int}}) = \frac{n_{\mathrm{ord}}}{n_{\mathrm{ext}}} \sin(\theta_0). \tag{3b}$$

Equation (3) holds both for positive and negative anisotropy (respectively, $n_{\mathrm{ext}} > n_{\mathrm{ord}}$ and $n_{\mathrm{ext}} < n_{\mathrm{ord}}$). The only difference is that the polarization dependence of the external and internal ring would be interchanged. Our case corresponds to positive anisotropy since incident TE gives an external ring and incident TM an internal ring.

Figure 3 reports the angles of the output rings as a function of the incident angles. Both the measured and the calculated data are shown. In the calculation the only free parameter was the ratio $n_{\mathrm{ext}}/n_{\mathrm{ord}}$. For a value of 1.08 (which corresponds to $\beta = 8\%$) we find an accurate fit. Since the absolute value of $n_{\mathrm{ord}} = 1.4$ has been measured independently, we deduce $n_{\mathrm{ext}} = 1.51$. We have also measured positive anisotropy in our samples by interferometric analysis of oblique reflectance spectra. The agreement found between the estimation of β by both methods confirms our explanation of the phenomenon.

We stress that the formation of the three rings is not caused by interference, as evidenced by the independence of the rings' radii from the layer thickness. However, interference effects are also present, and can be observed in Fig. 1 as weaker concentric rings around the main three. The different origin of such weaker rings is evidenced by the fact that they are more separated in samples with a thinner PS layer, while the radii of the main rings do not change when changing the thickness. Therefore, the origin of this weak effect can be attributed to interference between light scattered from both interfaces of the PS film.

In conclusion, we have studied the scattering pattern of PS layers and we have observed three bright rings. The vertical walls of the pores of this type of PS lead to conical scattering events, and to optical anisotropy, which is the cause of the internal and external rings. The position of these rings allows a very simple and direct measurement of the anisotropy parameter of the PS layer. For the sample under study we have found a 8% positive anisotropy.

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