Infrared Physics & Technology 69 (2015) 218–221

Contents lists available at ScienceDirect

Infrared Physics & Technology

journal homepage: www.elsevier.com/locate/infrared

Self-organized, effective medium Black Silicon for infrared antireflection

Martin Steglich [a,*], Thomas Käsebier [a], Frank Schrempel [a], Ernst-Bernhard Kley [a,b], Andreas Tünnermann [a,b]

[a] Institute of Applied Physics, Abbe Center of Photonics, Friedrich Schiller University Jena, 07743 Jena, Germany
[b] Fraunhofer Institute of Applied Optics and Precision Mechanics IOF, 07743 Jena, Germany

HIGHLIGHTS

- Silicon antireflection structures yield transmittances of 97% in the 3–5 μm range.
- Black Silicon antireflection structures are statistical and self-organized.
- ICP-RIE fabrication technique is maskless and applicable to curved substrates.
- Structure optical properties are not affected by temperature variations.
- An effective medium criterion serves as design rule.

ARTICLE INFO

Article history:
Received 16 January 2015
Available online 2 March 2015

Keywords:
Silicon optics
Reactive Ion Etching
Moth eye structures
Effective medium
Black Silicon
Antireflection

ABSTRACT

Statistical Black Silicon antireflection structures for the mid-infrared spectral region, fabricated by Inductively Coupled Plasma Reactive Ion Etching, are investigated. Upon variation of etch duration scaling of the structure morphologies is observed and related to the optical losses in specular transmittance. By means of statistical morphology analysis, an effective medium criterion for the examined structures is derived that can be used as a design rule for maximizing sample transmittance at a given wavelength. To obtain Black Silicon antireflection structures with elevated bandwidth, an additional deep-etch step is proposed and demonstrated.

1. Introduction

Silicon is a typically used material for optical elements like lenses or windows operating in the 3–5 μm range. Due to its high refractive index of about 3.6 in this wavelength range, however, antireflection coatings (ARC) are required to achieve high transmittance through these elements [1]. Another common approach is to use silicon antireflection structures (ARS, also “moth eye” or “surface relief structures”), i.e. tapered surface profiles that implement an effective medium index gradient to suppress interface reflection [2]. Compared to ARCs, ARS are believed to have a higher potential for providing low residual reflectances at very high bandwidths and an improved tolerance to high angles of incidence [3]. Also, they do not show reflectances raised above the substrate line for wavelengths or incidence angles outside the design bandwidth and are potentially less costly than rather thick large bandwidth ARCs (10–100 μm) consisting of a high amount of layers. In addition, conventional interference layer stacks often exhibit instabilities upon thermal cycling like adhesion loss and inherent stress as a result of different thermal expansion coefficients of the incorporated layers and substrate material [4]. Lastly, ARS exhibit higher laser induced damage thresholds than ARCs [5].

Besides the possibility to fabricate periodic ARS by deterministic methods [3–6], in the case of silicon as substrate material also statistical “Black Silicon” ARS obtained by a self-organized Inductively Coupled Plasma Reactive Ion Etching (ICP-RIE) process are of great interest since they offer an excellent cost-value ratio and are potentially easier applicable to non-flat substrates [7,8].

In this paper, such silicon ARS by ICP-RIE are analyzed in terms of morphology and optical transmittance in the near- to far-infrared (NIR-FIR) spectral region. Interdependencies between the fabrication parameters, structure morphology and resulting AR behavior are resolved and explained in detail.

2. Experimental

Black Silicon nanostructures are prepared on both sides of double-side polished, optical grade FZ-Si(1 1 1) via ICP-RIE in a Sentech SI 500 C. Fabrication was performed at a working pressure of 2 Pa. As precursors SF_6 and O_2 were used as etching and passivation

---

[*] Corresponding author.

http://dx.doi.org/10.1016/j.infrared.2015.01.033
1350-4495/

species, respectively. Different structure morphologies were achieved by varying the etching duration. Further information and a method to find suitable ICP-RIE conditions for Black Silicon fabrication can be found in [7–10].

For determination of the achieved antireflection behavior, specular Fourier Transform Infrared (FTIR) transmission spectra of double-side nanostructured samples were measured with a Varian 3100 spectrometer. It should be noted that, due to the finite detector aperture of the spectrometer, also light scattered into very low angles up to about 1° might be recorded. In order to relate the optical to the morphological properties of the nanostructures, both cross-section and large area top-view scanning electron microscopy (SEM) images were analyzed statistically (see Fig. 1). While mean structure depths were retrieved directly from cross-section images, for evaluation of lateral structure geometry a *radial autocorrelation function ACV_r* was determined from top-view images:

$$ACV_r\{f\}(r) = \frac{1}{2\pi}\int_0^{2\pi}\iint f(x,y)f(x + r\cdot\cos\phi, y + r\cdot\sin\phi)\mathrm{d}x\,\mathrm{d}y\,\mathrm{d}\phi$$

here, f is the grey-tone valued SEM image, normalized to have a zero mean value, and (r, φ) are the polar coordinates. Since Black Silicon, as can be clearly seen from the top-view image in Fig. 1, is composed of statistically arranged and partially connected circular etch pores, the ACV_r curves can be utilized to obtain a characteristic value of the lateral structure dimensions: Each structure's curve exhibits a characteristic minimum at a radius r that corresponds to the mean pore diameter. In the following, we will refer to this value of r as *lateral autocorrelation length* L_{corr} in order to quantify the lateral structure geometry.

3. Results and discussion

Applying Black Silicon nanostructures to silicon optics for antireflection purposes, one has to keep in mind that silicon optical elements are typically required to be image preserving. This means that forward scattering of the incident light into higher angles of propagation is undesirable. Thus, ARS should also behave as an effective medium for incident light at a given wavelength λ in order to inhibit diffraction into orders different from the 0th. In this spirit, regarding statistically distributed ARS, scattering is suppressed if the mean lateral distances of the single structure features (here reflected by the autocorrelation length L_{corr}) is smaller than a constant multiple of λ:

$$L_{corr} < const\cdot\lambda \tag{1}$$

For a specific structure this implies that the AR behavior for short wavelengths is restricted by a too high L_{corr} which leads to severe scattering losses.

Moreover, the structure's AR effect is also limited for long wavelengths. Here, the ARS act as effective medium, but the finite structure depth (i.e. the "thickness" of the effective medium gradient layer) yields a monotonically rising reflectance with increasing wavelength [2].

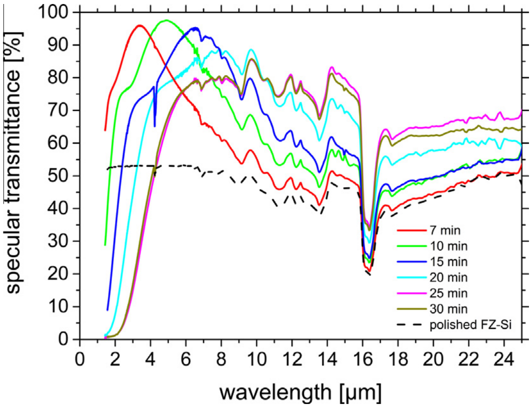

Fig. 2. Specular transmittance spectra of silicon substrates with identical Black Silicon ARS on both substrate sides. Different ARS were fabricated by simply varying the etch duration. For comparison, also the spectra of polished FZ-Si wafers with and without standard AR coating are displayed.

Both limits can be easily reenacted with the help of Fig. 2 which displays the measured FTIR (specular) transmission spectra of different, both side identically structured samples.

As it can be seen clearly, each sample with Black Silicon ARS exhibits a distinct maximum at a certain wavelength, with decreasing specular transmittance at lower wavelengths (scattering losses) and higher wavelengths (finite structure depth). The characteristic dips in transmittance, starting at wavelengths around 6 µm, are due to the lattice absorption of silicon [11]. As a matter of fact, before the onset of lattice absorption, the maxima of the shorter etched samples reach values close to the ideal value of 100%. This implies that at these maximum wavelengths, the ARS are at least *nearly* acting as effective media with sufficiently large structure depths, since otherwise the specular transmittance would be much lower due to scattering or too low structure depth.

Furthermore it can be deduced from Fig. 2 that the transmittance maxima shift monotonically to higher wavelengths with increasing etch duration. Following Eq. (1), the reason for this behavior is the continuously rising structure autocorrelation length L_{corr} with increasing etch duration. Fig. 3(a) displays this evolution of structure morphology parameters L_{corr} and depth over etch duration, as retrieved from statistical analysis of SEM images.

Obviously both the mean structure depth and L_{corr} grows monotonically with etch duration, with an approximate slope of 85 nm/min and 12.5 nm/min, respectively. As the formation of Black Silicon results from the development of interconnected, circular etch pores that become deeper and wider during continued etching (see Fig. 1), this is not surprising [7]. However, plotting each structure's depth over its lateral autocorrelation length, it can be realized that the structures' aspect ratios (i.e. depth/L_{corr}) remain nearly perfectly constant during the etching procedure, which is indeed astonishing (see Fig. 3(b)). From the physical point of view, this can be attributed to the constant selectivity of the

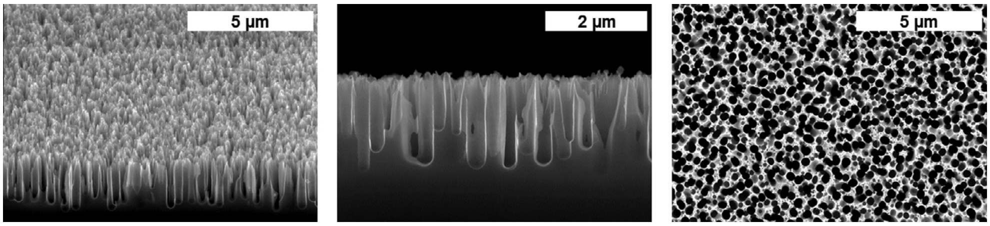

Fig. 1. Typical SEM micrographs of Black Silicon structures fabricated by ICP-RIE. An illustrative picture under a viewing angle of 30°, a cross-section and a top-view image is displayed.