Design and characterization of optical coatings with enhanced roughness for ultra-hydrophobic, low scatter applications

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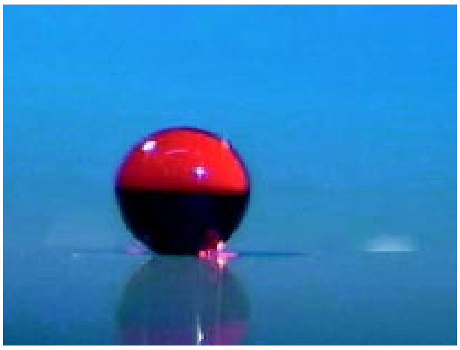

Fig. 1:
Water drop on an ultra-hydrophobic surface, contact angle = 174°.

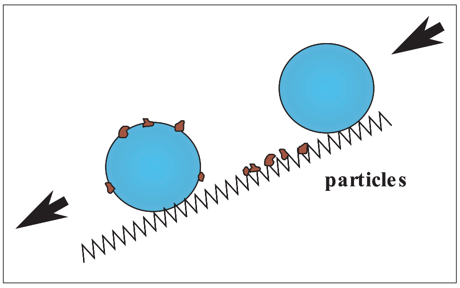

Fig. 2:
"Self-cleaning" by rolling water drops removing surface contaminations.

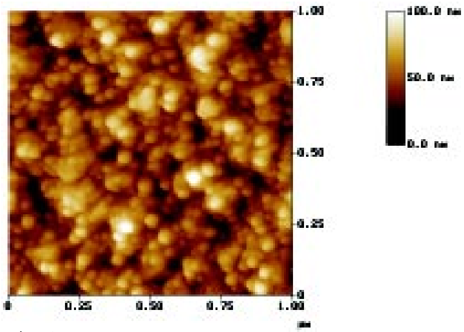

Fig. 3:
AFM image of the surface structure of the rough ZrO_2 layer deposited by e-beam evaporation.

Introduction

Studies into the wettability properties of solid surfaces are attracting drastically growing interest due to numerous new perspectives for practical applications. In particular, intensive research focuses on the development of hydrophobic surfaces. It has become well known that hydrophobicity depends on both the intrinsic material properties and surface morphology /1/.
Joint activities of SuNyx Surface Nanotechnologies GmbH and IOF are directed to a novel approach to create ultra-hydrophobic surfaces with the following properties:

- static contact angle considerably higher than 120° (Fig. 1), roll-off angle considerably lower than 10° (i.e. practically no adherence of water drops),
- real "self-cleaning", i.e., rolling water drops remove the surface contaminations (Fig. 2),
- surface morphology realized through enhanced statistical nanoroughness
- optically transparent with controlled scatter losses.

Usually, roughness in dielectric thin films constitute an undesirable property because it can result in scatter losses limiting the performance of the component. However, if enhanced roughness can be induced deliberately according to a quantified relation to contact angle as well as to an adjustable threshold for the resulting light scattering, then thin film roughness can be advantageously utilized to achieve the surface structure needed for ultra-hydrophobicity.

The coating technology is developed at SuNyx.
The contribution of the Fraunhofer IOF to this development focuses on the sample design and characterization.

Methodology

We have found that through wide-scale roughness analysis and subsequent data reduction the roughness characteristics can be directly related to the contact angle. As, on the other hand, vector scattering theories connect the roughness properties with scatter losses, a formalism has been accomplished, where both the wetting properties and scattering behavior can be expressed within the same "language" /2/.

The first step in this approach is the surface roughness determination of the thin film coating under study over a wide range of roughness spatial frequencies f, from 10^{-3} through 10^{3} µm^{-1}, which can be measured by combining white light interferometry, scanning force microscopy, and scanning tunneling microscopy /3/.
This is followed by calculating the Power Spectral Density PSD(f) for the whole frequency range and subsequent data reduction:

PSD(f) → amplitude spectrum A(f) → reduced amplitude spectrum b(f) = A•f → logarithmically averaged reduced amplitude I(β).

Our experimental investigations revealed that I(β) can be empirically related directly to the contact angle and, hence, to the wetting behavior. Light scattering is predicted by using the measured PSD together with the optical parameters of the film, the wavelength of consideration etc. as an input into our multilayer vector scattering program /4/. The resulting scatter is compared with a threshold which in turn depends on the particular application. For architectural glass coatings, for instance, this threshold will be determined by a visual perception threshold of scatter effects in the visible spectrum.
Together with the parameter I(β)

then two essential criteria are provided for a coating to become a potential candidate for low scatter ultra-hydrophobic application.

Experimental results

First experiments have been made with zirconium oxide films as model layers. Single layers were deposited onto BK7 substrates by e-beam evaporation with varied deposition conditions and thicknesses to achieve different roughness properties. Total light scattering (TS), defined in detail in /3/, was measured on the samples as deposited, and contact angle was determined after overcoating the samples with a thin sputtered gold film and a monolayer of n-decanthiol to deliver the necessary intrinsic hydrophobicity. Note that this is only for contact angle measurement, in transparent sample applications other overcoatings such as thin perfluorinated films will be used instead. The AFM image in Fig. 3 qualitatively reveals the enhanced roughness of a ZrO_2 layer evaporated at 590 K. The PSD curve for this coating is depicted in Fig. 4. The corresponding I(β) was > 0.3 and hence, a high contact angle could be predicted. Experimentally, a promising contact angle as high as 143° was obtained. Nevertheless, the light scatter losses remained reasonably low, as can be seen in Fig. 5 showing the results of forward and backward TS measurements at 633 nm.
For comparison, the PSD of another ZrO_2 layer deposited at ambient temperature has been also included in Fig. 4. The roughness characteristic of this coating was noticeably different, the calculated I(β) was considerably lower and so was the measured contact angle.

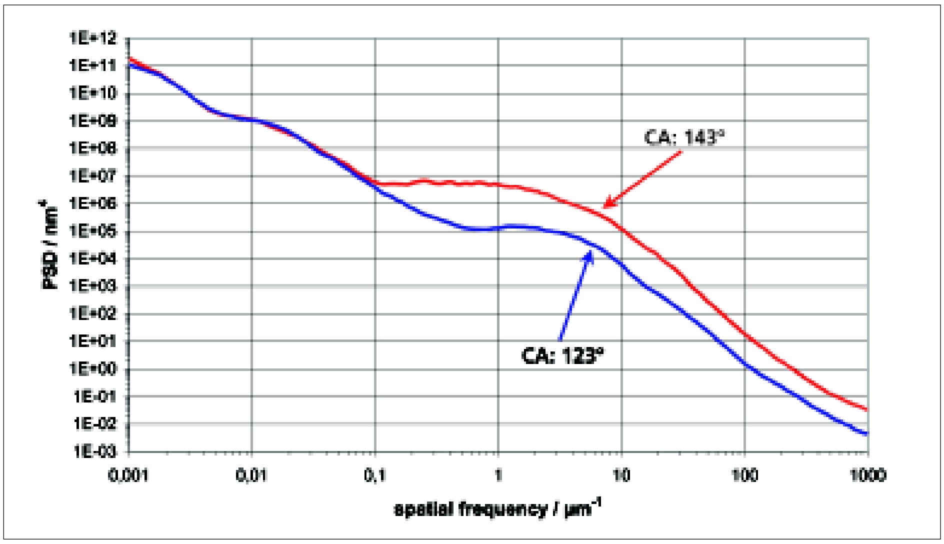

Fig. 4:
PSD curves and contact angles (CA) for ZrO_2 layers deposited by e-beam evaporation at different substrate temperatures, overcoated with a thin sputtered gold film and a monolayer of n-decanethiol.

References

/1/ E. Wolfram, R. Faust: Wetting, Spreading and Adhesion (J. F. Padday, Ed.; Academic Press: London, 1978), p. 213.
/2/ K. Reihs, A. Duparré, G. Notni: "Substrat mit gering lichtstreuender Oberfläche", patent pending.
/3/ A. Duparré, G. Notni: "Multi-type surface and thin film characterization using light scattering, scanning force microscopy and white light interferometry." in: Al-Jumaily, G.A.: Optical metrology. SPIE Critical Revue Series. vol. CR 72, Bellingham/Wash.: SPIE 1999, pp. 213–231.
/4/ J. Ferré-Borrull, A. Duparré, E. Quesnel: "Roughness and light scattering of ion-beam-sputtered fluoride coatings for 193 nm", Appl. Optics 39 (2000), pp. 5854–5864.

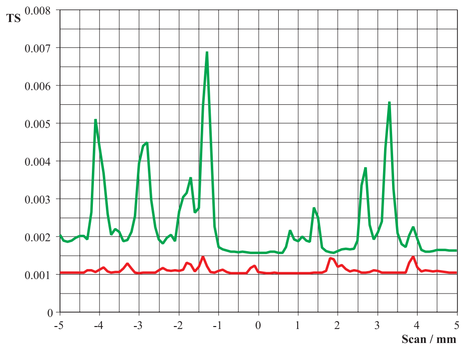

Fig. 5:
Total forward scattering (green curve) and backscattering (red curve) of the ZrO_2 layer which delivers a CA of 143°. Scatter measurements were performed at 633 nm.