

# Projektplan

## ULTRAHARD: Ultrahard optical diamond coatings (CORNET Projekt 263 EN)

## Forschungsziel

Transparente Beschichtungen zum Kratzschutz bestimmen oft die Einsetzbarkeit und die Lebensdauer optischer Bauteile. Sie werden in vielen Branchen mit Antireflex- (AR-) oder hochreflektierenden (HR-) Schichten kombiniert. Heutige Lösungen sind jedoch stark anfällig für Verschleiß, optische Eintrübungen durch Abrieb oder haben eine niedrige laserinduzierte Zerstörschwelle. Projektziel ist die Entwicklung ultraresistenter AR- und HR-Oberflächen mithilfe transparenter nanokristalliner Diamantschichten. Dazu sollen Substratreinigung und -bekeimung angepasst und mehrere Beschichtungsverfahren untersucht und optimiert werden.

work package	timescale																							
project month	01	02	03	04	05	06	07	08	09	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24
WP 1: Selection of addressed applica- tions and target specifications of the films stacks																								
WP 2: Preparation of coating technology																								
WP 3: Substrate preparation and seeding investigations																								
WP 4: Development and deposition of nanocrystalline CVD diamond layers in the AR stack																								
WP 5: Characteriza- tion and analysis of the deposited layers and layer stacks																								
WP 6: Deposition and analysis of the demonstrators																								
WP 7: Estimate of Economy																								
Milestones					MS1	٠					MS2	٠					MS3	٠					MS4	٠

#### Working plan

MS 1 = An optimal pre-treatment process for the coating of nanocrystalline diamond layers for each substrate is available

MS 2 = A demonstrator for a nanocrystalline diamond layer on quartz (10 cm x 10 cm) is build up MS 3 = A demonstrator for a nanocrystalline diamond layer on a substrate (10 cm x 10 cm) is available MS 4 = Demonstrators for ultrahard antireflection layers are built up which contain at least one diamond layer

## Approach

The HW-CVD technique is the only industrialized technique with efficient diamond coating processes on large areas (0.5 m<sup>2</sup>), for example to coat face seals and diamond electrodes. However, growth rates and purity are in general inferior to plasma-enhanced diamond CVD. The MW-C-PECVD with cavity geometry technique shows significantly higher deposition rate for diamond on small areas up to 6", but it is difficult to upscale the process for large area diamond coatings, however this can be done by the MW-LA-PECVD. Additionally, MW-LA-PECVD allows continues wave and pulsed mode depositions, which could be key for low temperature growth of high quality layers. Therefore, the deposition methods can be compared to develop the optimum in terms of quality and economics.

Diamond deposition will be carried out using large area HW-CVD at Fraunhofer IST, MW-C-PECVD at UHasselt and large area MW-LA-PECVD at IoP and UHasselt. Due to the plasma conditions created in the MW-LA-PECVD apparatus, the substrate temperature is disassociated from the plasma, and therefore can be carefully controlled, enabling coating of temperature sensitive glass. In addition, due to the modular design of the MW-LA-PECVD system, more plasma lines can be added, therefore it can be envisioned that substrates with huge lengths (jumbo glass size) could be accommodated for diamond coating.

Since diamond can only be grown onto a diamond base, the technologies require research into the necessary development of seed-layers for large areas. This is done with the help of purchased diamond suspensions. The difference in suspensions can lead to different results during growth (homogeneous growth, sufficient adhesion, etc.). Different refractive index layers can be compared by MW-C-PECVD, MW-LA-PECVD and HW-CVD in terms of performance in the layer stack.

The proposed solution is the use of nanocrystalline diamond for scratch resistance and the low refractive index materials SiO<sub>2</sub> as thin optical layers for broadband antireflection.

The use of diamond coatings in AR and HR coating systems produces some of the greatest challenges in modern coating technology. Specifically for this project, layer uniformity requirements must be satisfied, which by far exceed the specifications for other technological fields in which diamond films are successfully used. An additional challenge is to ensure that the topmost layers with high hardness of the AR stack (deposited above the top diamond layer) must be compatible with the temperatures and materials used in the industrial user committee.

The work program (Figure 1) covers the following research tasks according to the mentioned objectives:

 Pre-treatment, HW-CVD, MW-LA-PECVD and MW-C-PECVD processes are to be designed in such a way that the most uniform and low absorption diamond layer is grown, and stable interfaces are formed between the diamond layer and adjacent layers, or substrate.

- Diamond layers are polycrystalline in nature, therefore in order to avoid optical scattering, the crystallite grain size must be well below the wavelength of visible light (< 400 nm). For this reason, the production of the diamond layers must be nearly defect-free and create very small crystallite sizes (nano-crystalline). These layers have a higher grain-boundary density compared to coarsely crystalline layers. This slightly reduces the hardness of the diamond layers, and can lead to absorption losses. In order to produce such layers, a pretreatment step adapted to the previous coating, which produces a layer with exceptionally high nuclei densities (> 10<sup>11</sup> cm<sup>-2</sup>), will be investigated.
- All layers deposited before the diamond layer will be developed in a way that they are stable against the effects of atomic hydrogen. H radicals can chemically reduce the previous oxide layers, which could lead to sub-stoichiometric boundary layers with altered optical properties. This could be overcome with nanoscale (< 3 nm) transparent protective interlayers if there are reactions with the underlying layer prior to the diamond layer.
- The MW-LA-PECVD system that will be used provides a low temperature diamond growth compared to HW-CVD, thus reducing the negative effect on the stability of previously deposited layers in the AR system.
- Layer designs with determined refractive indexes are created as required, and the number of layers may not exceed 12 for economic reasons. In addition to diamond and SiO<sub>2</sub>, other materials which have a higher hardness as SiO<sub>2</sub> (e.g. Al<sub>2</sub>O<sub>3</sub>) within the layer stack (next to the substrate) will be considered in order to fulfill requirements. In addition, material available from the industrial user committee will be used.
- The layer stack is a composite (substrate, low, medium and high reflective materials and diamond seeds) such that the complete stack will be examined. Since the deposition of the diamond layer is the most costly process, as few diamond layers as possible – ideally one – should be used.
- In order to test the mechanical stability beside of the established practice of nanoindentation for the determination of the hardness, practical tests such as a Taber-Abraser, Sand-trickle and Bayer test, and micro-scratch tests will be used. In addition, further investigations like autoclavability will be carried out.

As a result of the project two demonstrator (broadband AR and HR mirror) will be produced.



Figure 1: Work packages (WP) and interactions in the project ULTRAHARD

## **Projektbegleitender Ausschuss**

Unternehmen
ASKANIA Mikroskop Technik Rathenow GmbH <sup>KMU</sup>
Berliner Glas KGaA Herbert Kubatz GmbH & Co.
Blösch AG <sup>KMU</sup>
CREAVAC Creative Vakuumbeschichtung GmbH <sup>KMU</sup>
GD Optical Competence GmbH <sup>KMU</sup>
Plasus GmbH <sup>KMU</sup>
PrinzOptics GmbH <sup>KMU</sup>
Qioptiq Photonics GmbH & Co. KG
W&L Coating Systems GmbH