DIAMOND FILMS FOR EXTREME APPLICATIONS
The deposition of polycrystalline diamond films by hot-filament chemical vapor deposition (HFCVD) is one of the core competencies of the Fraunhofer Institute for Surface Engineering and Thin Films IST. With the fully-automated HFCVD installations developed at the institute, areas measuring up to half a square meter can be coated. Also available are processes and installations for diamond coating of three-dimensional objects and for internal coatings of tools and parts. The diamond films are available in different modifications for various applications, such as with a rough or smooth surface or as an electrically conductive coating, respectively. We offer our customers and partners the following services:

**Development of substrate or base body**
- Design and material selection according to coating requirements
- Support for procurement of base bodies

**Film development**
- Development of new, application-oriented coating systems
- Combination with additional PVD or CVD thin films as interlayer or toplayer

**Process development**
- Adaption to base body geometry (3D as well as inside geometry) and up-scaling
- Economical optimization by increasing batch volume and/or high-rate processes
- Prototypes and small batches for application tests
- Simulation for process optimization (e.g. film thickness homogeneity, substrate position, reactor concept)

**Analytics and quality assurance**
- Measurement of chemical and physical film properties
- Spatially-resolved measurement of film thickness distribution and coating adhesion
- Application-specific characterization methods, also in cooperation with partners

**Technology transfer**
- Development of customized coating solutions
- Process transfer to clients or to coating enterprises
- Design of hot-filament CVD components (e.g. filament holders, substrate cooling)
- Support for design, procurement and implementation of hot-filament CVD reactors
- Support for implementation of processes for substrate pretreatment, e.g. finishing, cleaning, seeding
- Literature, patent and market research, studies, trainings

Within the context of publicly funded projects and industrial projects, film properties and coating processes and installations are adjusted to meet the requirements of the particular application.

1 CVDiamond XXL hot-filament chemical vapor deposition installation for manufacturing diamond electrodes.
FILMS, PROCESSES AND INSTALLATIONS

The Diamond Films

<table>
<thead>
<tr>
<th>Coating</th>
<th>Characteristics</th>
</tr>
</thead>
<tbody>
<tr>
<td>DiaCer®</td>
<td>Diamond-coated ceramics, high wear-resistance and corrosion protection, low friction coefficient</td>
</tr>
<tr>
<td>compeDia®</td>
<td>Microcrystalline with sharp-edged surface roughnesses from 0.2 to 20 µm for precision grinding</td>
</tr>
<tr>
<td>DiaTex®</td>
<td>Microcrystalline with preferential crystal orientation for machining applications and components</td>
</tr>
<tr>
<td>Boron-doped</td>
<td>Conductive diamond films for electrochemical processes</td>
</tr>
<tr>
<td>Nanocrystalline</td>
<td>Crystal size &lt;0.5 µm</td>
</tr>
<tr>
<td>Microcrystalline</td>
<td>Crystal size ≥ 0.5 µm</td>
</tr>
</tbody>
</table>

The Coating Properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hardness</td>
<td>80 up to 100 GPa</td>
</tr>
<tr>
<td>Modulus of elasticity</td>
<td>800 up to 1080 GPa</td>
</tr>
<tr>
<td>Density</td>
<td>3.52 g/cm³</td>
</tr>
<tr>
<td>Thermal stability</td>
<td>up to 600 °C in presence of oxygen, up to 1200 °C without oxygen</td>
</tr>
<tr>
<td>Thermal conductivity</td>
<td>up to 2000 W/(m K) (at 20 °C)</td>
</tr>
<tr>
<td>Thermal expansion</td>
<td>0.8x10⁴ K⁻¹ (at 20 °C)</td>
</tr>
<tr>
<td>Specific resistance</td>
<td>doped: 10⁻⁵...10 Ωm, undoped: 10⁻¹⁰...10⁻¹¹ Ωm</td>
</tr>
<tr>
<td>Disruptive strength</td>
<td>2.5 MV/cm</td>
</tr>
<tr>
<td>Coefficient of friction</td>
<td>≤ 0.1 (without lubricant)</td>
</tr>
<tr>
<td>Transparency</td>
<td>accessible in IR, in visible range and in near UV</td>
</tr>
</tbody>
</table>

The Coating Process

<table>
<thead>
<tr>
<th>Substrate materials</th>
<th>hard metals, SiC and SiN ceramics, refractory metals (such as Nb, W, Ti), carbon materials (graphite, glassy carbon)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Deposition temperatures</td>
<td>600...950 °C</td>
</tr>
<tr>
<td>Process pressure</td>
<td>5...100 hPa</td>
</tr>
<tr>
<td>Layer growth rates</td>
<td>0.15...0.5 µm/h</td>
</tr>
<tr>
<td>Coating thicknesses</td>
<td>50 nm...100 µm</td>
</tr>
<tr>
<td>Coatable areas</td>
<td>up to 500 mm x 1000 mm</td>
</tr>
<tr>
<td>Internal coating</td>
<td>up to l/d =10, d_min = 1 mm</td>
</tr>
<tr>
<td>Gases</td>
<td>methane, hydrogen, various boron dopant gases for setting electrical conductivity, precursor gases for intermediate-layer deposition: including TMS, silane, ammonia</td>
</tr>
</tbody>
</table>

The Coating Installations

<table>
<thead>
<tr>
<th>Installation</th>
<th>Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>CVDiamond M</td>
<td>Volume 85 l, coatable area approx. 500 cm², max. 5 kW</td>
</tr>
<tr>
<td>CVDiamond L</td>
<td>Volume 100 l, coatable area approx. 700 cm², max. 30 kW</td>
</tr>
<tr>
<td>CVDiamond XL</td>
<td>Volume 400 l, coatable area approx. 1800 cm², max. 60 kW</td>
</tr>
<tr>
<td>CVDiamond XXL</td>
<td>Volume 1000 l, coatable area approx. 0.5 m², max. 100 kW</td>
</tr>
</tbody>
</table>

2 Microcrystalline diamond film.
3 Nanocrystalline diamond film.
As the hardest material of all, diamond is an ideal cutting material for a wide range of machining operations. For our customers we develop CVD diamond tools which combine the highest wear resistance with great precision and a wide range of different shapes.

**Cutting tools**
For milling, turning and drilling tools made of hard metal we are developing among other things intermediate layers as cobalt diffusion barriers which can make etching pretreatment unnecessary and make it possible to coat hard metals with high cobalt contents. We are researching into intermediate layers which can be deposited in situ – in other words, during the same coating operation as diamond coating.

Other research topics in this field include:

- Diamond coating of ceramic cutting tools (DiaCer®)
- Micro-designing of cutting edges and cutting faces (DiaTex®, compeDia®)
- Additional top coatings for improved anti-adhesion effect (DiaTex® Plus)

**Grinding tools**
The Fraunhofer IST is developing the compeDIA® abrasive coating as an innovative solution for precision grinding operations. Ductile grinding, the highest surface qualities and low levels of damage in the subsurface layer become possible with the very finest grain sizes ranging down into the submicrometer range. With a 100 % diamond content, grinding ratios up to G = 30 000 are achieved as well as extremely long tool life. Cost-effective resharpening by recoating is also possible. Micro-abrasive pencils with extremely small diameters of down to 50 µm can be produced just like grinding wheels with diameters of down to 300 mm.

**Forming tools**
As an alternative to wire drawing dies with solid diamond inserts we are developing DiaCer® drawing dies made of ceramic with an internal coating of CVD diamond. Diamond drawing dies with complex cross-sectional profiles and internal diameters from 1 mm to more than 50 mm can be made in this way.

**The advantages**
- Extremely long service lives
- Low coefficient of friction
- Good anti-adhesion properties
- Complex tool geometries
- Internal coatings
- Small tool diameters down to 50 µm
- Large tool dimensions up to 1000 mm
- Adjustable surface roughness
- High geometrical flexibility for the most challenging applications
MACHINE COMPONENTS

CVD diamond coatings are being developed for machine components with a view to improving tribological behavior and being able to select specific surface properties – even in combinations of these properties. On the basis of the unsurpassed properties of CVD diamond films, machine components can be equipped with entirely new functionalities:

- Diamond films combine maximal wear resistance with minimal coefficients of friction even when lubrication is insufficient and in fact even with dry running.
- Diamond has an unsurpassed resistance to abrasion and even at high temperatures can resist a large number of chemical substances and radicals.
- Due to a thermal conductivity not reached by any other material, diamond offers the greatest possible protection against thermal overload during extreme local stress (mechanical, thermal or electromagnetic).
- The electrical conductivity of CVD diamond films can be specifically adjusted over many orders of magnitude (from the insulator to metal-like conductivities).

Examples of application of CVD diamond components
The most successful example to date of an application for diamond-coated components is diamond-coated ceramic face seals. On the basis of process and installation technology developed at the Fraunhofer IST a breakthrough innovation in the field of sealing technology has been achieved in collaboration with the Fraunhofer institutes IKTS, IPK and IWM and partners from industry. With hitherto unknown service lifetimes it is able to satisfy entirely new customer requirements and the most difficult application cases. Further information about diamond-coated face seals may be found at www.diamondfaces.com. Crucial to the success of our work was the process-reliable production of uniform diamond films of defined degrees of surface roughness. The existing technology is suitable for coating

- Face seals
- Sliding-contact bearings for linear and rotational movements (axial and radial bearings),
- Sealing and guidance elements,
- Components subjected to extreme tribological, abrasive or corrosive stress.

The substrate materials preferred for CVD diamond coatings are silicon-based ceramics such as SiC or Si₃N₄ and WC-Co hard metals.

The advantages
- Maximum wear resistance, for example in contact with abrasive substances
- Low coefficients of friction even with insufficient lubrication or dry running
- Resistance to most chemical substances
- Durability at high temperatures
- Adjustable electrical conductivity, for example, in order to prevent electrocorrosion
A new material for electrochemical applications
By coating industrial substrates with conductive diamond films a new electrode material with outstanding properties has been developed at the Fraunhofer IST: the diamond electrode. It is produced and marketed as DiaChem® electrode* by CONDIAS GmbH, a spin-off company of Fraunhofer IST.

Properties
Diamond electrodes are extremely stable chemically and have the largest known electrochemical window for the electrolysis of water. Oxidizing agents such as ozone, hydrogen peroxide and even hydroxyl radicals are formed efficiently at diamond anodes. Their oxidizing power can be utilized for highly efficient electrochemical processes for treating water without the use of additional chemicals. Furthermore diamond electrodes can be used for innovative or more efficient electrochemical synthesis processes.

Applications
As a member of the Fraunhofer Water Systems Alliance “SysWasser” the Fraunhofer IST is involved in working out sustainable system solutions for water supply, water infrastructures and wastewater treatment. Electrochemical systems with diamond electrodes are, for example, used for:

- Treatment of ballast water in ships
- Disinfection of ultra-clean process waters
- Chemical-free treatment of water in foodstuffs production
- Sanitization of pure and ultrapure water plants
- Removal of micropollutants in surface and groundwater
- Removal of persistent substances from production water, e.g. for process water recycling or for discharge without health risk
- Efficient synthesis of, e.g. persulfate

The advantages
- Maximum current efficiency for the generation of OH radicals in water on account of extremely high overvoltages (cathodic -1.2 V vs SHE and anodic +2.6 V vs SHE)
- High electrochemical stability, suitable for high current densities up to several A/cm²
- Extreme chemical resistance (no corrosion in aggressive media, such as, for example, HF)
- Oxidation and synthesis processes with simple electrical control without the addition of chemicals

*DiaChem® is a trademark of CONDIAS GmbH.

DIAMOND ELECTRODES
Technology transfer
An important objective of the Diamond Technology department is the transfer of hot-filament CVD technology into industrial processes for the manufacture of products for extreme applications. Advantages are:

- Simplified gas phase activation as compared with other low-pressure processes
- Scalability up to large areas and for complex base bodies
- Opening up an expanded utilization of the extreme property combinations offered by diamond

Best practice – DiaChem®
The process and plant technology for the hot-filament CVD deposition of electrically conductive diamond films for the world’s largest diamond electrodes has been transferred to the spin-off company CONDIAS. Since 2001 CONDIAS GmbH has been marketing DiaChem® diamond electrodes and together with the Fraunhofer IST and partners from industry developing system solutions for electrochemical water treatment and synthesis.

Best practice – DiaCer®
The HFCVD process technology for manufacturing diamond-coated ceramic face seals which was developed in the Fraunhofer alliance DiaCer® together with partners from industry since 2007 has been used by EagleBurgmann Germany and CONDIAS for producing DiamondFaces® face seals which are marketed worldwide. For the development of the composite material DiaCer® and for work on diamond-coated face seals the German Stifterverband granted its Award for Science for Joint Projects in Applied Research while the Braunschweig Chamber of Commerce and Industry awarded the 2009 Technology Transfer Prize. In addition, the Diamond-Faces® received the Best Practice Award of Frost & Sullivan, the US management consultants, and also the 2008 Innovation Award of the US magazine Flow Control which recognizes outstanding achievements in fluid-handling technology.

Profit from our years of experience with collaborative relationships with:

- Substrate material and base body producers
- Plant and equipment manufacturers
- Measuring instrument manufacturers and testing laboratories
- Institutes for conducting testing and application trials of coated components
- Contract coating services

<table>
<thead>
<tr>
<th>Industrial processes</th>
<th>Process-reliable coating of large parts and large batch sizes</th>
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<tbody>
<tr>
<td></td>
<td>Uniform film properties</td>
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<tr>
<td></td>
<td>Quality assurance procedures</td>
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<tr>
<td>Plant engineering</td>
<td>Large-area diamond coatings</td>
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<tr>
<td></td>
<td>PLC-controlled coating systems with fully-automated operational processes</td>
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<tr>
<td></td>
<td>Application-specific installation designs and components</td>
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<tr>
<td>New material systems</td>
<td>Conservation of resources and productivity boosting due to longer service life of materials</td>
</tr>
<tr>
<td></td>
<td>Improved or new products, such as, DiaCer®</td>
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</tbody>
</table>
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