



## ATMOSPHERIC PLASMA DEPOSITION OF CRYSTALLINE INORGANIC COATINGS

### KEYWORDS

- Atmospheric Plasma Technology
- Dielectric Barrier Discharge
- Crystalline inorganic coatings

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### THE TECHNOLOGY IN A NUTSHELL

The invention of an **induction heating system adapted to a reactor of the 'dielectric barrier discharge' type known as DBD**, opened up the field of **crystalline deposits of inorganic material** hitherto limited to deposition under vacuum or at very high temperature.

### STATE OF THE ART

Atmospheric plasmas have been used for a long time for ozone synthesis and for surface activation. Their main advantage is their ability to be run at atmospheric pressure, avoiding therefore the need of vacuum systems (chambers, pumps, transfer locks...).

When it comes to crystalline inorganic coatings, for years, the main option was to perform annealing on the quasi-amorphous deposited films.

### THE INVENTION

The invention aims at providing a **new and improved DBD plasma reactor for performing PECVD of a crystalline inorganic coating on a substrate**.

Thanks to an original coupling of a substrate heating device based on an inductive current loop and located under the electrode and an atmospheric pressure dielectric barrier discharge, we show that one can deposit in one step **crystalline vanadium oxide** and **titanium oxide**, with grain sizes bigger than those achieved by post-deposition annealing in less time.

This original setup avoids electrical interferences with the high frequency plasma circuit, and allows fast heating of the substrate, located on top of a dielectric.

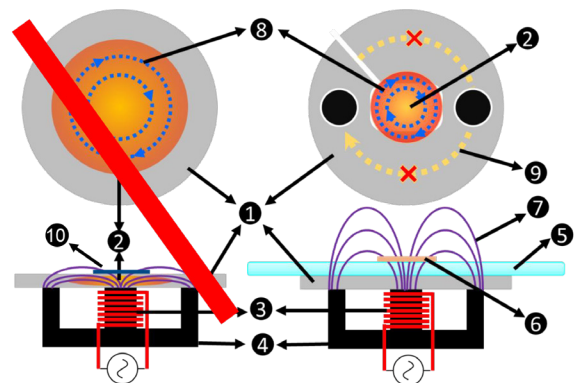


Fig. 1: configuration with non modified electrode (left) and with hollow electrode (right).  
1) Electrode; 2) Heated zone; 3) inductioncoil; 4) ferrite core; 5) Dielectric; 6) Susceptor; 7) Magnetic field lines; 8) induced Eddy current (arbitrary direction); 9) impossible current line; 10) Substrate.

### KEY ADVANTAGES OF THE TECHNOLOGY

- Environmental friendly
- Allows to deposit coatings with unique properties
- Better control of the heating
- Avoids costly post-treatment annealing
- Energy efficient

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## TECHNOLOGY READINESS LEVEL **1** **2** **3** **4** **5** **6** **7** **8** **9**

Two prototypes of this new deposition system have already been produced. Actual developments **focus on heating the system to temperatures of at least 1000 ° C**, in order to cover the crystallization temperatures of most of the deposits produced for the microelectronics industry.

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### POTENTIAL APPLICATIONS

These crystalline inorganic deposits are of paramount importance for the field of **semiconductors and therefore of current microelectronics**. These deposits are central to the operation of a large number of microelectronic systems and **the invention contributes to reduce the production costs** of these components, since it allows crystalline deposits to be brought into the field of deposits by atmospheric plasma and more precisely those produced by DBD.

Vanadium oxides can be used for infrared sensors, as catalysts, as thermo chromic or electro chromic devices and as cathode for Li-ion batteries. Titanium oxide is well known as photocatalyst, where it has been shown that, in most conditions, the anatases are the most active ones.

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### THE TEAM

The Chemistry of Surfaces, Interfaces and Nanomaterials (ChemSIN) department of the faculty of Sciences of ULB is supervised by Professor François Reniers. The plasma group of ChemSIN is focused on COLD ATMOSPHERIC PLASMAS and SURFACE TREATMENTS. The laboratory is equipped with 15 plasma reactors (among them, 12 are atmospheric plasmas), a monochromatized X-ray photoelectron spectrometer (XPS), a FTIR spectrometer, DCA dynamic angle contact measurement system, an atmospheric mass spectrometer and an optical emission spectrometer.

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### THE INVENTORS

**Professor François Reniers** is professor of chemistry at the Faculty of Sciences of the Université libre de Bruxelles (ULB). He got a PhD in Sciences (Chemistry) from ULB in 1991 and was a post-doctoral fellow at the University of California at Berkeley. He was an invited professor at the Chinese University of Hong Kong in 2000. He is currently the head of the ChemSIN group at ULB.

Initially, his research activities are fundamental physical chemistry at surfaces, which he studied using Auger electron and X-ray photoelectron spectroscopies. Since 1999, he oriented his team towards the development of atmospheric plasmas for interfaces, to modify or deposit new layers. François Reniers has published 5 book chapters, more than 130 articles and is the inventor of 13 patents. In 2018, he was made fellow of the AVS (USA) for: "seminal contributions to atmospheric plasma synthesis of organic, inorganic and hybrid coatings to attain a wide variety of functional properties and dielectric barrier discharge CO<sub>2</sub> conversion into useful molecules"

**Antoine Remy** is a PhD student currently working on nitrogen fixation in the ChemSIN (Chemistry of Surfaces, Interfaces and Nanomaterials – ULB). The idea of the new heating device based on induction currents came when he joined the ChemSIN in 2018. Indeed, Antoine was supposed to synthesize crystalline vanadium oxide coatings by atmospheric plasma, and the resistive devices used in the laboratory were creating major troubles with the high frequency and the sensitive materials needed for atmospheric plasma generation. Also, Antoine wanted to obtain coatings by a "all-in-one-step" process (i.e. without post-annealing).

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### RELEVANT PUBLICATIONS

> Improving the atmospheric plasma deposition of crystalline inorganic coatings, Remy A., Serigne Fall M., Segato T., Godet S., Delplancke-Ogletree M.P., Panini P., Geerts Y., Reniers F., Thin Solid Films, vol. 688, octobre 2019, p. 137437.

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