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# The potential of plasma activation for EB-PVD of EBC systems on CMC components

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# THE POTENTIAL OF PLASMA ACTIVATION FOR EB-PVD OF EBC SYSTEMS ON CMC COMPONENTS

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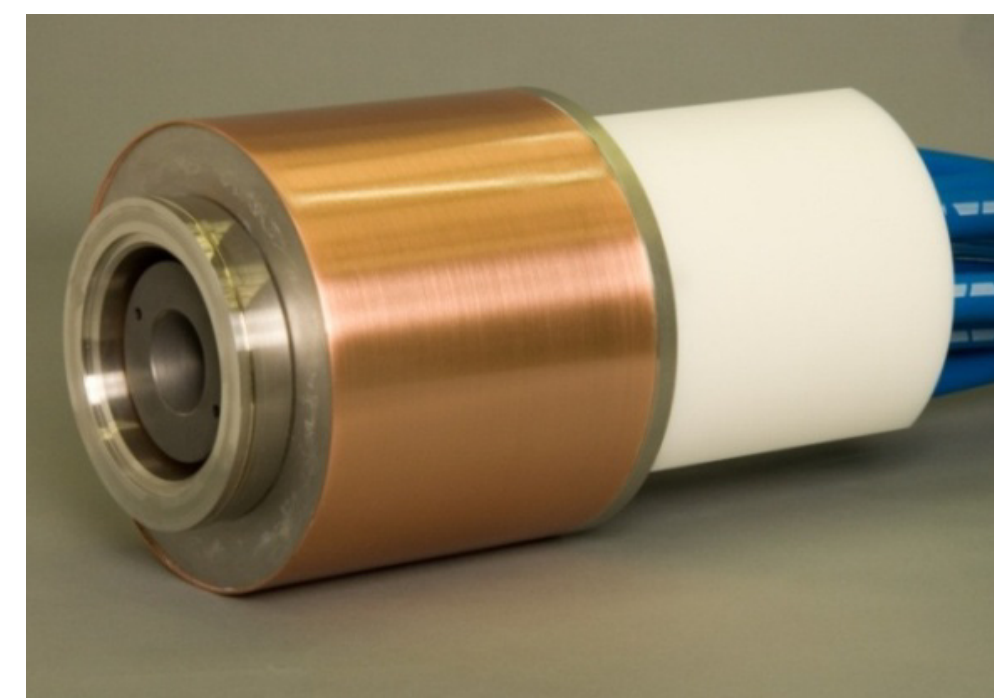
## INTRODUCTION

Current developments on gas turbines focus on fuel efficiency, reduction of emissions, or enhanced power. One of the major keys is improving the thermal efficiency by increased gas temperature within the turbine. Therefore silicon-based non-oxide ceramics are being investigated and introduced as new base materials for turbine components, allowing for lower-weight components operating at elevated temperatures. However, severe weight losses due to water vapor corrosion, increasing temperature demands and other challenges in terms of mechanical and chemical stability require further research and development on protecting environmental barrier coatings (EBC) to be applied on these ceramic components.

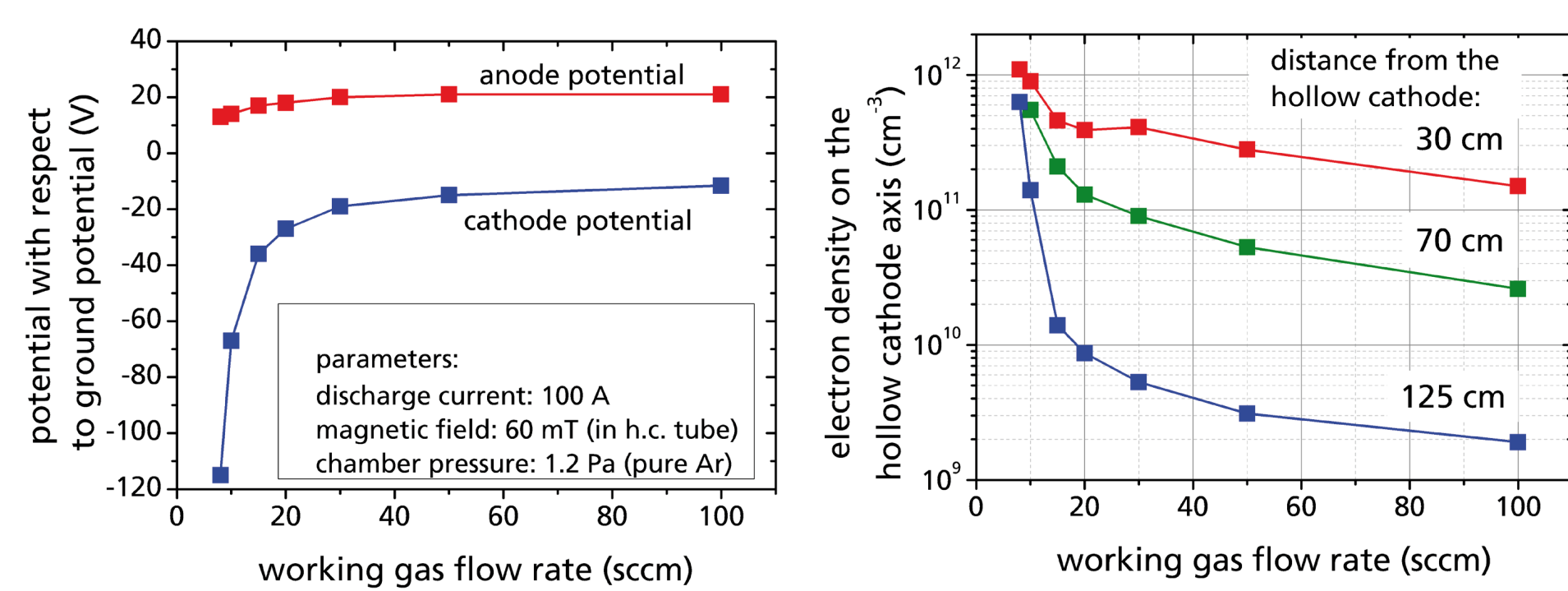
Fraunhofer FEP has been developing PVD processes as well as corresponding hardware such as customized EB guns and plasma sources in a large field of applications for decades. In the most cases, plasma activation of the PVD process is the key to success in order to combine high-rate film growth with the requested film properties. These experiences show that the beneficial effect of plasma activation could complement current EBC developments to address present challenges. In this paper, FEP's hollow cathode arc plasma source for activating high-rate PVD processes is presented. As an example of application, the plasma activation effect onto EB-PVD of yttria-stabilized zirconia (YSZ) layers is shown.

## HOLLOW CATHODE ARC PLASMA SOURCE LAVOPLAS

In order to achieve significant effects by plasma activation in high-rate PVD processes, it is imperative to utilize high-density plasma sources providing high ion current densities on the substrate. The hollow cathode arc discharge generates dense large-volume remote plasmas and fulfills the requirements of a robust high-power plasma source.



Picture of the hollow cathode arc plasma source LavoPLAS

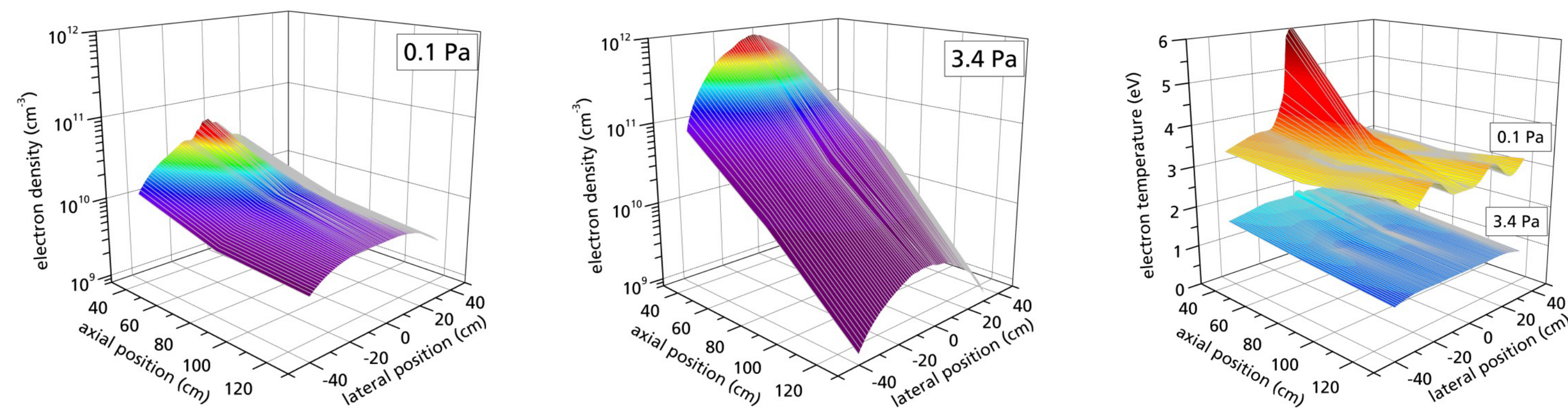
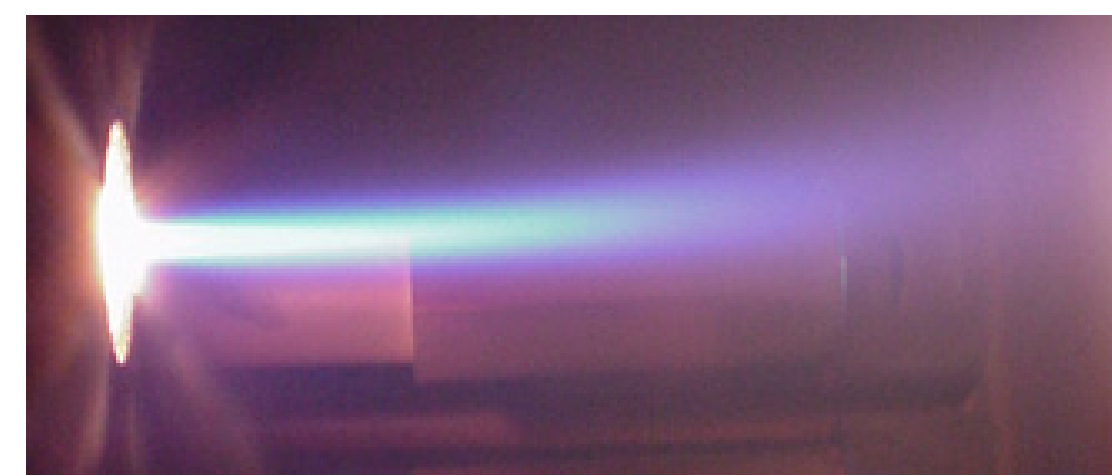


Cathode and anode potentials as well as the electron density on three axial positions from the plasma source

The hollow cathode consists of a tantalum cathode tube which is flown through by the working gas argon. A coaxially arranged coil generates a magnetic field which allows for reduced working gas flow resulting in strongly increased plasma density and range due to larger cathode fall potential and electron energies at a discharge power of up to 30 kW.

The hollow cathode arc consists of two plasma regions:

- **Internal plasma:** electrons are thermionically emitted from the hot cathode heated by impinging ions within the tube
- **External plasma:** a low-voltage beam of electrons from the internal plasma ionizes the gas in the process chamber

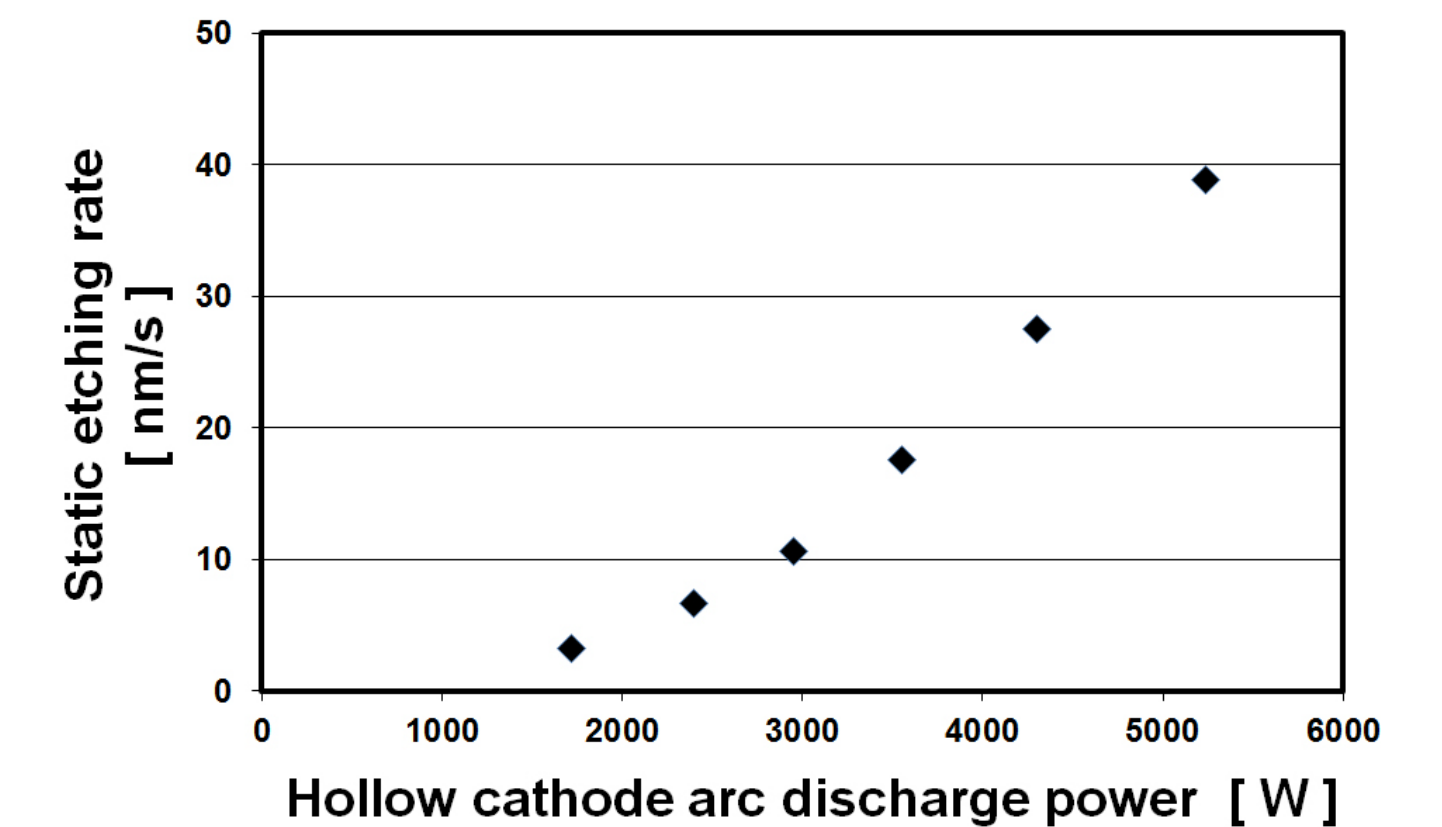


3D diagrams of the electron density and of the the electron temperature at a discharge current of 50 A and a chamber pressure of 0.1 and 3.4 Pa, respectively

## ASPECTS OF PLASMA UTILIZATION IN PVD PROCESSES

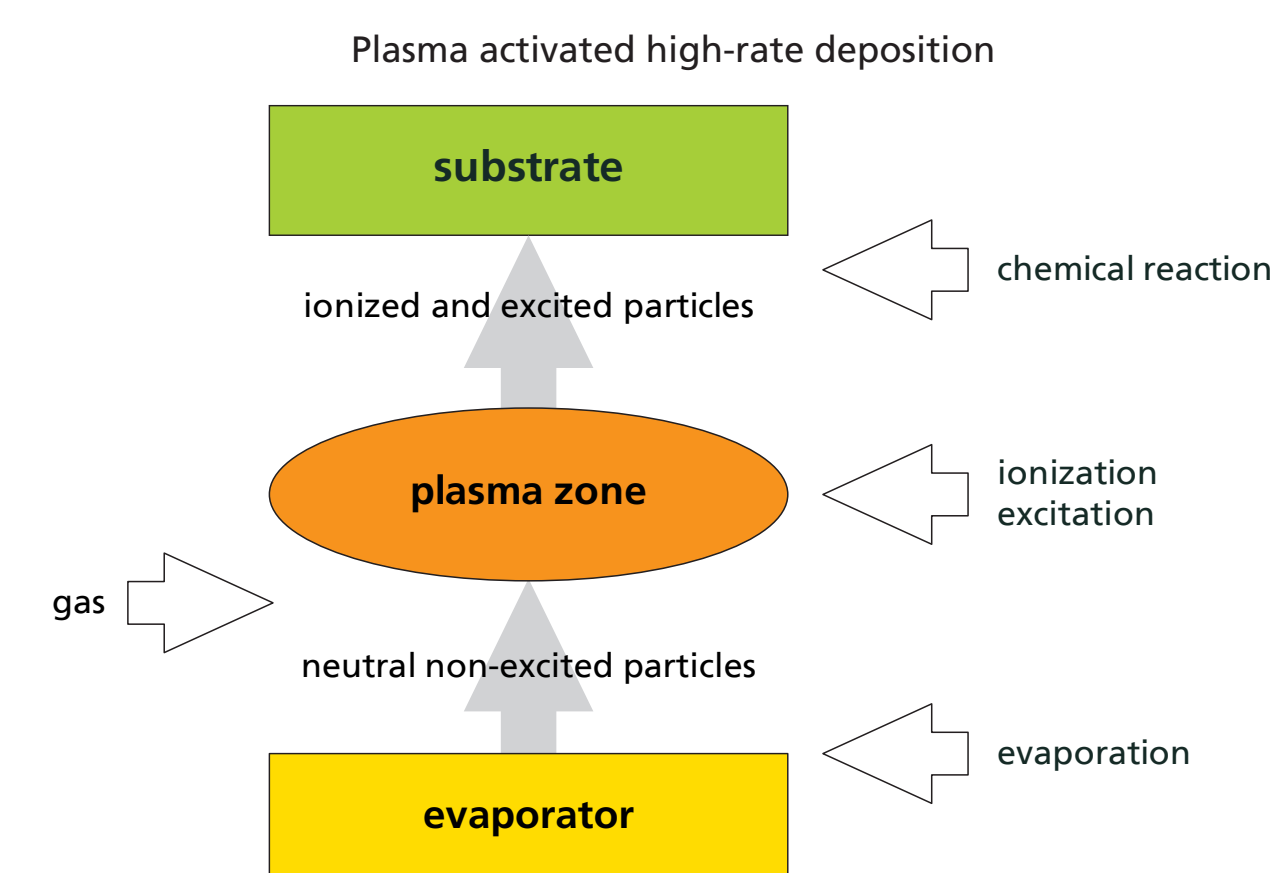
### PLASMA PRETREATMENT OF SUBSTRATES

Plasma pretreatment is an essential process step in order to clean the substrate surface before coating and to improve the layer adhesion. The charge carriers attracted onto the substrate reveal different effects and can be chosen by the polarity of a bias voltage. Whereas electron bombardment can be used for preheating, the ions allow for removing surface atoms by sputter etching. As an example, the etching rate of hollow cathode enhanced sputter etching of copper at a bias voltage of 500 V is shown, which increases with discharge power and reaches high static values of up to 40 nm/s.



Static etching rate on copper at different hollow cathode discharge powers

### PLASMA ACTIVATION OF REACTIVE PVD PROCESSES



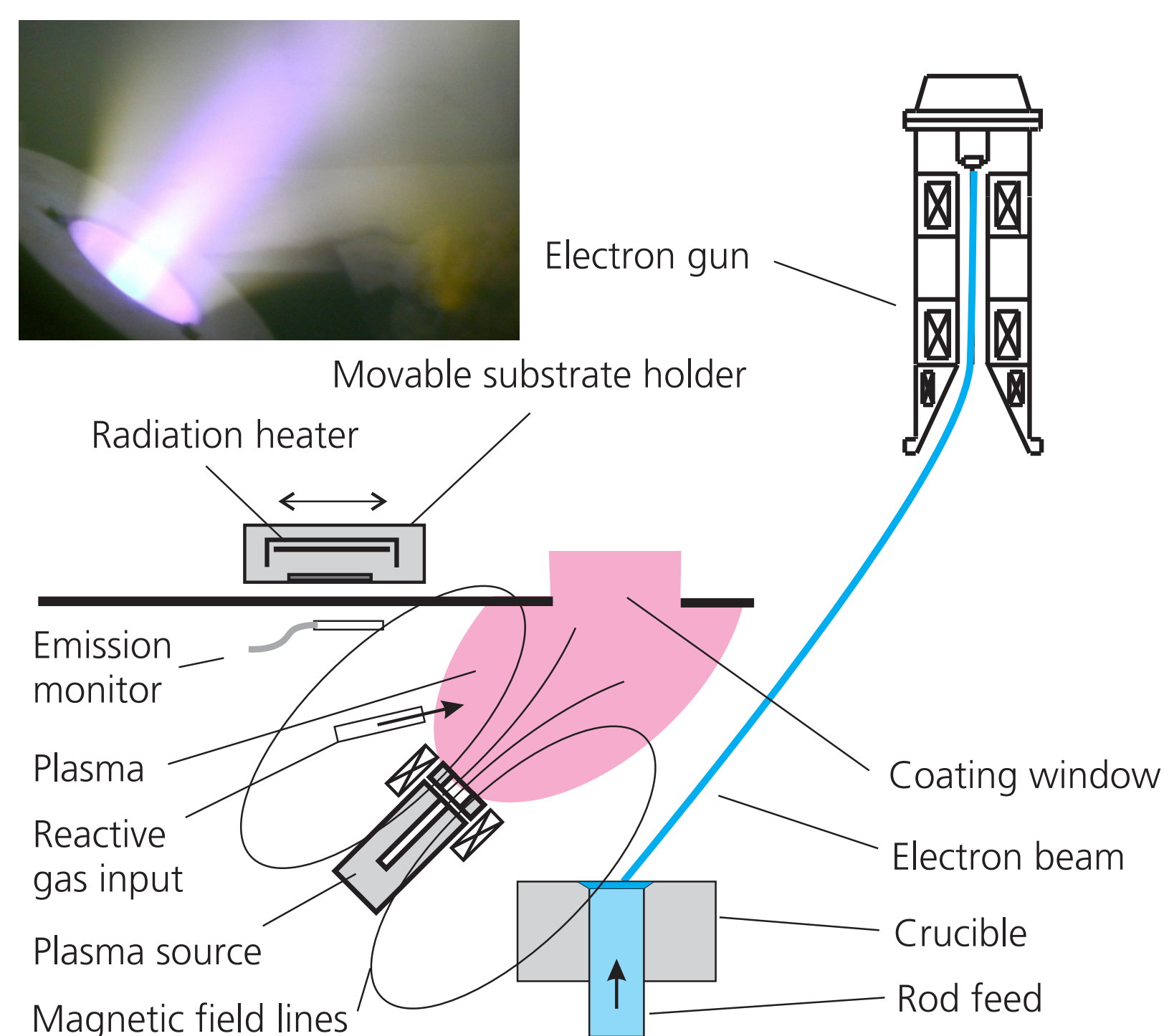
Besides the effects of electron and ion bombardment mentioned above, plasma activation includes excitation, ionization, and dissociation processes within the vapor and reactive gas before condensing on the substrate. Furthermore, their energy is increased due to acceleration within the plasma sheath or by a variable bias voltage. All these effects considerably influence the film growth and the eventual layer properties.

### SELECTED APPLICATIONS

- **EB-PVD of  $Al_2O_3$  on steel:** coatings deposited with plasma activation reveal a drastically increased hardness, higher density and improved chemical stability. Moreover, nanocrystalline films can form already at relatively moderate temperatures around 700°C.
- **Thermal evaporation of  $Al_2O_3$  on plastic web:** plasma activation allows for deposition of hard dense layers at low substrate temperatures. Due to the remarkably increased water vapor permeation barrier, this coating process has been transferred into the food packaging industry.
- **EB-PVD of  $SiO_x$  on steel and glass substrates:** with plasma activation, transparent abrasion resistant layers with high hardness have been produced.

## REACTIVE PLASMA-ACTIVATED EB-PVD OF YSZ LAYERS

### EXPERIMENTAL SETUP



Setup including a hollow cathode arc plasma source with magnetic enhancement

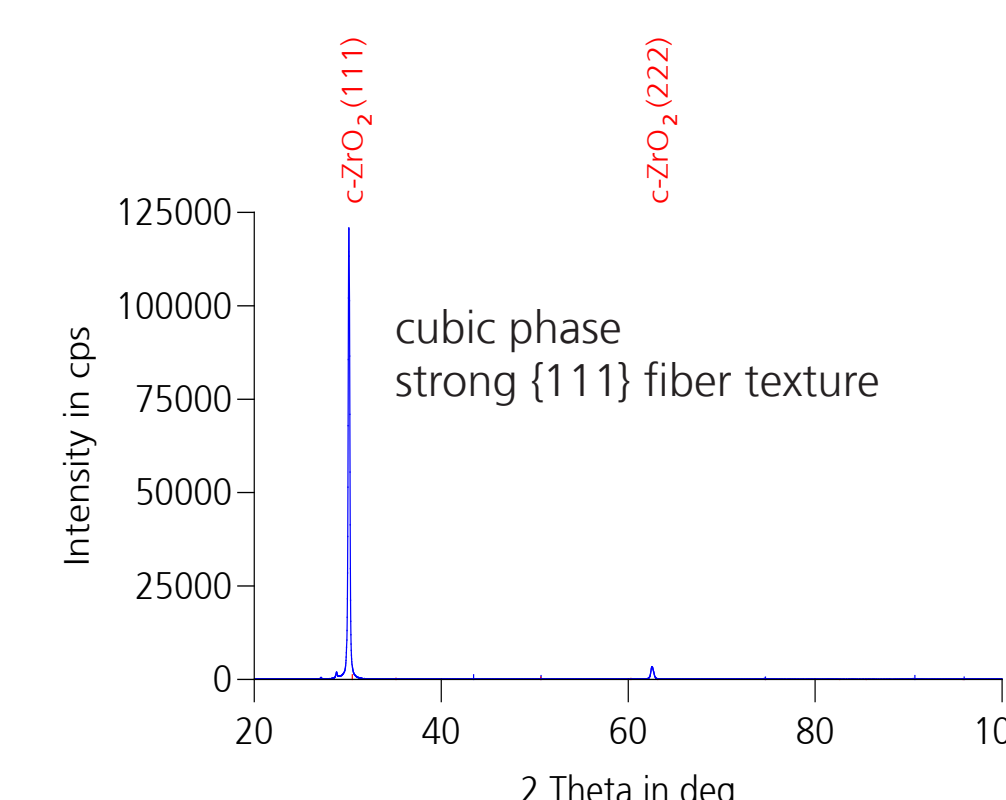
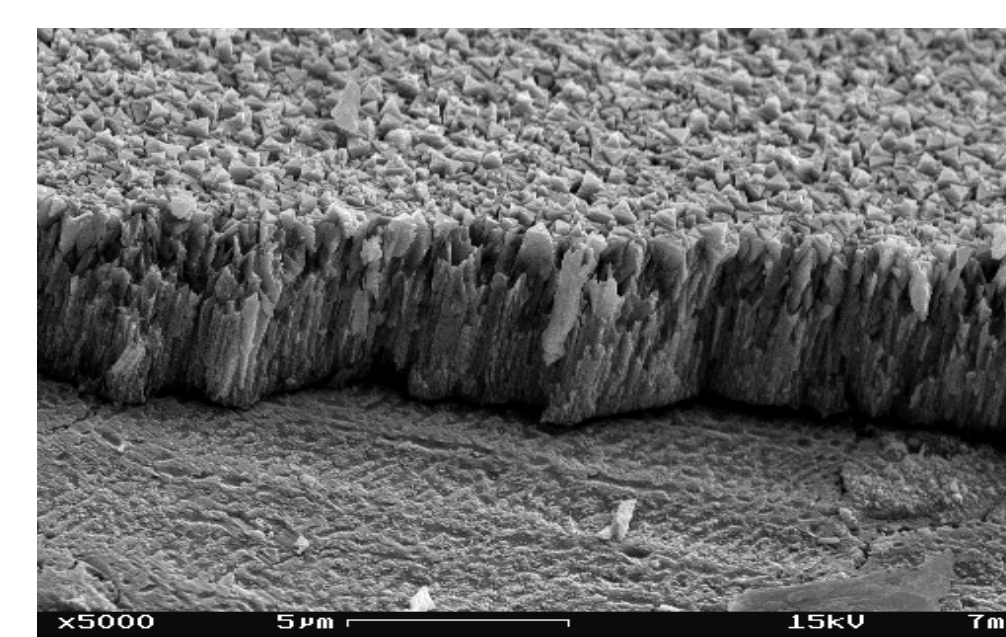
### PARAMETERS FOR EB-PVD OF YSZ (8 MOL%) ON STEEL SUBSTRATES

- pressure < 0.5 Pa
- substrate temperature: 900°C
- deposition rate: 40 nm/s
- thickness 5  $\mu$ m

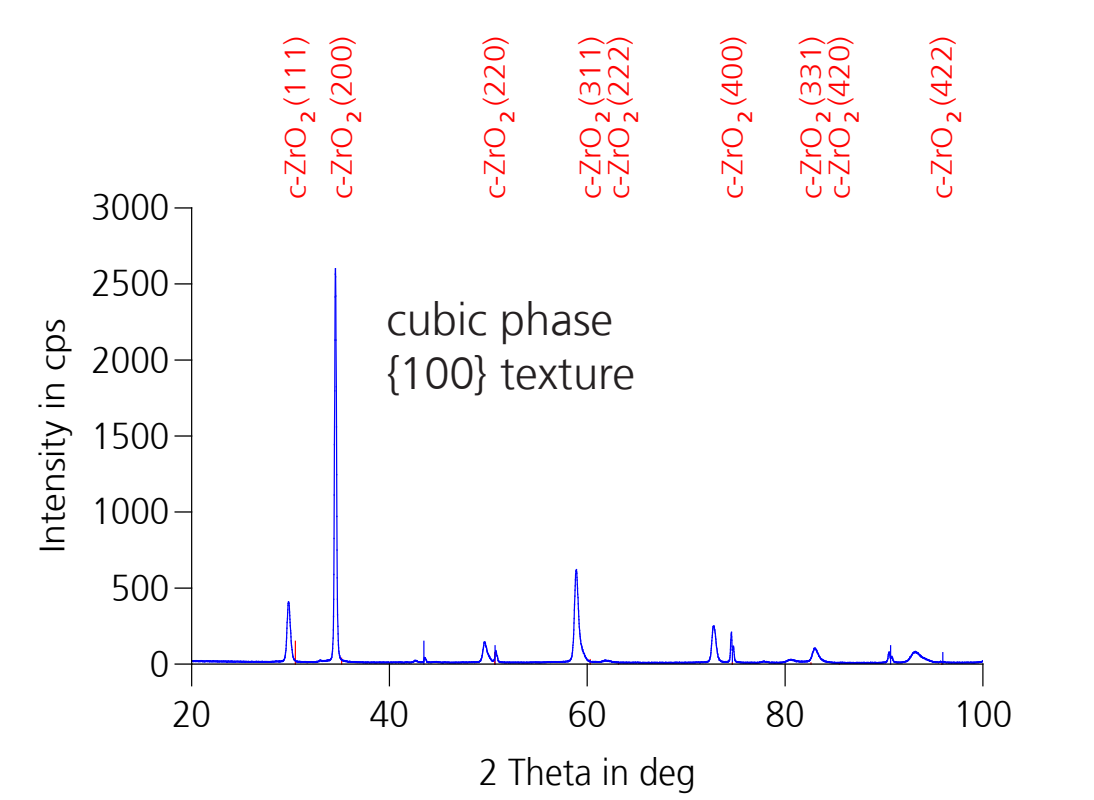
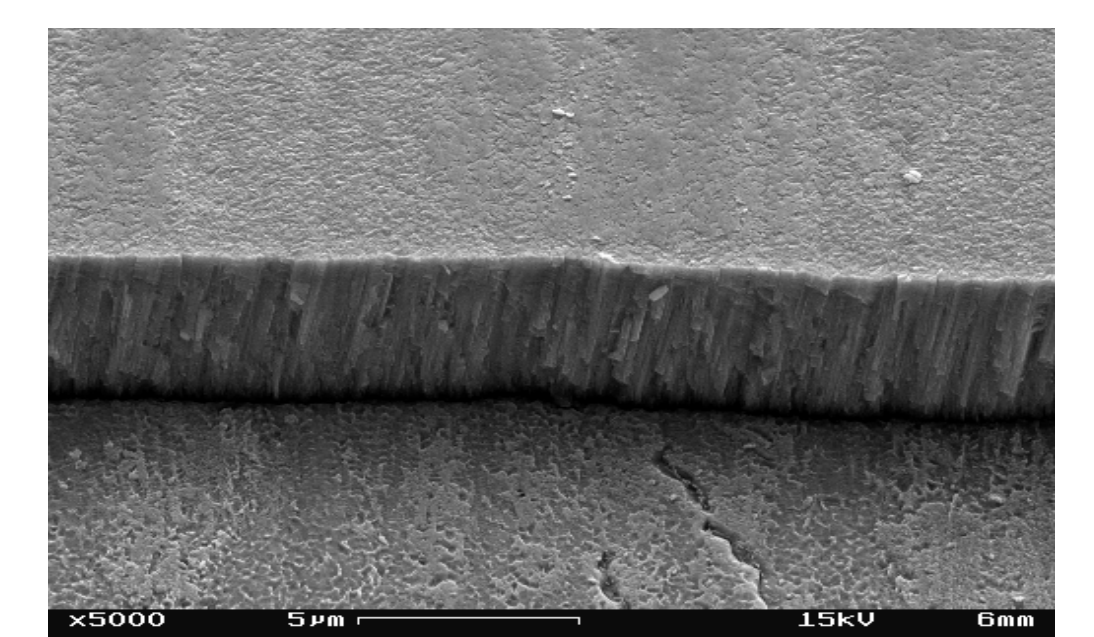
### RESULTS

The application of plasma led to an ion current density of 25 mA/cm<sup>2</sup> onto the substrate. REM shows denser and smoother microstructure. The XRD phase composition analysis revealed a significant change in texture. The microhardness was increased from 5 to 20 GPa. The objective was to achieve dense electrolyte membrane layers for solid oxide fuel cells, which are gas-tight, but allow for controlled ion diffusion.

### WITHOUT PLASMA



### WITH PLASMA



SEM pictures and XRD spectra of the YSZ films deposited by EB-PVD without (left) and with (right) plasma activation

## CONCLUSION

Dense plasmas are used for fast substrate pretreatment as well as for assisting high-rate PVD processes. The species prevalent in vapor and reactive gas are excited, ionized and dissociated, resulting in modified and customized film properties. Dense protective coatings with enhanced chemical stability can be deposited at relatively low substrate temperatures, which has been shown using the example of YSZ, the standard material for thermal barrier coatings on turbine components. It is expected that plasma activated processes could have great benefits for EBC applications as well.

## REFERENCES

- J.-P. Heinß et al., EP 2 087 503 B1, "Device for the pre-treatment of substrates", 31.10.2007  
 G. Mattausch et al.: 55th SVC Annual Technical Conference Proceedings, 179-185 (2012)  
 H. Morgner et al., Plasma Processes and Polymers 4, S551-S556 (2007)  
 S. Schiller et al., Surface and Coatings Technology 125, 240-245 (2000)  
 S. Schiller et al., Surface and Coatings Technology 125, 354-360 (2000)  
 B. Zimmermann et al.: Surface and Coatings Technology 205, S393-S396 (2011)  
 O. Zywitzki et al., Surface and Coatings Technology 151-152, 14-20 (2002)

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