

PLASMA-BASED TOOLS FOR ACTIVATED EB-PVD OF TBC SYSTEMS

B. ZIMMERMANN, G. MATTAUSCH, B. SCHEFFEL, J.-P. HEINSS, F.-H. RÖGNER, C. METZNER

FRAUNHOFER INSTITUTE FOR ELECTRON BEAM AND PLASMA TECHNOLOGY FEP, DRESDEN, GERMANY

INTRODUCTION

Physical Vapor Deposition (PVD) techniques are increasingly applied in the field of surface engineering, functionalization, and protection in order to meet high demands on corrosion resistance, thermal stability, or mechanical, optical, and electrical properties. However, depending on the required layer properties, high rate electron beam (EB)-PVD

processes often call for additional plasma activation in order to combine high rate film growth with outstanding film quality by ionized vapor, enhanced reactive gas reactivity and hence elevated or tailored particle energy. In this poster, plasma-based EB tools with reference to plasma-activated EB-PVD e.g. of TBC systems are presented.

A new type of cost-efficient EB guns has been developed at Fraunhofer FEP, where beam electron emission from the cathode is stimulated by ion bombardment from a high-voltage glow discharge. Furthermore, a high-power large-volume plasma source based on the hollow cathode arc discharge has been established as a compact, universal and

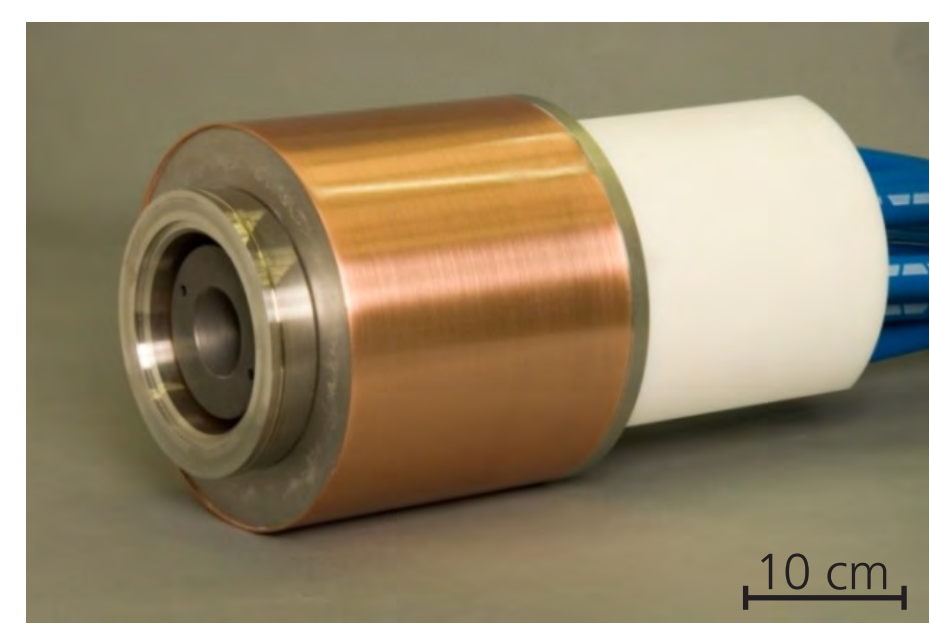
flexible tool for efficient substrate pre-treatment and etching as well as assisting high rate PVD processes. The effect of the hollow cathode arc plasma for sputter etching of metallic substrates and for plasma-activation of EB-PVD of YSZ is shown to document its high application potential for the field of PVD technology.

HOLLOW CATHODE ARC PLASMA SOURCE LAVOPLAS

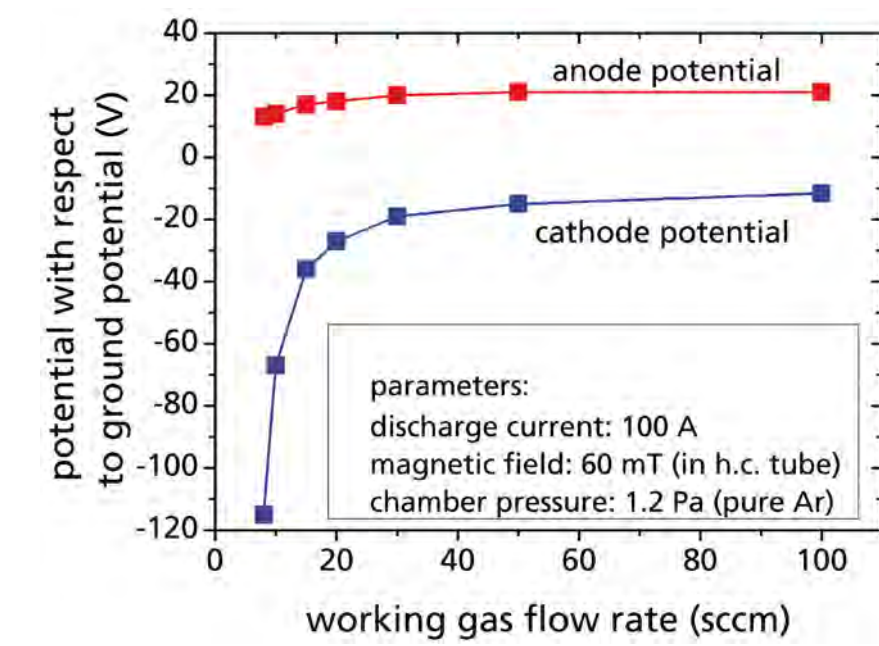
A ring anode and a magnetic field coil are arranged coaxially around the tantalum cathode tube flown through by the working gas argon:

- diffuse arc discharge within the cathode tube
- large volume plasma in the process chamber

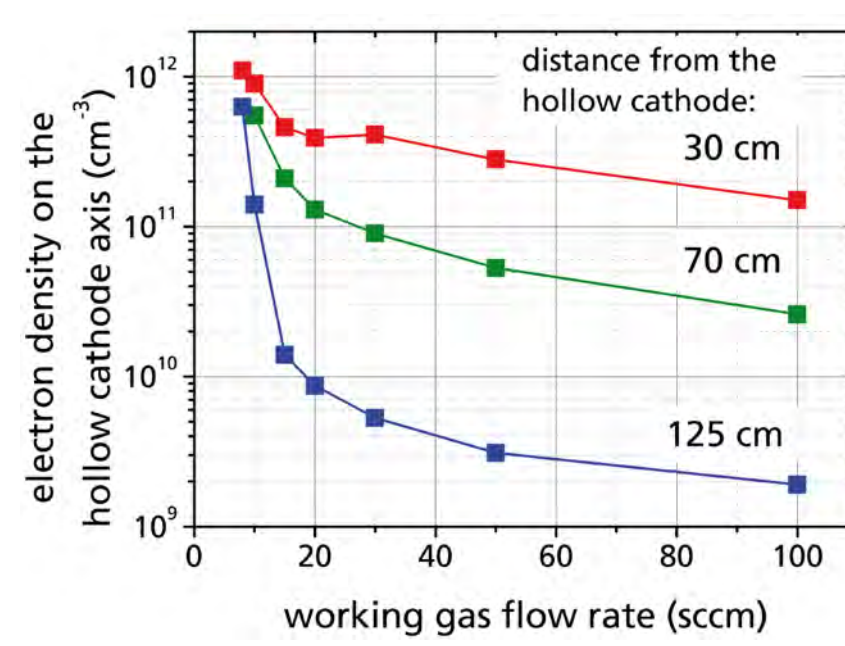
An axial magnetic field allows for reduced working gas flow rates resulting in strongly increased plasma density and range due to a larger cathode drop potential and electron energies at discharge powers of up to 30 kW.



Picture of the hollow cathode arc plasma source LAVOPLAS

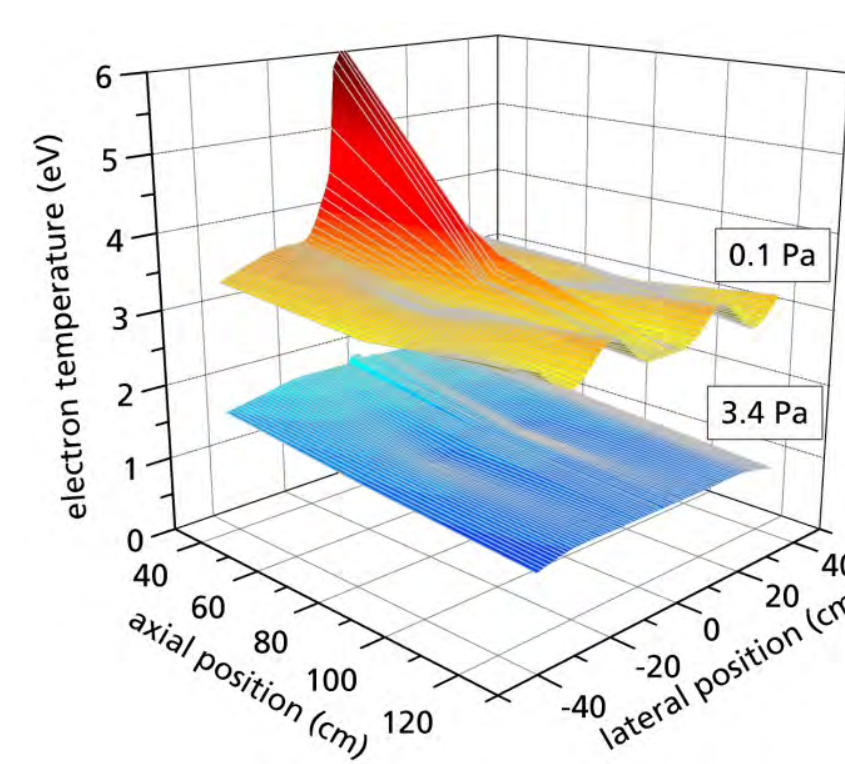
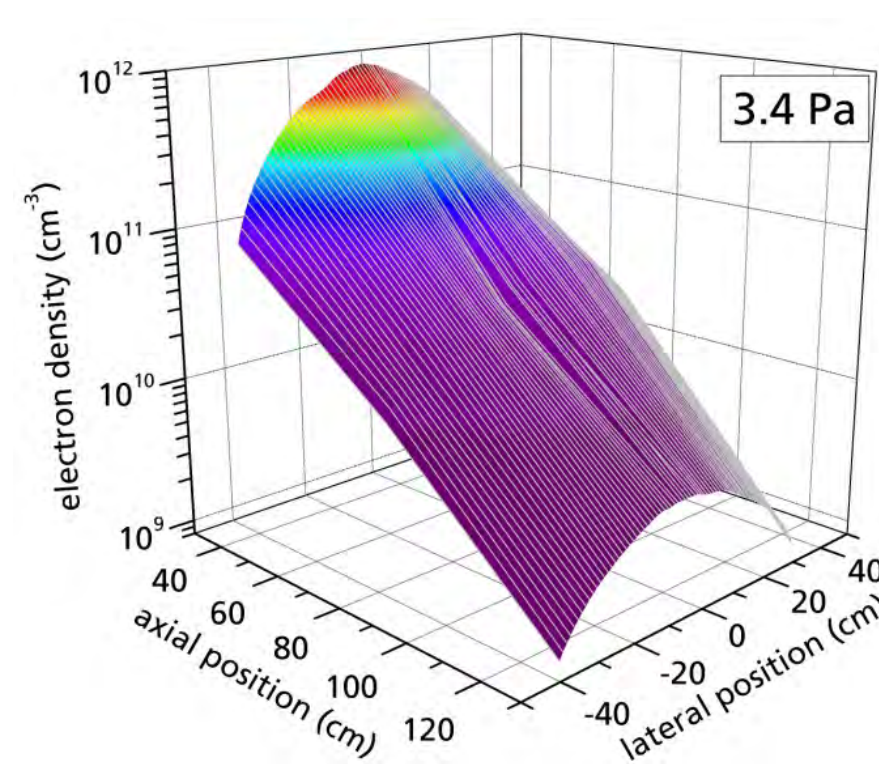
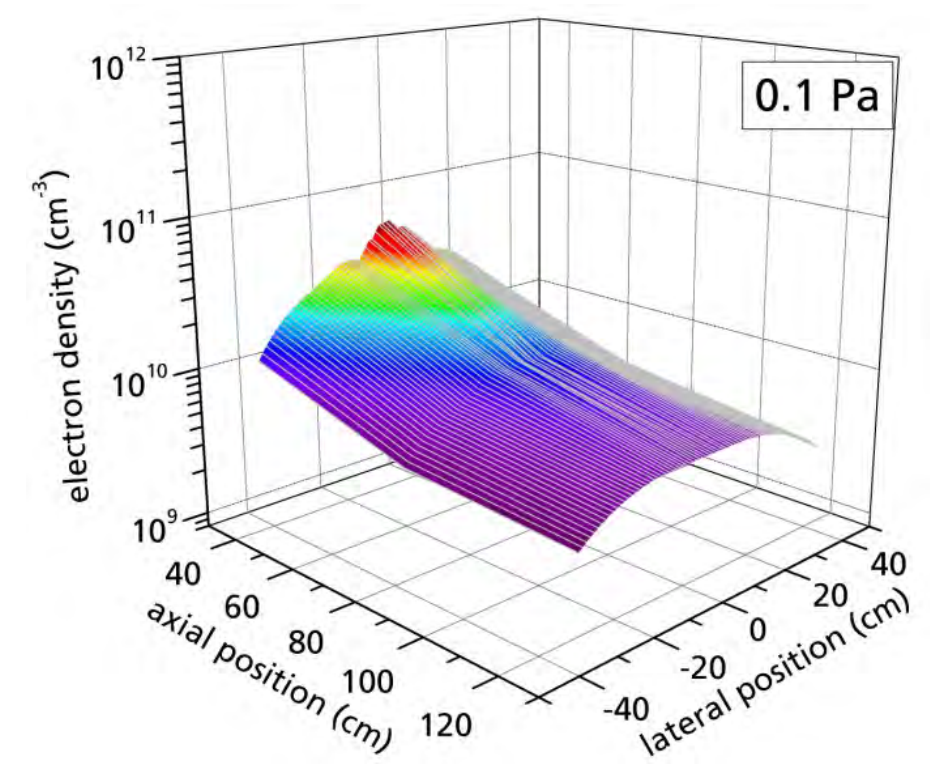


Cathode and anode potentials as well as the electron density on three axial positions from the plasma source



The hollow cathode 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 ionize the gas in the process chamber

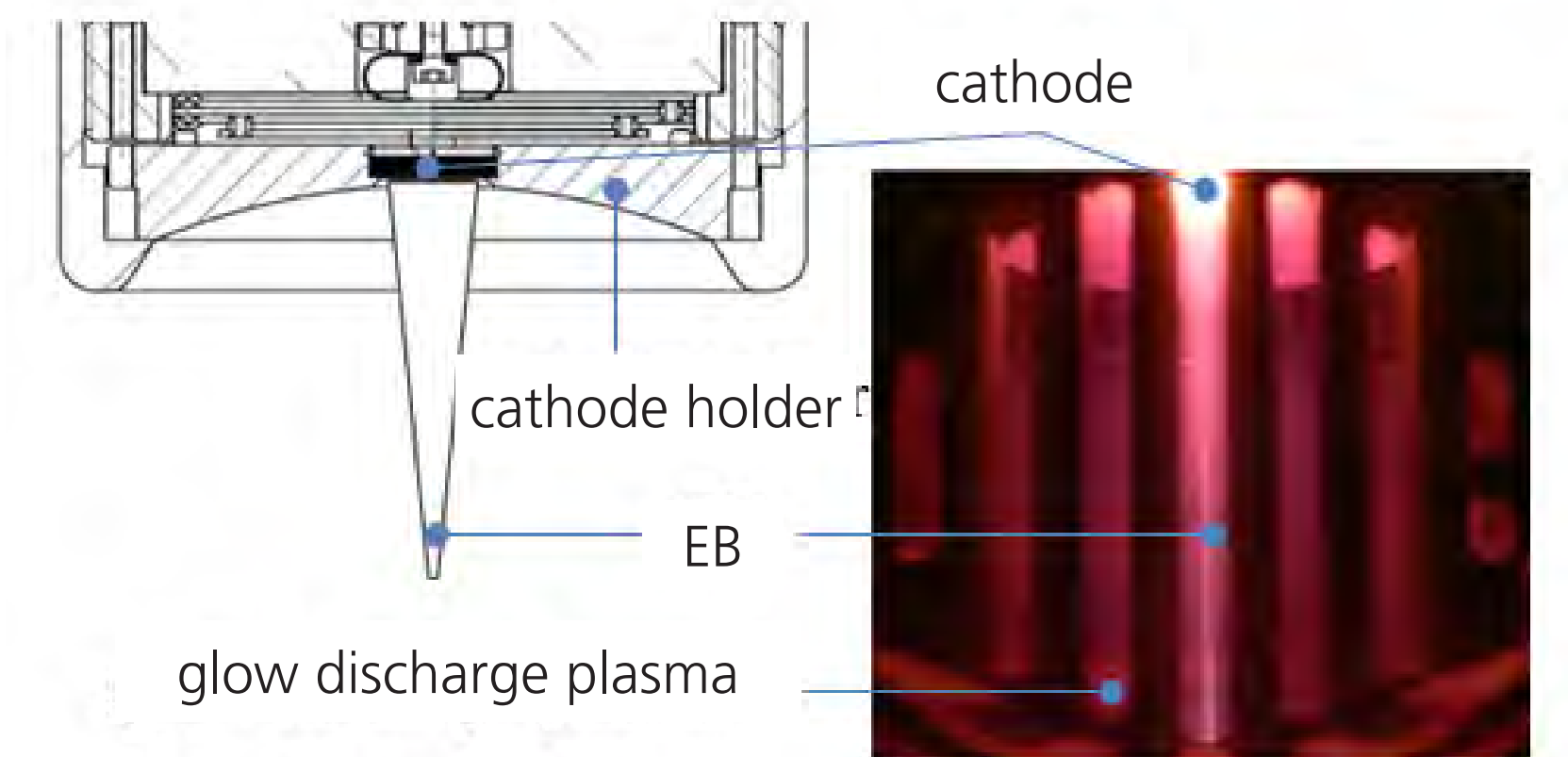


3D diagrams of the electron density and of the electron temperature at a discharge current of 50 A and a chamber pressure of 0.1 and 3.4 Pa, respectively

DISCHARGE-BASED HIGH-POWER EB GUN EASYBEAM

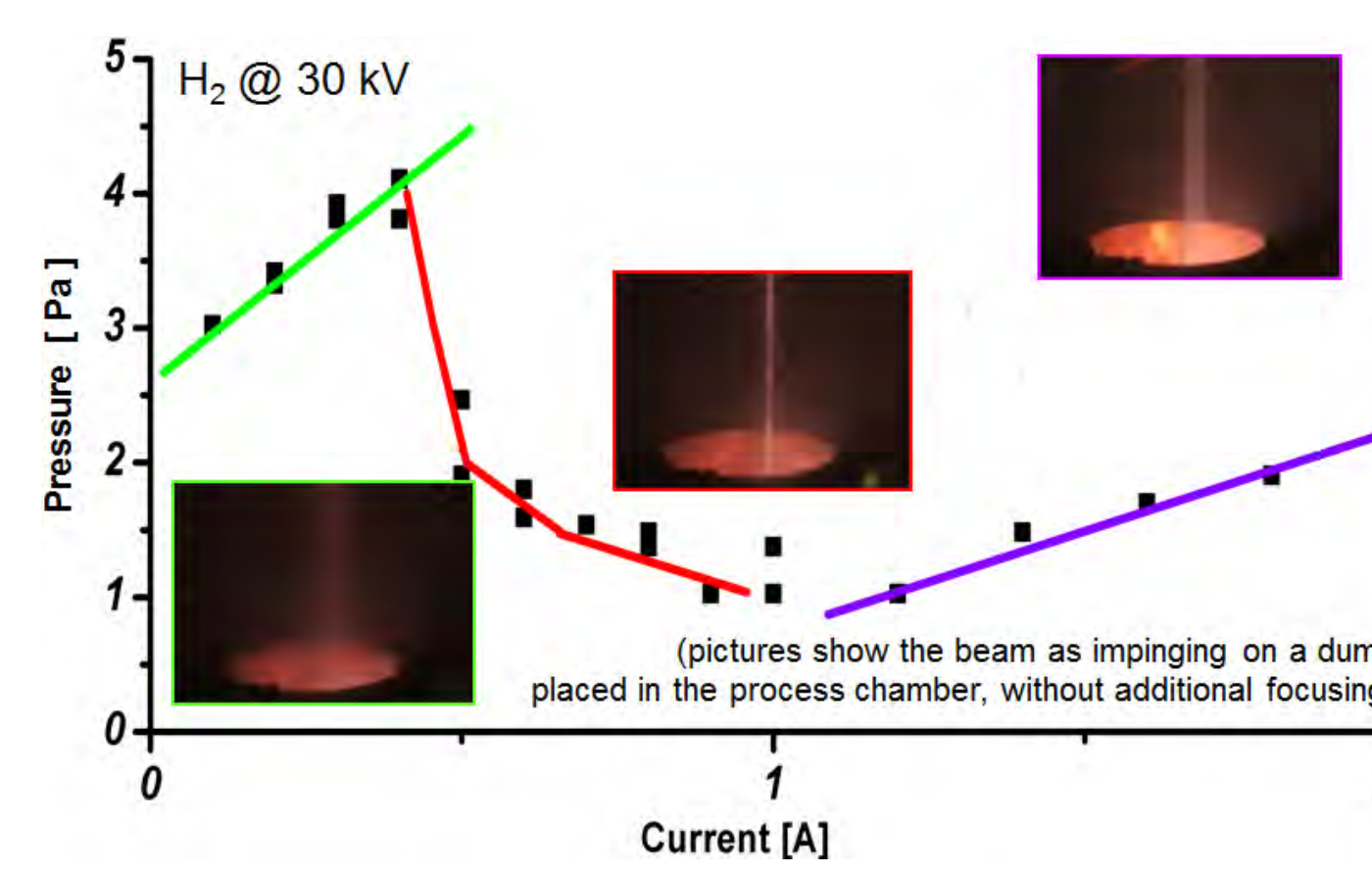
A block cathode made from LaB₆ or a refractory metal is mounted in a field-forming cathode holder. In the cathode chamber, a high-voltage glow discharge (HVGD) is ignited (working gas: He, H₂)

- cathode is heated up by plasma ions
- free electron generation via secondary electron emission as well as thermionic emission

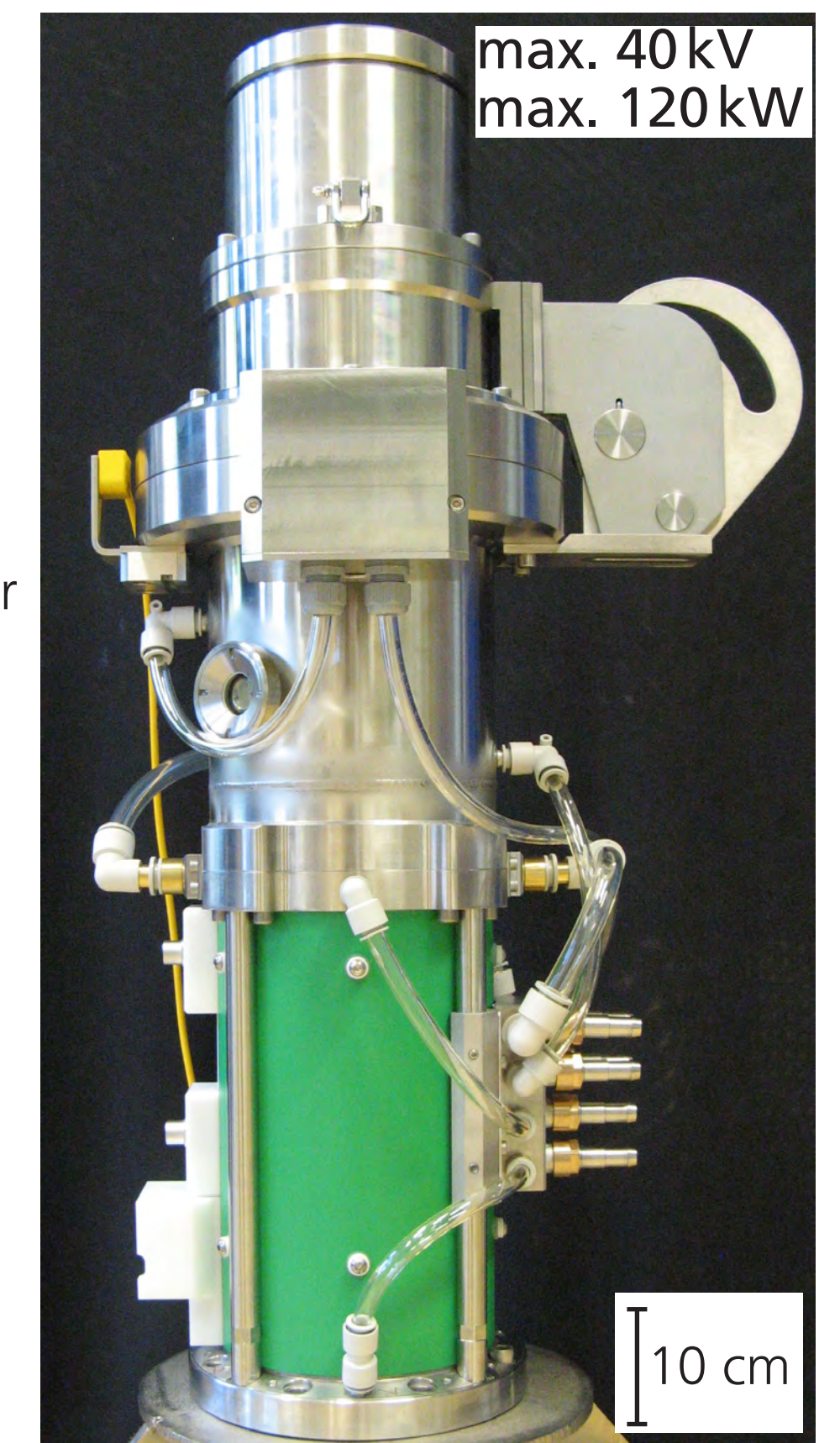


Features in comparison to conventional EB guns:

- **simplified electrical circuitry and high-voltage (HV) supply:** only one high voltage cable – no additional cathode heating supply floating on HV level necessary
- **simplified mechanical setup:** cathode unit consists of a simple cathode holder without sophisticated heating system components
- **gun-specific vacuum stage can be omitted:** cathode chamber work pressure in the range of 1-5 Pa, consequently no differential pumping necessary – evacuation of the gun via process chamber for process pressures up to 0.5 Pa



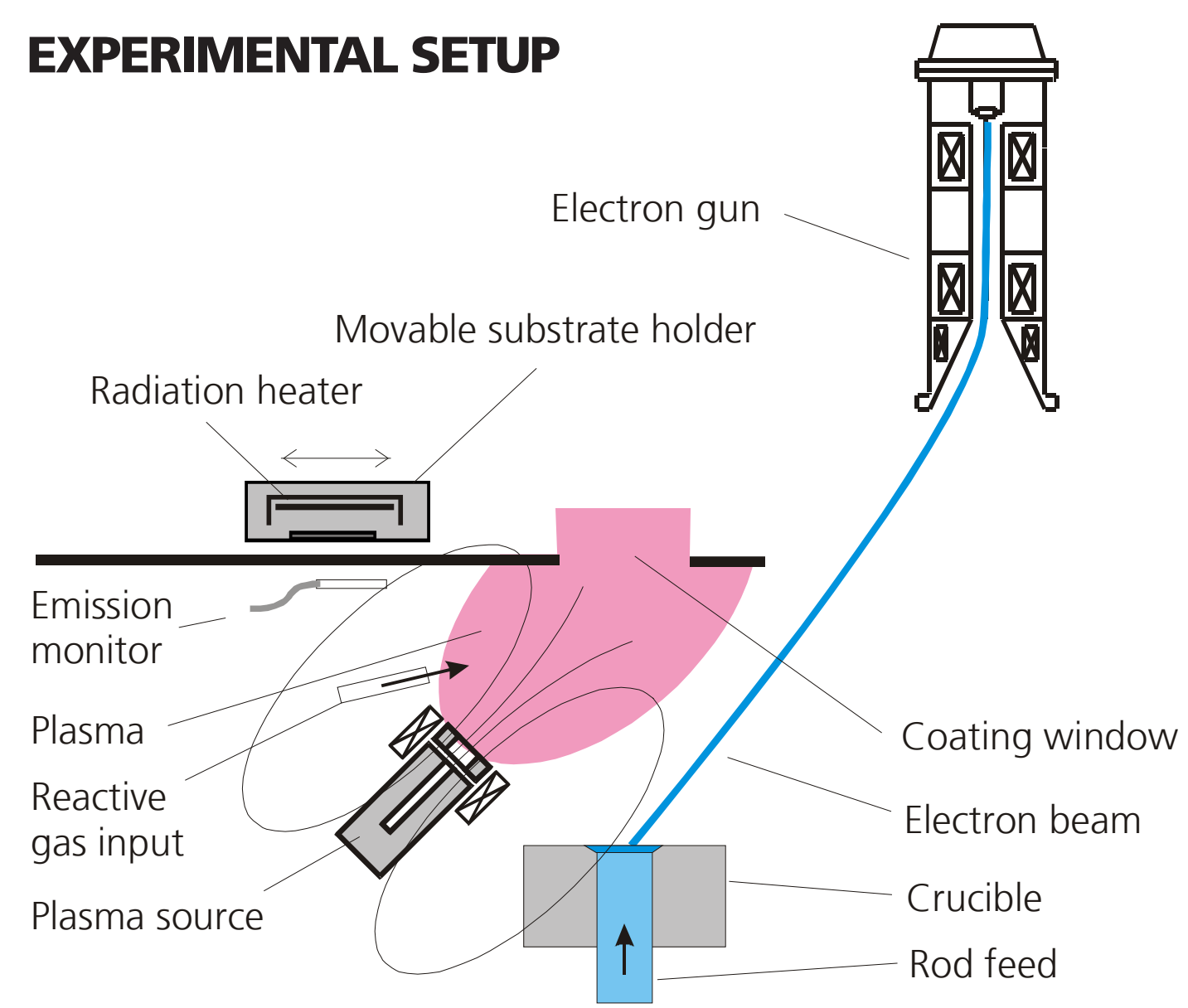
The EB current is controlled by the cathode chamber pressure, which is set by the working gas flow rate



Fraunhofer FEP's high-power EB gun EasyBeam

REACTIVE PLASMA-ACTIVATED EB-PVD OF YSZ LAYERS

EXPERIMENTAL SETUP



HOLLOW CATHODE ENHANCED SPUTTER ETCHING

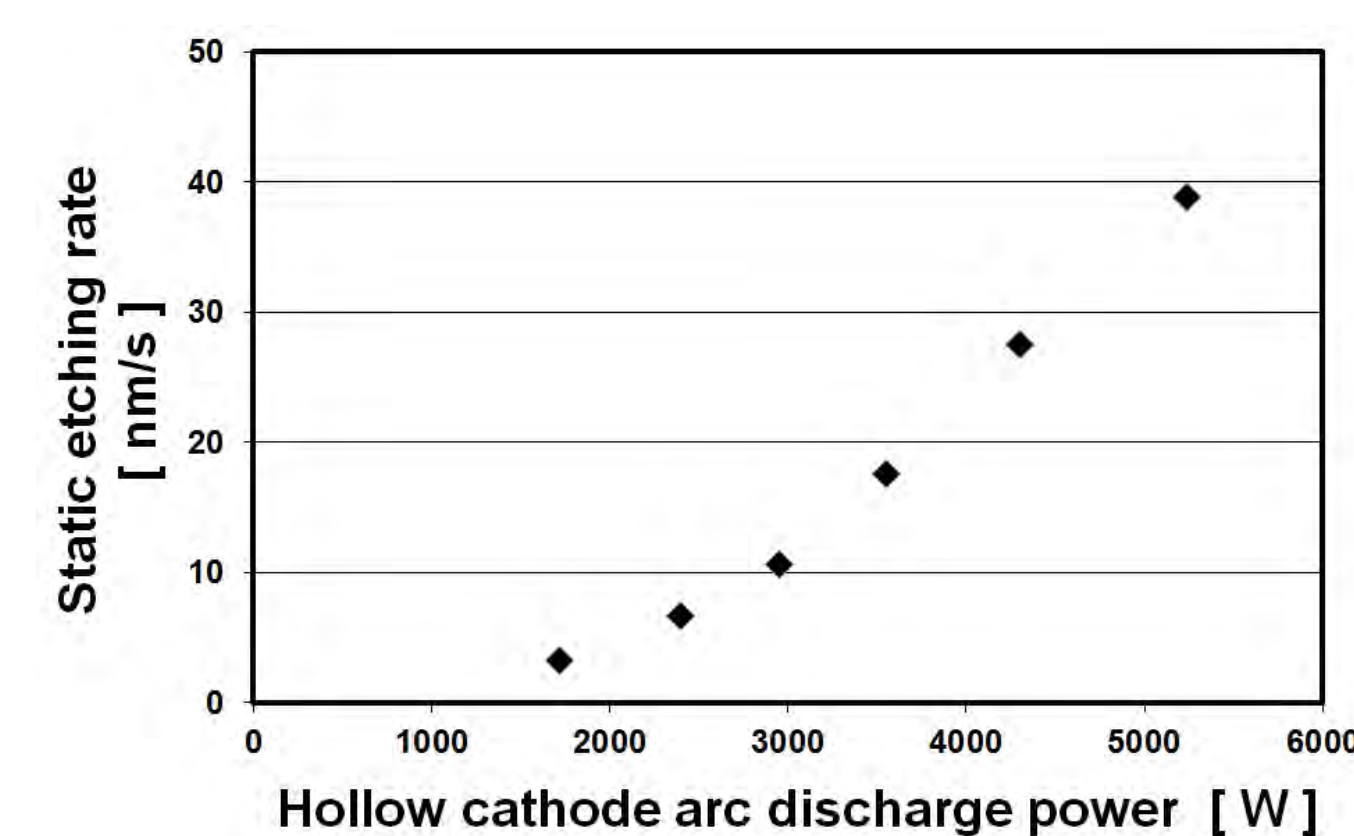
Plasma pretreatment is an essential process step in order to clean the substrate surface before coating and to improve the layer adhesion. In the following, hollow cathode enhanced sputter etching for the example of Cu is shown.

Parameters:

- discharge current: 100-200 A
- substrate at ground potential
- positive hollow cathode bias voltage: 500 V

Result:

The etching rate 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-ACTIVATED PVD OF YSZ LAYERS

During plasma activation, vapor and reactive gas are excited and ionized before condensing on the substrate. Furthermore, their energy is increased due to acceleration within the plasma sheath or by a variable bias voltage resulting in densified and smoothed coatings. For high-rate processes such as EB-PVD, the high-density plasma of the hollow cathode arc is necessary in order to reach sufficient ion current densities on the substrate.

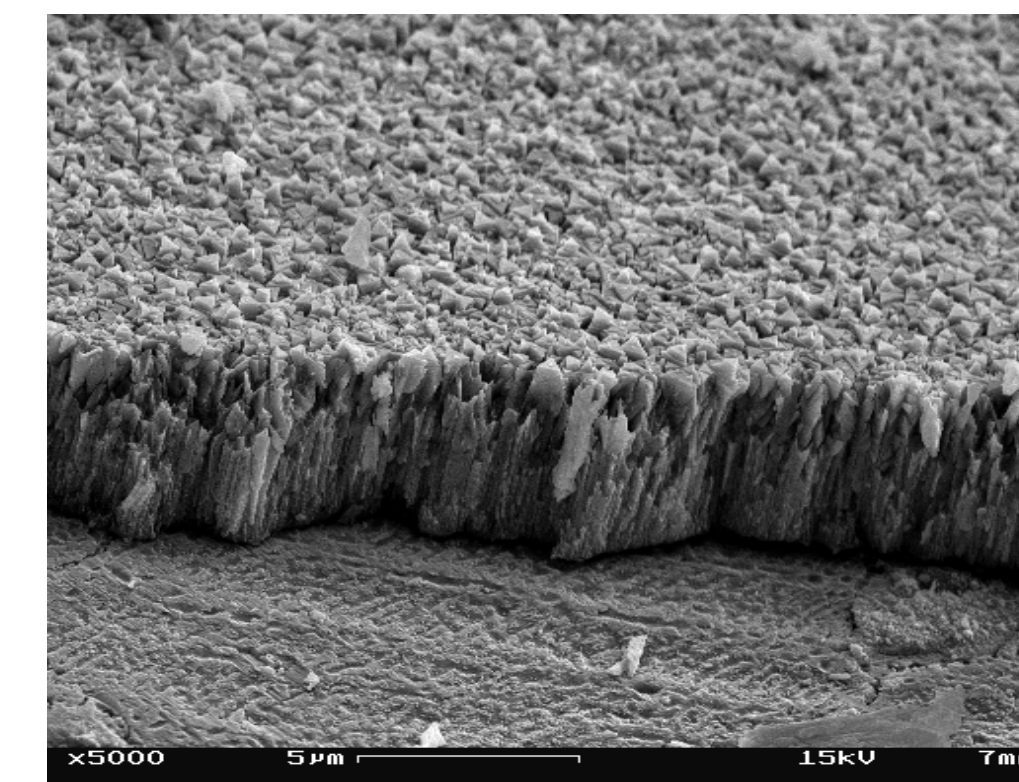
Parameters for EB-PVD of YSZ (8 mol%) on steel substrates:

- pressure < 0.5 Pa
- substrate temperature: 900 °C
- deposition rate: 40 nm/s
- thickness 5 μm

Results:

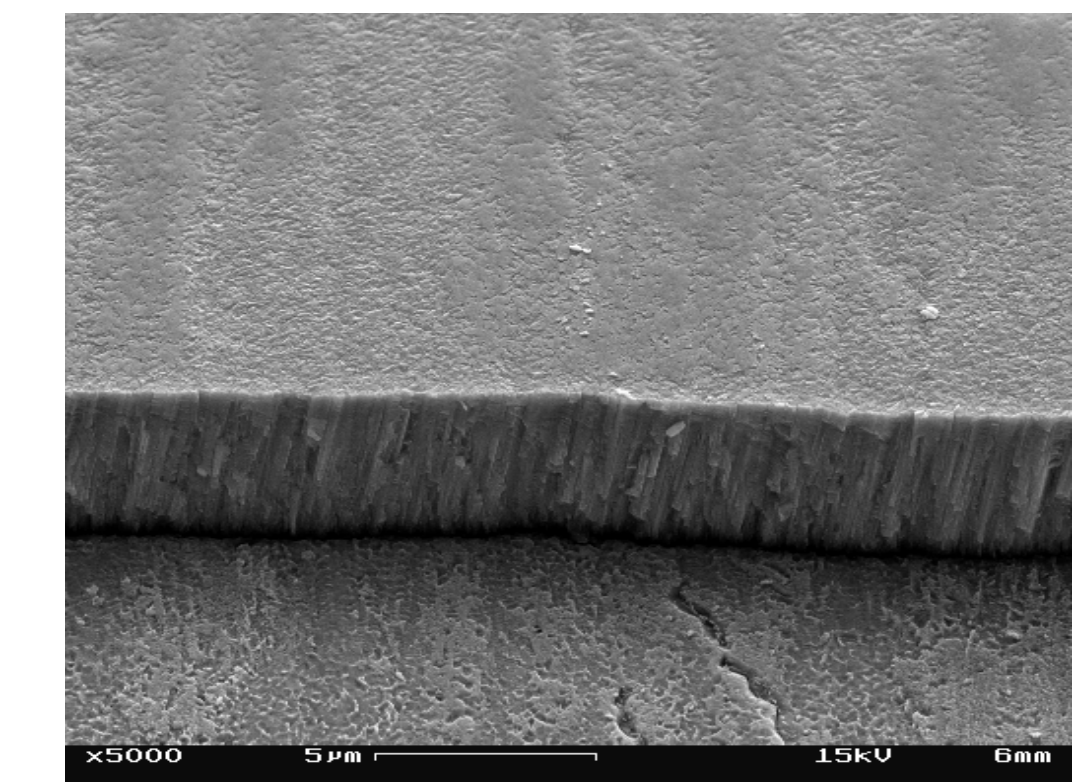
The application of plasma led to an ion current density of 25 mA/cm² onto the substrate. REM shows denser and smoother microstructure. The XRD phase composition analysis revealed a significant change in texture from {111} to {100}. The microhardness was increased from 5 to 20 GPa.

without plasma



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

with plasma



CONCLUSION

The discharge-based EB gun has been presented as a tool, which has the potential to allow for lower purchase cost in existing EB applications, or to cover coating application fields, where the EB technology has been too expensive so far. The high-power plasma activation with the hollow cathode arc plasma source has been shown to be very effective for plasma etching as well as for layer densification or tailoring of film properties, which is interesting e.g. for the fields of diffusion barrier coatings or morphology-adapted interlayers.

CORRESPONDING CONTACT

Fraunhofer-Institut für Elektronenstrahl- und Plasmatechnik FEP

Winterbergstraße 28
01277 Dresden, Germany

www.fep.fraunhofer.de

Dr. Burkhard Zimmermann
burkhard.zimmermann@fep.fraunhofer.de

Phone +49 351 2586 386



Corresponding Contact

REFERENCES AND ACKNOWLEDGEMENTS

G. Mattausch et al.: 55th SVC Annual Technical Conference Proceedings, 179-185 (2012)
P. Feinäugle et al.: 54th SVC Annual Technical Conference Proceedings, 202-209 (2011)
B. Zimmermann et al.: Surface and Coatings Technology 205, S393-S396 (2011)
J.-P. Heinß et al., EP 2 087 503 B1, "Device for the pre-treatment of substrates", 31.10.2007
Essential results were obtained in public project funded by Free State of Saxony „New technologies for solar applications“ (FZ 14274/2473)



Poster online (PDF)