A NEW TECHNIQUE FOR THIN-FILM COATING OF VACUUM VESSELS WITH METAL OR CARBON CONTAINING NON-EVAPORABLE GETTERS

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Abstract

A special CVD plasma-spraying technique has been suggested for thin-film coating of inner walls of vacuum vessels with two different kinds of non-evaporable getter materials. Whereas coatings of getters consisting of titanium-zirconium alloys and other metallic components are directly sprayed, new kinds of getter materials on carbon basis, like carbon whiskers, different fullerenes and carbon nanotubes, will be produced. Carbon radicals are formed in the gas phase during the CVD process first, and then thin films of carbon getters are generated on surfaces of different substrate materials. The high speed of the deposition process allows to control the layer thickness in a wide range. Coating parameters will be varied to optimize getter capacity and pumping speed, with the aim of obtaining low getter-activation temperatures. Micro-characterization of surface structures with electron microscopy and STM will be performed, as well as measurements of pumping speed, ultimate pressure and gas release. Photon-stimulated desorption of getter-coated substrates irradiated by synchrotron radiation at the DELTA accelerator are also planned.

1 INTRODUCTION

The University "Institute for Accelerator Physics and Synchrotron Radiation" operates the **D**ortmund 1.5 GeV **EL**ectron **T**est-Storage-Ring **A**ccelerator **DELTA**, a 3rd-generation synchrotron radiation source for basic research and technological developments. In the course of construction of this facility many years of experience have been gathered with non-evaporable getter (NEG) pumps, which are essential for the operation of an electron storage ring to obtain the necessary extreme ultra-high vacuum (UHV) in the range of 10^{-11} to 10^{-12} mbar. In order to get a uniform low-level pressure profile all around the circumference of the machine continuously distributed, so-called integrated ion getter pumps are mandatory. To have also sufficient noble-gas pumping capacity, DELTA is the first machine which has both types of integrated getter pumps installed side by side in an antechamber, namely integrated ion-sputter and NEG pumps (St707, the low-activation-temperature material), respectively. This guarantees stable conditions and lifetimes of the stored beam of at least 10 to 20 hours, a necessary condition for highly accurate long-time experiments.

Although used in all cathode-ray tubes nowadays, it turned out in the past, that the only commercially available NEG pumps of the material type St 707, which is a ternary metallic alloy of Zr-V-Fe, have several drawbacks. They are manufactured by pressing the metallic powder on to both sides of thin non-magnetic (constantan) sheet metal. The thin layers of getter material made this way suffer from poor adhesion and homogeneity and are not perfectly optimized concerning layer thickness, sorption speed and capacity.

With the aim of improving the quality of NEG pumps, which will be of great importance also for the next-generation of large high-energy accelerators, it was suggested by us to apply for the first time the vacuum-plasma-spraying (VPS) technique for depositing thin films of getter material on large substrate surfaces. By carefully adjusting the parameters of the production process, the intention is not only to optimize the sorption abilities of known metallic getters on Ti-Zr basis. One also wants to develop a highly effective class of completely new getter materials by applying the same well-known VPS-technology, in order to produce thin layers of nanostructured carbon (C) modifications, preferentially C-nanotubes (or C-fibers, C-whiskers, nanocrystalline C_{60}-fullerenes or mixtures of them [1]). All these nanostructured carbon modifications, deposited as finely-distributed, thin layers, lead to an enormous enlargement of active surface, which is perfectly suitable for sorption of large volumes of gas. Of particular interest is the ability of this new kind of getter materials, mainly layers of C-nanotubes, for sorption or storage of large quantities of hydrogen gas. It is very likely that not only the process of chemisorption is responsible for this effective gettering, but also the effects of physisorption and diffusion. This offers the far reaching possibility to have a material which is also suitable for storaging of noble gases, which does not exist so far.

Moreover, with the VPS technique, characterized by high deposition growth rates, it is possible to cover large surface areas of inner walls of vacuum vessels, beamline tubes, UHV measuring devices etc. with NEG material of sufficient thickness within reasonably short times, which is not possible so far with any other deposition technique. By doing so, one can combine two conditioning processes, namely the backing at elevated temperatures (several hours at 250 - 300 ^{0}C under high vacuum), necessary to obtain good UHV conditions, with the NEG activation procedure, for which also temperatures of at least 350 ^{0}C are needed. If one succeeds in constructing such NEG-coated pumping systems, where both steps of processing are performed simultaneously, a self-pumping system would be to hand, which does not need any power of its own (besides first-time activation) or additional expensive UHV pumps with power supplies (like ion-getter, Ti-sublimation or cryo-pumps).

2 EXPERIMENTAL DETAILS

Whereas one needs basic substances in powder form in case of metallic, metallic alloys, oxide or ceramic materials, C-nanostructures condense out of the vapor phase of a very hot (temperatures up to 15000 K) plasma of hydrocarbons under the influence of the generated metastable CH-radicals (methyl CH_3 or CH_2) in a CVD process. For both deposition techniques, the VPS coating with substances in powder form and the DC plasma-jet CVD process using gaseous precursors (e.g. CH_4, CO, H_2), a standard VPS machine, as shown in Figure 1, is used.

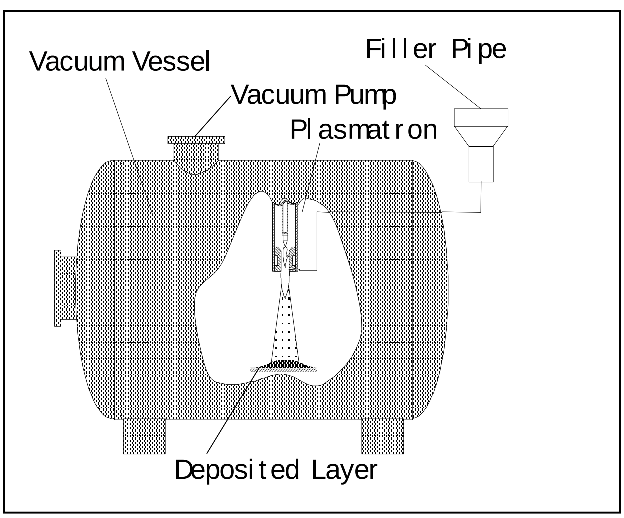

Figure 1: Schematic view of the vacuum-plasma-spraying (VPS) equipment for fabricating thin films of metal alloys or of carbon nanotubes.

The condensation and deposition processes are controlled by administering the required doses of cooling gas (helium), inert carrier gas (Ar, H2, N2 or mixtures thereof) and catalysts (like Fe, Ni or Co, in powder form). Of particular importance is maintaining the homogenious surface temperature of the substrate. But also all other process parameters, like pressure, H_2 concentration, gas temperature, torch-substrate distance, nozzle geometry, electrical power, gas-flow velocities, length of free plasma beam etc. are important for curving of C-platelets and formation of fulleren-like structures.

For obtaining high adhesion of the layers on the substrate, a suitable, very thin (<1µm) intermediate layer (intercalation compound) has to be formed, which can also be done by the VPS procedure. This interface can be a metal (e.g. W, Mo), metal alloy (like Ti-Zr, in particular), metal carbide, - nitride, - oxide or graphite. The intention is to optimize the production process with respect to most effective getter abilities, large-area deposition, high growth rates and adhesion of coating.

So far, first experiments for producing layers of an alloy of Zr-V-Fe by the VPS technology have been successfully performed. Generating coatings of fullerene-like carbon nanostructures, in particular of C-nanotubes, are expected to go very similar to the diamond film synthesis with the DC plasma-jet CVD process, for which great experience exists already in the laboratory of one of the authors (T.D.). Enthalpy-probe measurements for determining the gas-phase temperature and determination of its composition with the help of a mass spectrometer will be applied, very analogous to the case of diamond synthesis.

3 CHARACTERIZATION

Besides extensive measurements of partial outgassing rates on the basis of a highly accurate difference method, it is foreseen to determine the capacity and speed of gas sorption or gas-storage behaviour. An optimization of getter materials with respect to lower the activation temperature will also be performed. Moreover, the various possibilities of microcharacterization of the nanostructured surfaces and of the identification and measurement of the nanostructures and their morphology are planned. In particular, investigation with REM and UHV-STM/AFM systems, which are at the disposal of the collaboration, will be performed. Chemical characterization of getter surfaces will be done by using AES/XPS systems. Finally, a special beamline for measurements of photon-stimulated gas desorption of getter-coated sub-strates will be installed at the 1.5 GeV DELTA electron storage ring.

4 FUTURE APPLICATIONS

If one succeeds in producing these new kinds of highly effective getter layers on the basis of metals (mainly Ti-Zr) and on C-nanostructures, there will be far-reaching technical consequences, possibly also for the development of new products of commercial value. Besides the advantages in all areas of UHV generation, gas filtration, elimination of harmful chemicals, gas cleaning (also of isotopically enriched or radioactive gases), in particular, the very effective gas storage (mainly of hydrogen, but also of other active gases and of noble gases, like helium) will have numerous technical applications. In this respect, certainly the development of light-weight reservoirs for safe storage of large volumes of hydrogen gas, which is indispensable for energy storage in fully exhaust-free mobile vehicles ("hydrogen cars") is the most spectacular one. But such getters will play also an important role for constructing the next generation of very large high-energy particle accelerators, for sound and heat-loss vacuum insulation and in the development of batteries, fuel and solar cells and various micro-sensors.

REFERENCES

[1] T. W. Ebbesen, "Carbon Nanotubes, Preparation and Properties", CRC Press, Inc., 1997.