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A SETUP FOR PREPARATION OF GLASS-CARBON COATINGS ON TiO_2-Nb_2O_5 INTENDED FOR HIP JOINT PROSTHESES

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ABSTRACT

A setup for preparation of glass-carbon coatings on TiO_2-Nb_2O_5 ceramic materials, intended for implants for surgery of hip joint prostheses, is described. The setup described consists of vacuum tight ceramic chamber; programmable high temperature furnace and a system for controlled introduction of inert gas into the chamber.

The setup allows working with temperatures up to 1350^{o}C, controlled heating rates from 1 to 15^{o}C/min and chamber pressures down to 10^{-2} mmHg.

KEYWORDS: ceramics, glass- carbon coating, coating setup

1. Introduction

There exist several materials used for production of endoprostheses: biocompatible stainless steels, pure titan or its alloys, cobalt alloys, some polymers, inert bio-ceramics (e.g. Al_2O_3, ZrO_2), glass-carbon etc. All these have their strong and weak sides but all of them have one common quality, namely, they are biocompatible and comply with the ISO 5832-1 standard, stating the requirements for the materials used for bio-implants.

One, still unsolved, problem at the utilization of biomaterials for production of hip joint prostheses is their limited life span, which at present is less or around 20 years. Obviously, the increase of the life span of these materials is highly desirable, especially when these are intended for implantation in relatively young patients.

In a series of papers Jordanova et al. studied the synthesis of a composite material based on Rutile ceramics TiO_2-0.3-10 mol.% NbO_5 [1,2].

This composite material depends strongly on the structure and reactivity of the ceramics. The same authors found that the composite prepared by coating of the TiO_2-NbO_5 ceramics with amorphous carbon is not rejected from the human body and is thus suitable for preparation of human implantants.

The glass carbon is an amorphous modification of the crystalline carbon, not found in the nature. Compared to the crystalline carbon the amorphous (glass) carbon is lacking the characteristic for crystalline substances long distance ordering of the atoms. It should be noted, that the glass carbon modification is unstable and that it tends to transform to crystalline form, during coating process. Therefore, to obtain the desired glass carbon modification and keep it stable during the subsequent deposition on the TiO_2-Nb_2O_5 ceramic's surface, it is required a specialized setup for preparation of glass-carbon and its deposition on ceramics.

The aim of the present paper is to describe such experimental setup, designed and constructed in the BAS, and its tuning to working conditions.

2. Description of the equipment

The setup designed (see Fig.1), consists of: vacuum tight ceramic chamber, which is a ceramic tube with a 120mm diameter and length of 1500mm, furnished with vacuum tight flanges; programmable high temperature ceramic Silit-furnace, allowing reach temperatures up to 1350^{o}C and controlled heating rates from 1 to 15^{o}C/min; a vacuum system, allowing pressures down to 10^{-2} mmHg, consisting of rotational vacuum pump **2DS-2**; and a system for controlled introduction of inert gas into the chamber, made around needle-valve, allowing controllable gas flow through the chamber.

Fig.1. *Photo of the setup for deposition of glass carbon coatings.*

Obviously, the production of the desired carbon coating requires application of predetermined temperature regimes inside the chamber. Fig.2 shows the block-scheme of the furnace and the temperature controlling unit. The thermal block of the setup consists of: thermo-chamber with Silit heaters (1) and computer-model-selected positions for positioning of the specimens; temperature controller (2); power electronics block (3); and PC-based system for collecting and storing of data.

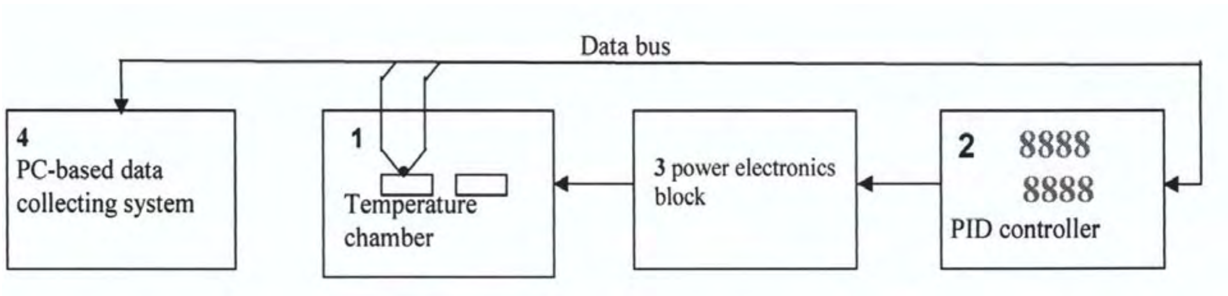

Fig. 2. *Block-scheme of the furnace, the temperature controlling and data collecting units.*

The data collecting procedure during the coating process is as follows: the specimens, to be coated with glass-carbon, are placed into the chamber (1) and are submitted to a pre-selected temperature regime, monitored by the PC-system (4). The desired temperature regime, i.e. the electric current to the Silit heater, is controlled by the controller (2) via the power electronics block (3). During the coating experiments carried out, it was found that a quality coating is obtained only if the temperature-time regime inside the furnace closely follows the one preset by the operator. This imposes certain requirements to the controller, such as ability to keep a linear interpolation between two preset points on the coordinate system temperature-time. The circumstances above predetermined the choice of the controller.

It is well known that the most suitable for the requirements imposed are the universal PID (proportional–integral–derivative) controllers. The PID controller calculates an "error" value as the difference between a measured process variable and a desired set-point. The controller attempts to minimize the error by adjusting the process control inputs. In the absence of knowledge of the underlying process, which is actually the case here, PID controllers are the best choice. However, for best performance of the controlled item, the parameters of the specific PID device used must be tuned according to the nature of the controlled system, in our case the furnace. Since the design of the furnace is unique the parameters controlled by PID would be also unique and would depend on the specific system.

In other words, a successful controlling of the temperature regimes desired would depend on successful tuning of the PID controller unit.

$$y(t) = P.x(t) + I.\left[\int_0^t x(t)dt\right] + D.\left(\frac{dx(t)}{dt}\right) \quad (1)$$

The PID control scheme is named after its three correcting terms, whose sum constitutes the manipulated variable *y(t)* see Eq.(1).

Here: ***P***, ***I*** and ***D*** are the tuning parameter termed, respectively; Proportional gain, Integral gain and Derivative gain.

For the specific case here, controlling of the temperature inside the furnace, an eight channel PID controller "**COMECO-RT1800**" type is used.

All eight channels of the controller allow pre-programmable temperature-time profiles of the type "initial temperature - plateau - final temperature" with linear temperature-time laws for heating and cooling.

The tuning of the PID controller above was carried by heating the furnace on its maximal power without back feed up of the controlling system. From the results obtained, the time constant τ, absolute delay *d* and amplification coefficient *K*, have been graphically determined. The tuning parameters of the controller were graphically calculated from the **S-**curve, *T= f(t)* shown in Fig.3, using the dependencies:

P=1,2.T/K.d; I=0,6.T/K.d2 D=0,6.T/K (2)

according to Ziegler-Nichols PID tuning Rules [3, 4]. The concrete temperature profile used for the tuning procedure and obtaining of the PID parameters for the COMECO-RT1800 controller is shown in Fig.4.

Fig. 5 shows a selection of glass-carbon coated ceramics produced with the setup described.

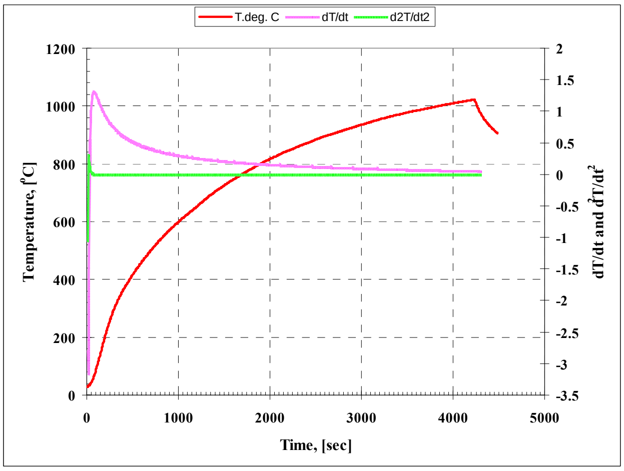

Fig. 3. *Temperature-time dependencies used for tuning of the* ***COMECO-RT1800 PID*** *controller.*

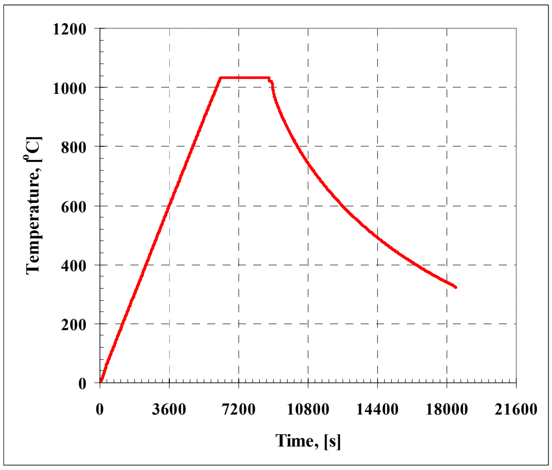

Fig. 4. *Temperature profile used for the tuning procedure and obtaining the parameters for the COMECO-RT1800 PID controller.*

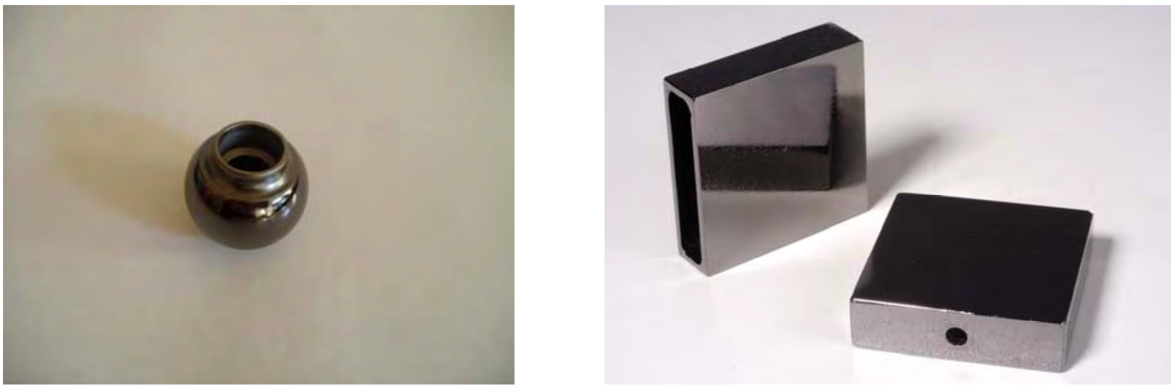

Fig. 5. *A selection of glass-carbon coated ceramics produced with the setup described.*

3. Conclusion

Based on the experience gained by using the setup for preparation and deposition of glass-carbon coatings on ceramics it could be concluded that the controller, selected and used in the setup, allows:

Maximum flexibility at preprogramming of different temperature regimes depending on the coating applied and the substrate ceramics used.

The obtained PID parameters keep the controlled parameters well within the desired limits, required for production of high quality coatings, respectively, for a successful practical application of the glass-carbon coating setup.

The designed and constructed setup allows development of new technologies for coating of different materials.

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