



# Deposition of W/a-C:H:Zr and W/a-C:H:W multilayer coatings on substrate made of porous graphite by arc – Electron beam hybrid method



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## ABSTRACT

The W/a-C:H:Zr and W/a-C:H:W multilayer coatings were prepared on a porous graphite substrate by hybrid method combines of arc evaporation and electron beam evaporation processes (Arc-EB). The tungsten interlayer was deposited first to enhance coating adhesion. Phase composition of coatings was examined by Raman spectroscopy and X-ray diffraction. The measurement of hardness and Young's modulus has indicated a significant increase in mechanical properties after the deposition of upper layer. Microstructure was investigated by SEM and TEM revealing columnar morphology and lack of porosity in both samples. The a-C:H:Zr coating was characterized by lower wettability by liquid copper than a-C:H:W coating and pure tungsten interlayer.

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## 1. Introduction

Graphite is characterized by high thermal conductivity, satisfactory strength at high temperatures and low thermal expansion, and as such is widely used in various industrial applications. The disadvantage of graphite is its high porosity, inhomogeneity and easy oxidation [1]. The graphite plate moulds are covered during casting process with the solidifying metal. This reduces the life of moulds and increases the rate of production rejects.

A concept of prolonged mould life consists in improvement of the mould working surface through application of special coatings. The diamond and DLC coatings seem to offer the best solution for protection of the mould working surface due to their high conductivity, very high smoothness and lack of porosity, which guarantees that liquid metal shall not penetrate inside the coating [2].

There is not much information about the possibility of coating the graphite substrate by diamond layers [3,4,5]. The process is very difficult due to nearly no solubility of carbon in the graphite substrate. Some data indicate that by introducing an intermediate layer, the applicability of DLC coatings onto different substrates is considerably enhanced [6].

In addition, the deposition of appropriate interlayer can successfully reduce the residual stress in coatings and improve the adhesion [6,7]. Another solution involves using of carbide-forming metallic elements as a target materials. Such coatings are often denoted in literature as

Me-DLC coatings [8,9,10,11,12,13]. So far, the majority of these coatings have been deposited by reactive magnetron sputtering system. Coatings are prepared in argon-hydrocarbon gas mixtures using metal or metal carbide. Recently, the target materials considered in all the research works on Me-DLC coatings are W, Zr, WC, Ti, Cr.

The aim of the present paper is to report on carbon coatings prepared by hybrid method combines of arc evaporation and electron beam evaporation processes (Arc-EB) with the use of two different target materials, i.e. Zr and W.

## 2. Experimental details

The W/a-C:H:Zr and W/a-C:H:W multilayer coatings were produced on samples made of isostatically pressed graphite material, whose characteristics are shown in Table 1. The applied material is used for the manufacture of high-temperature resistant products operating in the metallurgical industry, such as crucibles, crystallizers, permanent moulds and dies, and mould assemblies for continuous casting.

Developed Arc-EB hybrid surface treatment technology has been implemented using the hybrid multisource device produced by the Institute for Sustainable Technologies – National Research Institute in Radom (Poland) [14]. The configuration of Arc-EB hybrid surface treatment technology is shown in Fig. 1. The device has been equipped with a  $\phi = 80$  mm cathode arc source and a 60 kW electron gun with the dynamic electron beam deflection circuit and steering system. The device is also equipped with modern, reliable power systems, substrate polarization system, multichannel process gases dosing system as well as the

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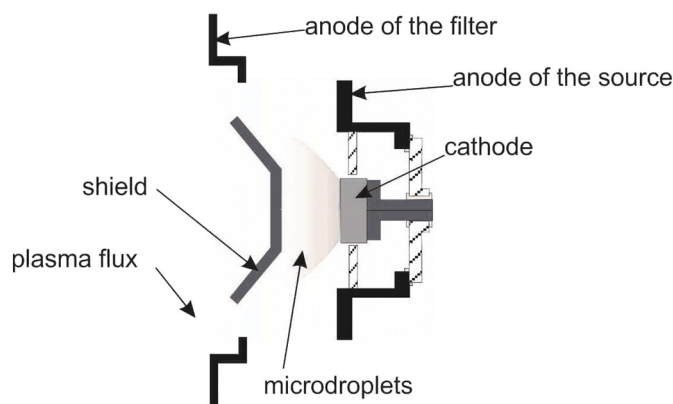
E-mail address: [pauzaw@agh.edu.pl](mailto:pauzaw@agh.edu.pl) (P. Zawadzka).

**Table 1**  
Parameters of the graphite used as a substrate.

Grain size	10 $\mu\text{m}$
Apparent density	1.83 $\text{g}/\text{cm}^3$
Flexural strength	60 MPa
Open porosity	10%

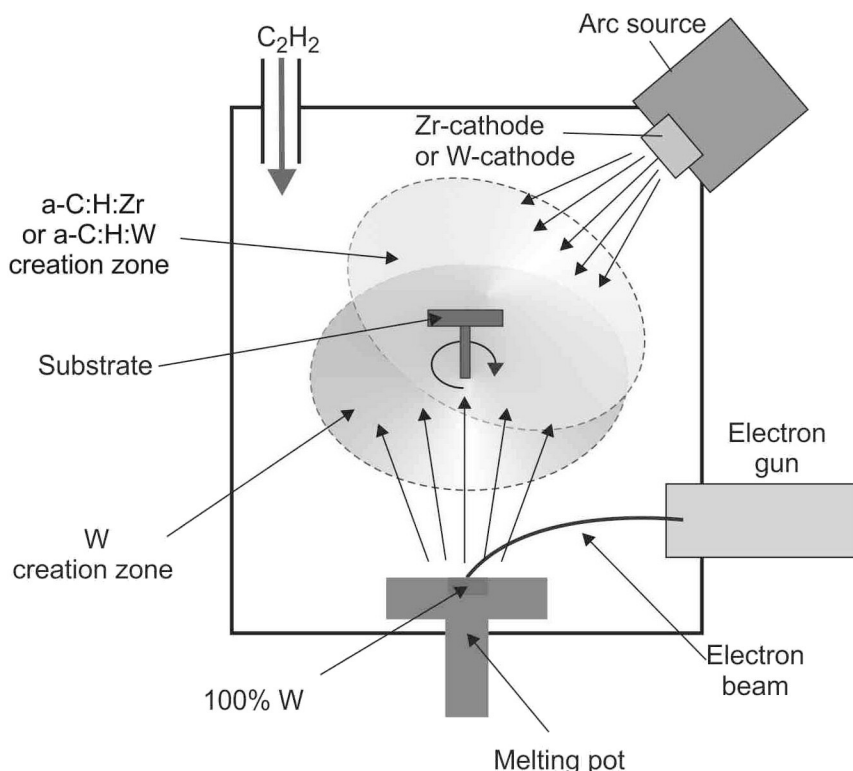
systems of monitoring and measuring substrate temperature and atmospheric gas pressure. The arc source is equipped with electrostatic separation system (Fig. 2) which allows separation of the droplet phase. The operation of electrostatic separation systems consists in using a shield placed between the plasma arc source and the substrate, perpendicularly or at an angle to the plasma and flux of microdroplets. The shield is isolated from the arc source electrodes and properly polarized. Simultaneous application of a positive potential on the source anode a filter anode in the relation of the cathode potential compresses the ion flux. As a result, the ion flux avoids the filter and reaches the substrate surface [15].

Fig. 3a shows a schematic diagram of the two-step process for producing samples. The first step includes the deposition process of tungsten interlayer on graphite substrate by electron beam (EB) technique. The interlayer of tungsten sticking the pores into the graphite substrate and as result build the solid, tight surface. The operation of the deposition of tungsten interlayer consists of the two main stages: (i)  $\text{Ti}^+$  ion etching for substrate cleaning by Arc method, and (ii) deposition of W – interlayer by EB technique. The second step of hybrid surface treatment includes the deposition process of metal hydrocarbon (Me-DLC) coating by Arc technique. This process consists of the four main stages: (i)  $\text{Me}^+$  ion etching for substrate cleaning by Arc-PVD method, (ii) thin Me interlayer coating deposition, (iii) cooling the substrate in high vacuum to a temperature of the Me-DLC coating deposition, and (iv) the deposition of Me-DLC coating using  $\text{C}_2\text{H}_2$  as a carbon source. During this process as metals sources the targets made of Zr and W were



**Fig. 2.** The construction scheme of electrostatic separation system of microdroplets.

used. The technological parameters of individual steps of Arc-EB hybrid surface treatment technology were selected based on the state of the art [16] and are presenting in Tables 2 and 3. The main parameters which are controlled during the Arc process include the substrate bias voltage, the source current which supplies the plasma source and current coil which shapes the magnetic field necessary for positioning arc discharges at the cathode. Substrate bias voltage determines the energy of ions reaching to the coated substrate. It has a decisive effect on substrate temperature during the process and next on the adhesion of the coating to the substrate. The main parameter which is controlled during the EB process is beam current. Additionally, both Arc and EB processes, are controlled by the pressure and flow of working gas and by the time and temperature of the process. For the sake of comparison, samples without the interlayer deposited on graphite substrate were also prepared. Fig. 3b shows schematically the cross-section of samples investigated in this study.



**Fig. 1.** The scheme of hybrid Arc-EB PVD technology.

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