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# Characteristic STATE of substrate and coatings interface formed by Impulse Plasma Deposition method

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ABSTRACT

This paper presents a study of the structural, phase and chemical states of coating–substrate interface, formed during the Impulse Plasma Deposition method, operating under conditions of pulsing pressure. The plasma process was carried out using a gas valve operating in a pulsing manner, controlling the pressure between threshold values — forming and extinguishing the plasma. We assumed that using pulsing pressure would significantly enhance the plasma particles' energy, preserving their momentum by reducing the gas accumulation at the front of plasmoid. We expected that impinging by enhanced plasma species could form a specific state of the coating and substrate interface. The titanium nitride coatings deposited on Fe substrates were selected as the model materials. The secondary ion mass spectrometry method was used to study the chemical state of the transition zone of the interface. A complex composition of the interface was observed, suggesting a shallow implantation of titanium atoms. Cross sectional transmission electron microscopy revealed local areas of epitaxial growth.

## 1. Introduction

The Impulse Plasma Deposition method (IPD) was developed in the 80s at the Warsaw University of Technology [1,2]. The standard version of the IPD method operates in a pulsating manner, generating plasma by the coaxial accelerator installed in a vacuum chamber and driven by spark ignitron [3] (see Fig. 1a). The $\sim 10^2\,\mu F$ battery of capacitors, charged to $10^3$–$10^4$ V voltage, is used as the electric energy source. During the discharge, a single, radially symmetric current sheet is initiated in the inter-electrode space of the coaxial accelerator and accelerated by Ampere force toward the accelerator outlet [4]. Plasma is excited by this specific arc discharge and forms a paraboloidal shape, called as plasmoid [5]. This plasma is highly ionized [6,7], so it provides a favorable environment for the nucleation of the metastable phases. At first, the size of critical nucleus is reduced if the nucleus is electrically charged [8,9]. At second, the energy exchanged during the inelastic collision of electrons and clusters formed in plasma can be used to change the hybridization of valence bands instead of dissipation for exciting the phonons, so the high-energetic, monotropic phases can be nucleated [10,11]. This feature resulted in the IPD method's use for the synthesis of various coatings: DLC [12,13], boron nitride [14], titanium nitride [15], metallic [16], etc. It should be mentioned that the synthesis occurs at substrates not intentionally heated.

In 2010, we proposed a modification of the IPD method which eliminates the spark ignitron from the electric circuit of a plasma accelerator [17] (see Fig. 1b). In this modification, we used a fast-pulsed gas valve, installed at coaxial accelerator to control plasma generation. The modified IPD process operated in condition of a periodically pulsing pressure, in the range of threshold values: $10^0$ Pa (pressure initiating the discharge) – $10^{-4}$ (no discharge). Titanium nitride (TiN) coatings, deposited by the modified version of IPD method, were distinguished by the extraordinary wear – resistance [17]. In this reported study, we tested the durability of the coating by TIN cutting tools during the turning of a roller made of 41CrAlMo7 steel for nitriding. During the test, we achieved a 16 – fold increase of durability, which is a remarkable result for TiN coatings. As literature reports, durability increases from tens to a few hundred percent are usually observed [18–22] depending of the test method, type of cutting tool, tool material and coating synthesis method. In the case of the IPD method, operating in its standard version, the durability improvement was reported as 200–300% [23]. This manuscript presents the study in which we sought to answer what caused such a high wear resistance of cutting

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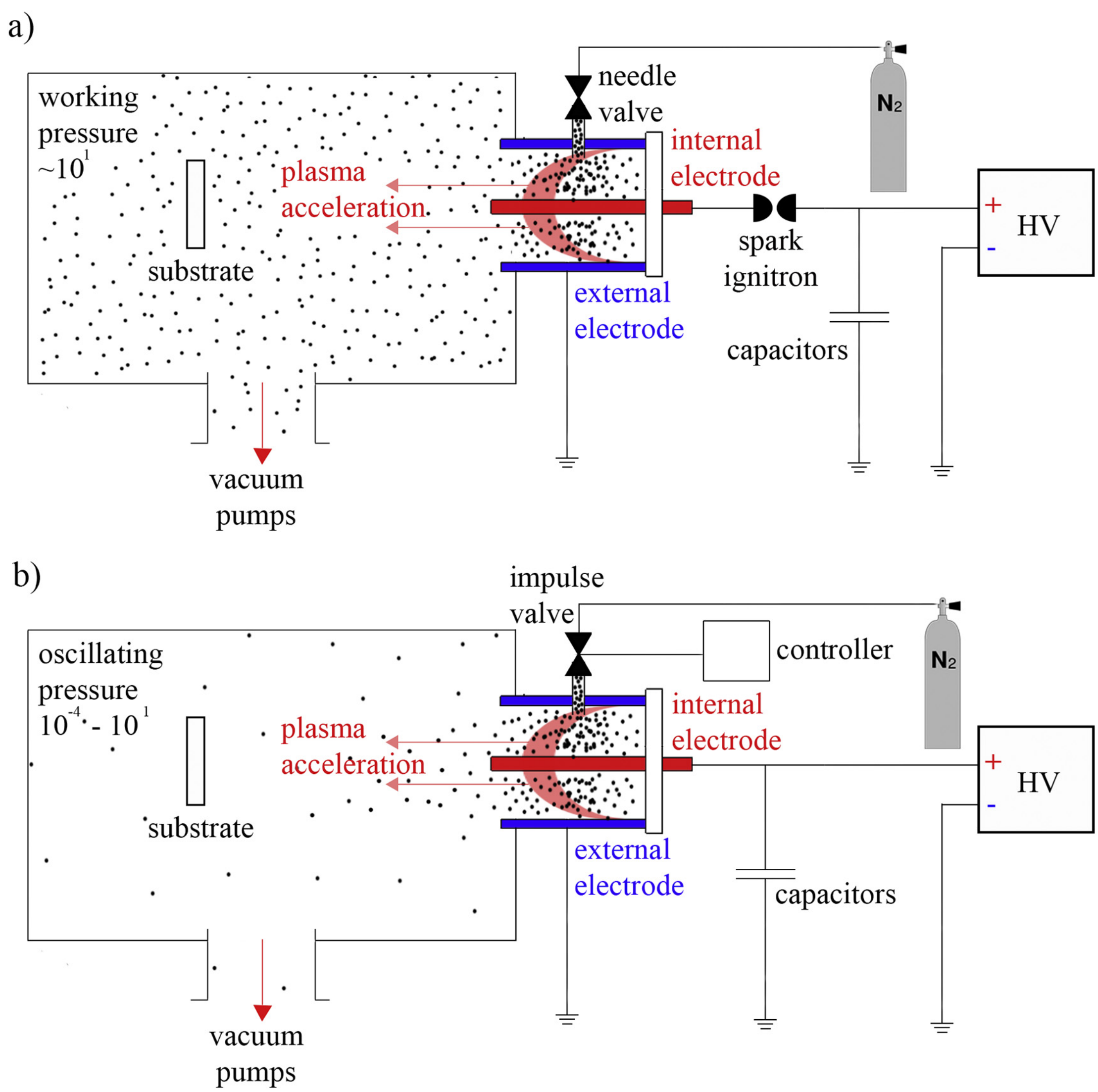


**Fig. 1.** Diagrams illustrating setup of apparatus: a) standard IPD method, b) modified IPD method.

tools coated by the TiN. The reason behind taking a closer look at the coating – substrate interface, formed during the synthesis process with pulsing pressure, is the characteristics of pulse plasma we presented in one of our latest papers [24]. We registered a highly-ionized spectrum of a plasma generated under this condition with constituents observed before only during processes using much higher energies [6,7]. Consequently, we observed an excited species in plasma spectrum, coming from surface sputtering, measured at the substrate surface when exposed to plasma stream [25]. We concluded that the condition of generating the plasma by gas pulse is more favorable to preserve the plasma particles kinetic energy. This conclusion corresponds well with computational results which describe the spreading of plasma pulses in an ambient pressure characteristic for the standard version of IPD [5]. A characteristic feature of plasma propagation is decelerating its motion by gas molecules accumulation before the head of plasmoid. From the modified version of the IPD process, we expected a reduction of this effect. OES study, performed by Walkowicz, revealed that the head of the plasmoid is enriched by ions originating from working gas excitation [26], whereas in the modified IPD method, the head of the plasmoid is enriched by “metallic” ions [24]. Taking into account the presented results, we expected an interaction of plasma with the surface occurring on a different scale and with a different mechanism, so forming the specific state of boundary region of the coating and the substrate, which may be the cause of stunning anti-wear properties of reported cutting tools. In this work, our goal was to investigate the structural, phase and chemical state of this boundary region to find out if its state is somehow linked with extraordinary adhesion of coatings deposited in condition of pulsing gas pressure. The secondary ion mass spectrometry was used to investigate chemical profiles of interfacial region. Transmission electron microscopy was utilized to revealed structural and phase state of that region. Mechanical properties were measured on the cross-section of coating/substrate system.

## 2. Experimental

### 2.1. Apparatus setup

The setup of the experimental apparatus is presented in Fig. 1b. Plasma is generated in coaxial accelerator by the high-current discharge between electrodes. As a source of electric energy, the 150 μF battery of capacitors charged up to 3 and 4 kV by HV supply was used. A detailed mechanism of plasma initiation, acceleration and spreading in the vacuum chamber was described elsewhere [5,27]. In our experiment, by using different voltage, 3 and 4 kV, we tried to compare how the state of interfacial region would evolve under exposure on different plasma streams. In this version of the IPD method, the discharge was triggered by a gas dose, distributed by a pulse valve to the inter-electrode space of the plasma accelerator. The time of opening the valve was optimized

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