Effect of the substrate temperature and bias voltage on the properties of ZrO$_2$/Al$_2$O$_3$ coatings produced by pulsed magnetron sputtering and filtered vacuum arc deposition

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ABSTRACT

The influence of deposition parameters (substrate temperature and bias voltage) on the properties of ZrO$_2$-Al$_2$O$_3$ coatings (composition, microstructure, hardness, adhesion, and wear coefficient) was studied. The properties of the coatings that were produced by Pulsed-DC Magnetron Sputtering (PMS) were compared to those prepared by Filtered Vacuum Arc Deposition (FVAD). The coatings were deposited on Si and WC substrates at temperatures, $T_{s}$ of 25–500 °C and biases, $V_b$, floating and down to -200 V. The PMS deposition rate was 0.15±0.01 nm/s, while that of FVAD was 10±2 nm/s. X-ray diffraction indicated that PMS coatings had a stabilized cubic-ZrO$_2$ structure at all $T_s$ when $V_b$ ≤ -50 V or a tetragonal-ZrO$_2$ structure at $V_b$ ≥ -100 V. FVAD coatings had a stabilized tetragonal-ZrO$_2$ structure at all $T_s$ and $V_b$. Coatings deposited on unheated substrates by both PMS and FVAD at floating potential had low hardness, <10 GPa. However, increasing $T_s$ to 370 °C increased the hardness of both PMS and FVAD coatings to 16±1 GPa. In the FVAD coatings, a further increase of $T_s$ to 500°C raised the hardness to 22±1 GPa. The maximum hardness, 25.9±2.6 GPa, was achieved at $V_b$ = -150 V (PMS coatings, unheated substrate) and was partly due to an increase in compressive stress (1066 MPa). The coating adhesion increased with $T_s$, reaching more than 100 N at $T_s$ = 500°C (FVAD coatings, $V_b$ = -50 V).

I. INTRODUCTION

Vacuum Arc Deposition (VAD) and Magnetron Sputtering (MS) are leading techniques for depositing metallurgical coatings. VAD is a high-current, low-voltage technique characterized by high plasma ionization, high energy of depositing particles (15–170 eV), and high deposition rates (~10$^6$–10$^7$ µm/min [1], but even up to more than 400 µm/s [2]). VAD tends to form dense coatings that adhere well to the substrate. However, one of the major disadvantages of VAD is the formation of liquid droplets of the cathode material, usually referred to as macroparticles (MPs), which, if incorporated into the coating, can degrade its properties [3-4]. The MPs can be ‘filtered’ out by magnetically diverting the plasma beam around an obstacle that blocks them [5]. This variation is called Filtered Vacuum Arc Deposition (FVAD).

In contrast, MS is a high-voltage, low-current technique characterized by long-range process stability, high flexibility in coating composition, and high purity of the deposited film [6]. MS is performed in a low-pressure gas atmosphere (usually argon, 0.1–10 Pa), unlike VAD, which can be carried out in vacuum (10$^{-3}$ Pa). MS has a lower deposition rate (~10$^{-2}$–10$^{-1}$ µm/min) and a lower ionization fraction (~5%) than VAD. The deposition of dielectric coatings, e.g. oxides, can be difficult due to “poisoning” [7], which limits the use of standard direct current (DC) MS. Traditionally, radio-frequency (RF) power supplies were used to overcome this problem. However, RF-MS has a low deposition rate (10$^{-2}$–10$^{-3}$ µm/min) and is complex to scale up, which limits its industrial application [8]. Pulsed Magnetron Sputtering (PMS) enables deposition of dielectric coatings at deposition rates approaching those obtained for the deposition of pure metal, while eliminating arcs [8]. PMS uses DC pulses in the mid-frequency range (10$^3$–10$^4$ kHz) and a duty cycle (the ratio of “pulse-on” time to the total period of the signal) of 50–70% [8-9]. FVAD has an advantage for the deposition of oxides, as no cathode poisoning takes place in the high erosion rate conditions of the vacuum arc [10].

Among the oxide coatings, zirconium oxide (ZrO$_2$) and Aluminium oxide (Al$_2$O$_3$) have attracted much attention due to high-temperature applications of ZrO$_2$ [11] and high wear resistance applications of Al$_2$O$_3$ [12]. Moreover, it is well known [13-15] that...
the addition of $\text{Al}_2\text{O}_3$ stabilizes the $\text{ZrO}_2$ coating in its high-temperature phases ($\text{ZrO}_2$ exhibits, at ambient pressure, three phases: monoclinic phase, m-$\text{ZrO}_2$, which is stable from room temperature up to 1170 °C, and two high-temperature phases, tetragonal, t-$\text{ZrO}_2$, and cubic, c-$\text{ZrO}_2$ [14]). On the other hand, in $\text{ZrO}_2$-toughened $\text{Al}_2\text{O}_3$ composite, small amounts of $\text{ZrO}_2$ can improve the mechanical strength and fracture toughness compared with monolithic $\text{Al}_2\text{O}_3$ [15]. Furthermore, the $\text{ZrO}_2/\text{Al}_2\text{O}_3$ system has almost no mutual solubility and therefore it offers the possibility of forming as a hard or super-hard nanocomposite [16].

$\text{ZrO}_2/\text{Al}_2\text{O}_3$ coatings have been deposited by both VAD [17-18] and PMS [13,19-23] as well as by DC-MS [24-25], RF-MS [26-28] and other techniques such as Chemical Vapor Deposition [29], Pulsed Laser Deposition [30-32], electron-beam deposition [33], and sol gel [34] and spray techniques [35-37]. Most of the early works deposited the $\text{ZrO}_2/\text{Al}_2\text{O}_3$ coatings as nanolaminate systems [21,27,38-41] and focused on t-$\text{ZrO}_2$ phase stabilization using alternating layers of the alumina phase. For example, in nanolaminate $\text{ZrO}_2/\text{Al}_2\text{O}_3$ coatings, the formation of the t-$\text{ZrO}_2$ phase is controlled by the $\text{Al}_2\text{O}_3$ and $\text{ZrO}_2$ layer thickness [24,40] (in these types of coatings, the $\text{Al}_2\text{O}_3$ layers are amorphous). These coatings showed excellent phase stability during annealing at $T = 600 \degree \text{C}$ for 56 hours [40].

Nanocomposite $\text{Al-Zr-O}$ coatings exhibited a t-$\text{ZrO}_2/\text{a-Al}_2\text{O}_3$ (a=amorphous) or c-$\text{ZrO}_2/\text{a-Al}_2\text{O}_3$ (c = cubic) structure, in contrast to pure m-$\text{ZrO}_2$ coatings [19-20,23,25-26]. The addition of $\text{Al}_2\text{O}_3$ was accompanied by an increase in hardness [20,22] or in resistance to cracking [19]. With high $\text{Al}_2\text{O}_3$ content, the coatings became amorphous and their hardness decreased [20,22].

The deposition conditions of PVD techniques have a strong influence on the structures and properties of coatings [42]. There are some indications regarding the influence of the deposition temperature and sputtering method on the $\text{ZrO}_2/\text{Al}_2\text{O}_3$ system [25-26]. However, the influence of the substrate bias on the coating structures, as well as, the influence of the deposition parameters on the hardness and wear behaviour, was not published so far. The objective of this study was to determine the influence of the substrate temperature ($T_s$) and voltage applied to the substrate ($V_s$) on the structure, hardness and tribological properties of $\text{ZrO}_2/\text{Al}_2\text{O}_3$ coatings deposited by the PMS and FVAD techniques.

II. EXPERIMENTAL DETAILS

Pulsed magnetron sputtering

The PMS system is illustrated in Fig. 1a. The chamber was equipped with two MS units, one mounted with a 51 mm aluminium target (99.999% purity) and the second with a 51 mm zirconium target (99.9% purity). Each sputtering unit was powered by a pulsed-DC generator and had its own oxygen inlet. The substrates used in this work were 300 µm thick (001) crystalline silicon wafers, 51 mm in diameter, and WC-Co cutting inserts polished to a mirror-like finish. Both were cleaned in acetone using an ultrasonic bath before being placed on a heatable and rotatable substrate holder.

Before deposition, the chamber was evacuated using turbomolecular and rotary pumps
to a base pressure of $1\times10^{-4}$ Pa, after which a mixture of argon (99.999% purity) and oxygen (99.999% purity) was introduced. The oxygen flow rate was 20% of the total flow of 100 sccm. All depositions were performed in the power controlled mode, with a pulse repetition rate of 350 kHz and a duty cycle of 55%. The composition of ZrO$_2$/Al$_2$O$_3$ coatings was controlled using a constant Zr target power of 200 W and an Al target power of 50 W. The substrate temperature was varied between $T_s<100$°C (RT, no external heating) and 370°C using a halogen lamp heater. During deposition, the substrate holder was kept at floating potential or a negative bias voltage of up to $-200$ V, provided by an RF (13.56 MHz) power supply [43]. The actual bias voltage was measured by the RF power supply and was regulated by changing the power between 0-40 W. The substrate holder was rotated at 1 RPM. The deposition time was 120 minutes for the Si wafers and 240 minutes for the WC-Co cutting inserts.

**Vacuum arc deposition**

A triple-cathode VAD system with a 1/8 torus magnetic duct for MP filtering, described previously [17], is illustrated in Fig. 1b. Two cathodes, Al and Zr, were mounted in two of three holes spaced equally along the circumference of a 100-mm diameter circle centred on the system axis (Fig. 1c) [44]. Both cathodes had a frustum cone shape with front and back base diameters of 49 and 54 mm, respectively, and a height of 15 mm. Each cathode had its own welding power supply and trigger mechanism. The plasma jet produced by the cathode-spots entered the deposition chamber via 54-mm diameter holes in the anode, coaxial with each cathode. An axial DC magnetic field, $B=12$ mT, was used to confine the cathode spot motion on the front cathode face, to screen the MPs, and to guide the plasma flow from the cathode spots via the anode aperture to the substrate holder [5,45]. Silicon wafer and WC-Co cutting insert substrates, identical to the ones used in the PMS deposition, were mounted on a substrate holder located at an axial distance of 150 mm from the end flange to which the 1/8 torus filter was connected, that is, approximately midway between coils 4 and 5 in Fig. 1b.

Before arc ignition, the vacuum chamber was pumped down to an initial residual pressure lower than 0.01 Pa. The arcs were operated under an oxygen/argon background pressure of 1.06 Pa (80 vol.% O$_2$). The coatings were deposited while simultaneously operating the Al and Zr cathodes with arc currents of 75 and 100 A, respectively. The substrate holder was heated before deposition to a temperature that varied between RT and 500 °C and was biased between and $-50$ and $-200$ V, powered by a DC power supply. During deposition, the substrate temperature sometimes increased due to the surface bombardment by energetic ion flux; however, this increase was not more than 20°C. The deposition time was 6 minutes.

**Characterization of coatings**

After deposition, the coating cross-sectional morphology was studied using high resolution scanning electron microscopy (HRSEM, JOEL JSM-7400D). The coating thickness was measured using a white-light profilometer as well as by cross-sectional SEM images. The structure of the coatings was analysed by X-ray Diffraction (XRD) using a Rigaku D Max 2000 diffractometer with Cu-Kα radiation. The grain size was calculated from the XRD spectra using the Williamson-Hall method [46]. The coating composition was studied with X-ray Photoelectron Spectroscopy (XPS) using an ESCALAB 250 spectrometer with an Al X-ray source and a monochromator. The coatings were also studied using high-resolution transmission electron microscopy (HRTEM) with a JEOL JEM-2100F microscope and the coating structure was studied using selected area diffraction (SAD). The hardness, $H$, of the layers was measured using a diamond Vickers indenter with a load of 5 mN. Residual stress, $\sigma$, was analysed by the curvature method using the Stoney equation [47]. The coating adhesion was assessed by a scratch test (CSM-REVETEST) using a diamond indenter whose radius of curvature was 0.2 mm. Each scratch was 5 mm long at constant load, after which the scratch track was examined by optical microscopy. The scratch load was increased until the first appearance of coating failure (defined as substrate exposure), which determined the critical load, $L_c$. The wear coefficient was studied using a rotating sliding wear test (CSM-TRIBOMETER) against a 3.0-mm WC ball in ambient air. The linear sliding speed was 0.1 m/s and the load was 1 N. The sliding distance was 300 m at a sliding diameter of 2 mm. After the wear test, the cross section of the wear track was measured using a white light profilometer and was used to calculate the wear coefficient, $W=V/ Ls$, where $V$ is the removed volume, $L$ is the load, and $s$ is the sliding distance [48].

**III. RESULTS**

FVAD coatings had a very low macro-particle concentration and a deposition rate of 10 ± 2 nm/s. The coatings were electrically insulating and transparent; multiple-interference fringes were visible where the coating thickness was not uniform. The coatings contained 70 at.% ZrO$_2$ and 30 at.% Al$_2$O$_3$, without metallic phases [17]. PMS coatings were smooth and uniform (on a wafer diameter of 50 mm) and the deposition rate was 0.15±0.01 nm/s. The coatings contained 83 at.% ZrO$_2$ and 17 at.% Al$_2$O$_3$ without metallic phases [22]. The coatings obtained using both techniques were found by XPS to be homogeneous in depth [17,22].

**Influence of substrate temperature**

All PMS coatings presented here were
deposited at floating $V_b$ and all FVAD coatings were deposited at $V_b = -50$ V. The coating structure changed with $T_d$ (Fig. 2).

Figure 2 XRD patterns at various deposition temperatures for PMS and FVAD coatings. c cubic; t tetragonal.

PMS coatings deposited at $T_d = RT$ had c-ZrO$_2$ structure, with (200) preferred orientation. Increasing $T_d$ to 200°C did not change the crystallographic orientation. However, coatings deposited at $T_d = 370$ °C had (111) orientation with some small amount of the t-ZrO$_2$ phase (peak at 34.2°). We believe that the t-ZrO$_2$ phase, as revealed by the 34.2° peak, was present at $T_d < 370$ °C and was masked by the (200) peak. The grain size of the PMS coatings calculated from the XRD analysis was 20±5 nm, regardless of $T_d$. Similar zirconia grain size values were measured by HRTEM (Figure 3a,b).

FVAD coatings deposited at $T_d$=RT had a t-ZrO$_2$ structure with (101) orientation (Fig. 2). However, the peak width and intensity indicate low crystallinity. Increasing $T_d$ to 250°C improved the coating crystallinity while maintaining its t-ZrO$_2$ (101) orientation. No major changes in the XRD pattern

Figure 3 A) TEM photograph of PMS coating deposited at 370°C and floating $V_b$. Inset - SAD pattern showing c-ZrO$_2$ structure. B) TEM high resolution photograph showing the ZrO$_2$ grains in the coatings.
were observed at $T_d > 250$ °C. It is difficult to distinguish between the t-ZrO$_2$ and c-ZrO$_2$ structures by XRD as there is a large overlap in their diffraction patterns. However, HRTEM analysis and the SAD pattern (Figure 4) showed the presence of t-ZrO$_2$ nano-grains with a size of ~14 nm in coatings deposited at elevated temperatures. In both PMS and FVAD coatings, no Al$_2$O$_3$ crystalline phase was identified in the XRD analysis. Figure 5 shows the influence of $T_d$ on the coating hardness. FVAD and PMS coatings deposited at $T_d = RT$ had low values of hardness of 10.4±0.2 GPa and 5.0±0.6 GPa, respectively. The coating hardness increased with $T_d$ and reached maximum values of 22.5±0.9 GPa ($T_d = 500$ °C) and 16.1±0.9 GPa ($T_d = 370$ °C) in FVAD and PMS coatings, respectively. All PMS coatings showed low tensile residual stress, below 100 MPa, regardless of the deposition temperature and without any clear trend. Similar coating characteristics were observed by Koski et al. [49], in ZrO$_2$ coatings deposited without substrate bias or at relatively high working pressure (~4 Pa). It was impossible to evaluate the residual stress of FVAD coatings due to their low lateral homogeneity.

**Figure 4** HRTEM bright field photograph of FVAD coating deposited at 400 °C and $V_b = -50$ V. The lines indicate a t-ZrO$_2$ grain area, as was found in the SAD pattern (inset).

**Figure 5** Coating hardness as a function of the deposition temperature (PMS coatings- floating $V_b$; FVAD coatings- $V_b = -50$ V). Vickers indenter; 5 mN load.

**Figure 6** XRD patterns for various values of the substrate bias for PMS and FVAD coatings. All coatings deposited without external heating ($T_d = RT$). c – cubic; t – tetragonal.

All coatings presented here were deposited without external heating. Applying $V_b = -50$ V (Fig. 6a) changed the PMS coating orientation from c-ZrO$_2$ (200) with floating bias to c-ZrO$_2$ (111). The
coatings deposited at \( V_b = -50 \text{V} (T_d=\text{RT}) \) and at \( T_d = 370^\circ\text{C} \) \((V_b = \text{floating})\) (Fig. 2a) had identical structures and orientations. This indicates that the energy delivered to the surface is similar in both cases. At higher \( V_b \), the coating structure changed to a mixture of c- and t-ZrO\(_2\) \((V_b = -100 \text{V})\) and then to t-ZrO\(_2\) \((V_b = -150 \text{V})\). Coatings deposited at \( V_b = -200 \text{V} \) were cracked after deposition, probably due to structure changes and/or high compressive stress. FVAD coatings deposited with \( V_b = -50 \text{V} \) had a t-ZrO\(_2\) structure with \((101)\) orientation. With increases in \( V_b \), the coating orientation changed to \((112)\) and \((200)\). Again, no Al\(_2\)O\(_3\) peaks were identified in the XRD spectrum of both PMS and FVAD coatings.

**Adhesion and wear coefficient**

This section presents the influence of the deposition parameters on the adhesion and wear behaviour of Zr-Al-O coatings deposited on WC-Co substrates. The coating thickness was 3 \( \mu \text{m} \) and their structure was identical to the structure measured on Si wafers and presented above. A large variation of \( L_c \) as a function of the deposition temperature was found (Figure 8). While PMS coatings deposited at RT and floating potential failed under a load of 5 N, FVAD coating deposited at \( T_d = 500^\circ\text{C} \) and \( V_b = -50 \text{V} \) did not fail under a load of 100 N, which was the highest load tested in this study. For the PMS coatings, the highest \( L_c = 40 \text{N} \) was found for coatings deposited at \( T_d = 370^\circ\text{C} \) and \( V_b = -150 \text{V} \).

Table 1 presents the wear coefficient of PMS and FVAD coatings under constant \( V_b = -50 \text{V} \). It was found that the wear coefficients of the PMS coatings deposited without external heating and at \( T_d = 250^\circ\text{C} \) was \(3.2 - 3.7 \times 10^{-6} \text{mm}^3/\text{Nm}\), while \( W \) decreased to \(1.9 \times 10^{-6} \text{mm}^3/\text{Nm}\) at \( T_d = 500^\circ\text{C} \). FVAD coatings had wear coefficient of \( W = 1.9 \times 10^{-6} \text{mm}^3/\text{Nm}\) at \( T_d = 250^\circ\text{C} \) and the lowest wear coefficient, in this study of \( W = 0.6 \times 10^{-6} \text{mm}^3/\text{Nm}\) at \( T_d = 500^\circ\text{C} \).

**Table 1** The wear coefficient of Al-Zr-O coating deposited on WC-Co substrates deposited at different deposition temperatures. All coatings were deposited under \( V_b = -50 \text{V} \).

<table>
<thead>
<tr>
<th>Method</th>
<th>( T_d ) [(^\circ\text{C})]</th>
<th>Wear coefficient ( \times 10^{-6} \text{[mm}^3/\text{Nm]} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>PMS</td>
<td>No external heating</td>
<td>3.2 ( \pm ) 0.3</td>
</tr>
<tr>
<td>PMS</td>
<td>250</td>
<td>3.7 ( \pm ) 0.3</td>
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<tr>
<td>FVAD</td>
<td>250</td>
<td>1.9 ( \pm ) 0.2</td>
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<tr>
<td>PMS</td>
<td>370</td>
<td>1.7 ( \pm ) 0.2</td>
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<tr>
<td>FVAD</td>
<td>500</td>
<td>0.6 ( \pm ) 0.1</td>
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IV. DISCUSSION

The ZrO₂ to Al₂O₃ content ratio is the main parameter controlling the coating crystallographic structure and properties of ZrO₂/Al₂O₃ coatings [20,22,25]. However, it is well established that deposition temperature and substrate bias, as well as gas pressure during deposition, crucially affect coating structure and physical properties, as they control the energy delivered to the growing film [42].

All coatings deposited in this study had a stabilized ZrO₂ structure - either t-ZrO₂ or c-ZrO₂. The FVAD coatings had a t-ZrO₂ structure, regardless of Tₐ or Vₕ, whereas the PMS coatings deposited under low Vₕ (at all Tₐ) had a c-ZrO₂ structure and the PMS coatings deposited under Vₕ≥100V had a t-ZrO₂ structure (Figure 6). The formation of c-ZrO₂ is favoured under sub-stoichiometric oxygen conditions, while t-ZrO₂ formation is favoured with small grain size or under elastic restraint [26]. The high deposition rate and high ionization of the deposited species characterizing FVAD are conducive to low grain size (Fig. 4) that stabilizes the t-ZrO₂ phase. Also, although residual stress could not be measured in the FVAD coatings, it is known that compressive stress increases with Vₕ [1,9]. In the PMS technique, the c-ZrO₂ structure originated from coating sub-stoichiometry [22]. However, when Vₕ increased to Vₕ≥100 V, the intrinsic stress (Fig. 7) induced the formation of t-ZrO₂ instead of the c-ZrO₂ phase.

Coatings deposited by either PMS or FVAD at low Tₐ and Vₕ had lower crystallinity, hardness, and Lₜ and inferior wear behaviour than obtained at higher Tₐ and Vₕ. Increasing Tₐ or Vₕ increased the coating hardness (Figs. 5 and 7a). The relationships between Tₐ and hardness are usually explained by the influence of Tₐ on grain size and microstructure [42,50]. Recently [23] we showed that the annealing of ZrO₂/Al₂O₃ coatings at temperature higher than 700°C enhances the composition re-arrangement and leads to formation of a nano-composite structure due to aluminum enrichment at grain boundaries and depleted aluminum in the grains. This may be the reason for the increased hardness of ZrO₂/Al₂O₃ coatings. Similar hardness increase was observed during in-situ annealing as Tₐ increases [42]. The relationship between hardness and Vₕ is more straightforward, coupled with a stress increase, and is well documented in many coating systems as well as in ZrO₂ coatings [49]. Thus, the increase in H with Vₕ is explained by the increase in compressive stress (Fig. 7). The hardness decrease in Vₕ≥100 V FVAD coatings is probably a result of a decrease in compressive stress due to relaxation during growth. The residual stress of the FVAD coating could not be measured due to low lateral homogeneity of the coatings [17]. However, the increase in ion energy with Vₕ and the high ionization of the plasma species in the FVAD plasma can produce in-situ recovery, stress release, and a decrease in hardness [9,51].

Lₜ clearly increased with increasing Tₐ for both PMS and FVAD coatings, while a clear relationship between Lₜ and Vₕ was not found in this study (Fig. 8). We believe that the increase in Lₜ with Tₐ is related to improved coating ordering. Lately, ordering to nc-ZrO₂/a-Al₂O₃ was found after annealing [23]. Vₕ was also found to induce structure changes (Fig. 6) and improve the hardness (up to the point where it induces in-situ recovery [51]). However, increased Vₕ was accompanied with stress increase, which reduces Lₜ [52]. Thus, Vₕ can be beneficial in improving Lₜ only at low values, Vₕ=50 to -100 V in this study. Tₐ is also beneficial in improving ZrO₂/Al₂O₃ coating wear resistance (Table 1). The reduction of W with Tₐ is related to the increase in H and Lₜ, but is probably also related to the improved coating ordering.

V. CONCLUSIONS

The deposition parameters, as well as the deposition technique, influence the ZrO₂/Al₂O₃ structure and properties. Coatings deposited using FVAD were stabilized in the t-ZrO₂ structure due to the small grain size, while PMS coatings were stabilized in the c-ZrO₂ structure due to the sub-stoichiometric oxygen content. However, the ZrO₂ phase and the crystal orientation can be controlled by Tₐ and Vₕ. Increasing Vₕ changes the coating structure from c-ZrO₂ to t-ZrO₂ due to the increase in the compressive stress, which creates elastic restraint. Tₐ was found to be the most effective deposition parameter for increasing the coating hardness and adhesion for a given coating composition. FVAD coatings deposited at high temperature (500°C) had the highest wear resistance, with Lₜ > 100 N and W=0.6×10⁻⁶ mm³/Nm.

VI. ACKNOWLEDGEMENTS

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