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TiN coating on an electrical discharge machined WC-Co hardmetal: surface integrity effects on indentation adhesion response

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Abstract Electrical discharge machining (EDM) is an alternative-shaping route for manufacturing complex component shapes of hard and brittle materials such as hardmetals (WC-Co cemented carbides). It is well established that in these materials EDM typically induces a heat affected surface layer with poor integrity. This degradation effect may be compensated through specific post-EDM surface treatments either by thermomechanical means or material surface deposition. In the latter case, a key property for optimal performance is the level of coating to substrate adhesion and how this is affected by the EDM-induced surface. The main objective of this investigation was to evaluate the adhesion strength of TiN coatings on EDMed hardmetals. A series of hardmetal samples that had been subjected to different multi-pass sequential EDM levels were coated with TiN. Adhesion behavior was assessed using the indentation adhesion test and comparing the critical load for crack extension (P_c) and the interfacial fracture toughness ($K_{Ic,interface}$) to those exhibited by the TiN coating deposited on a ground and polished substrate (used as baseline control). Experimental results indicated that indentation adhesion increased with finer-executed EDM, almost reaching baseline level values. The results are discussed on the basis of the compromising EDM influence on both surface integrity of the substrate and tortuousness at the interface, the latter resulting in mechanical anchoring of the TiN coating to the hardmetal substrate.

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Introduction

Hard coating of tools and machine components is a well-established technique for achieving significant performance improvements in applications demanding wear resistance, low friction, and high chemical and environmental stability. Recently, such surface modification technology has also shown to be an effective treatment for improving relatively poor surface integrity, resulting from electrical discharge machining (EDM) of tool steels [1, 2] and hardmetals (WC-Co cemented carbides) [3]. Physical vapor deposition (PVD) of a thin surface layer of metallic/covalent hard materials (e.g. TiN, TiAlN) was found to improve their mechanical strength as a consequence of coating-related beneficial changes on surface texture, nature and severity of critical flaws, and residual stress state. Since EDM is one of the most important abrasionless machining methods for hard materials, particularly for applications where precise, intricate and complex geometries are primary design requirements, it is critical that a fundamental understanding of how and why surface modification works be developed.

It was the aim of this investigation to establish and understand EDM-induced surface integrity effects on TiN coated cemented carbides. The indentation adhesion response of TiN coated WC-Co cemented carbide substrates, as a function of the surface finish from different sequential and upgrading EDM was investigated. Values of the critical load for crack extension (P_c), and interfacial fracture toughness ($K_{Ic,interface}$) were compared with those exhibited by the TiN coating deposited on control ground and polished substrate.

Experimental procedure

The base material studied was a commercial fine-grained WC-10%$_{wt}$ Co hardmetal grade supplied by DURIT Metalurgia Portuguesa do Tungsténio. Longitudinal sections of rectangular bars (3 mm × 4 mm × 45 mm) were shaped by EDM in a commercial wire-cut machine equipped with an advanced pulse-type generator. Four different surface finish variants were attained by sequential EDM [4]. Control based on imposed voltage between the hardmetal sample and the brass wire (within a range of 15–70 V) was applied for rough- and finish-shaping operations (conditions A and B). Meanwhile, aiming to optimize surface integrity rather than accuracy or time, limited maximum cutting rates (between 1.2 and 2 mm/min) and multistep machining were imposed for attaining the surface finish conditions C and D. For comparison purposes, one baseline surface finish condition, referred to as P, was produced by conventional grinding and polishing up to optical finish. Characterization of each surface finish variant was carried out in terms of roughness and surface integrity. The former was determined using a surface texture measuring system and the results are given, on the basis of five measurements per sample, in terms of R_a (arithmetic deviation from the mean line through the complete profile) and R_y (maximum profile depth). On the other hand, the latter was evaluated on top- and cross-sections by means of a scanning electron microscopy (SEM). Specific substrate shape operations involved and resulting roughness for each surface condition examined, together with the nomenclature used in this work, are given in Table 1.

Titanium nitride (TiN) films were deposited on all the different surface finished substrates using BALZERS's PVD arc ion plating process. Microstructural and morphological characteristics of the films and substrate/coating interfaces were determined from cross-sections of coated specimens through SEM.

Indentation adhesion behavior of all the coated specimens was evaluated with a diamond Rockwell C indenter (tip radius of 0.2 mm and angle of spherical head of 120°) at discrete applied loads over a range of 98–1470 N. Post-indentation examination of the contact response, attempting to differentiate cracking and/or coating delamination, was performed by optical microscopy and SEM.

Results and discussion

Surface integrity

Surface integrity involves the study and control of both topography and metallurgy alterations produced at the surface level during manufacture, including their effects on material properties and the performance of the surface in service [5]. This is particularly true in the case of coated components, where the surface characteristics of the substrate influence not only on the engineering properties at the effective subsurface level but also at the interface and surface (coating) ones [6].

Transverse polished sections corresponding to sequential EDM for the coated specimens are shown in Fig. 1. Besides the TiN coating and the hardmetal substrate discerned for all the surface finish variants, condition A also exhibits a 10 μm thick brass layer, homogeneously deposited on the shaped surface, from the wire used as a tool electrode. As expected from a previous work [4, 7], EDM-induced damage (cracks, craters, etc.) and surface roughness are significantly decreased with finer-executed EDM. Since thin films tend to inherit the substrate topography [8], roughness parameters evaluated for uncoated and coated samples were found to be similar (Table 1). However, since intrinsic irregularities associated with the coating

Table 1 Surface finish conditions evaluated: nomenclature, involved substrate machining sequence and resulting roughness for both uncoated and TiN coated specimens

Condition	Machining operations (WC-Co substrate)	Uncoated		TiN coated	
		R_a (μm)	R_y (μm)	R_a (μm)	R_y (μm)
A	Rough wirecut – EDM	3.75 ± 0.62	20.17 ± 4.53	3.89 ± 0.55	22.54 ± 3.95
B	As A + fine shaping	1.33 ± 0.12	8.30 ± 2.01	1.36 ± 0.18	8.38 ± 2.47
C	As B + surface finishing	0.41 ± 0.04	2.84 ± 0.66	0.49 ± 0.06	3.69 ± 0.79
D	As C + surface microfinishing	0.11 ± 0.02	0.88 ± 0.12	0.10 ± 0.04	1.22 ± 0.21
P	Grinding + polishing with diamond	0.01 ± 0.01	0.07 ± 0.02	0.07 ± 0.03	0.40 ± 0.07

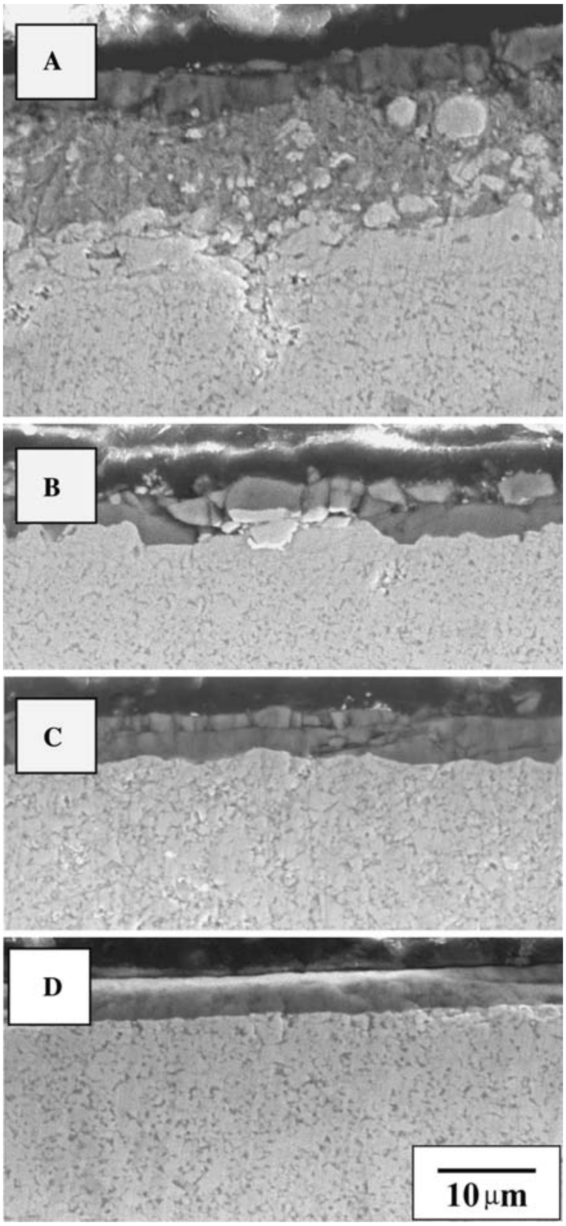

Fig. 1 SEM micrographs showing surface integrity (transverse to cut, polished sections) associated with sequential EDM on coated specimens

deposition process seemed to be relatively high as compared to the extremely smooth substrate baseline surface [9], as evidenced in Fig. 2, control condition P was the exception. As a result, roughness values for the coated P samples were observed to be close to those exhibited by the microfinish surface condition D.

Extensive microstructural and mechanical characterizations, including residual stresses assessment, for the coatings deposited on the substrates have been reported elsewhere [3]. Typically the coatings were dense and uniform (3 μm in thickness) with fine-grained columnar structures, exhibiting a residual compressive stress of about 2 GPa. Furthermore, the deposited coatings did not show significant morphological difference associated with the different substrate surface conditions.

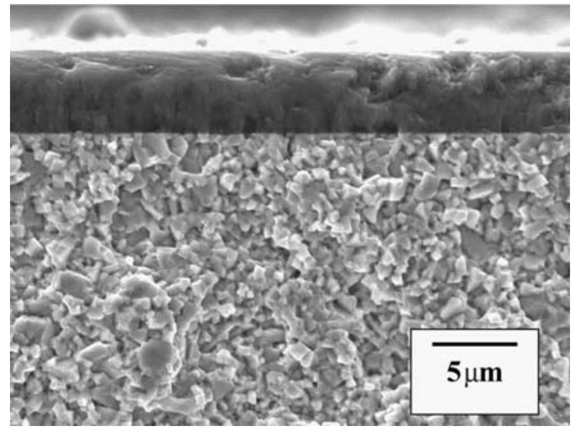

Fig. 2 Cross-sectional SEM micrograph of TiN coating deposited on a previously ground and polished substrate

Coated system cross-sections were examined to determine machining effects on surface texture, a relevant factor from the viewpoint of adhesion of the coating to the substrate. From Fig. 1, it is clear that the interface of coating/substrate for the EDM-related conditions consists of evenly distributed microcracks and craters. Their depth and irregular aspect was found to diminish as the EDM execution got finer, in agreement with the roughness measured at the top surface. As a consequence, the interface for these surface conditions exhibit a variable and discrete tortuousness, different from the flat profile observed for the control condition P, and affects interfacial fracture toughness, as discussed in the following sections.

Indentation adhesion behavior

In order to investigate the effect of EDM on the adhesion of the TiN coating to the hardmetal substrates, indentation adhesion tests were performed on coated specimens. The indentation method was chosen because it is a simple, practical and well-established procedure for assessing adhesion in coating/substrate systems [10–14]. It is based on the original idea of Chang et al. [10] for measuring adhesion as the resistance to propagation of a mechanically stable crack introduced at the interface by employing conventional indentation procedures. Two indentation adhesion parameters: the critical normal load for lateral crack generation (P_c) and the interfacial fracture toughness ($K_{Ic,interface}$), were estimated for each surface condition investigated.

Figure 3 shows the fracture pattern diagram for all the coated systems as a function of normal indentation load using a conical diamond indenter. As expected, in all the cases, generation of radial cracks around the contact area was followed, with increasing applied load, by spalling due to propagation of lateral cracks at the substrate/coating interface [14]. However, the load level at which initial flaking of the coating occurred,

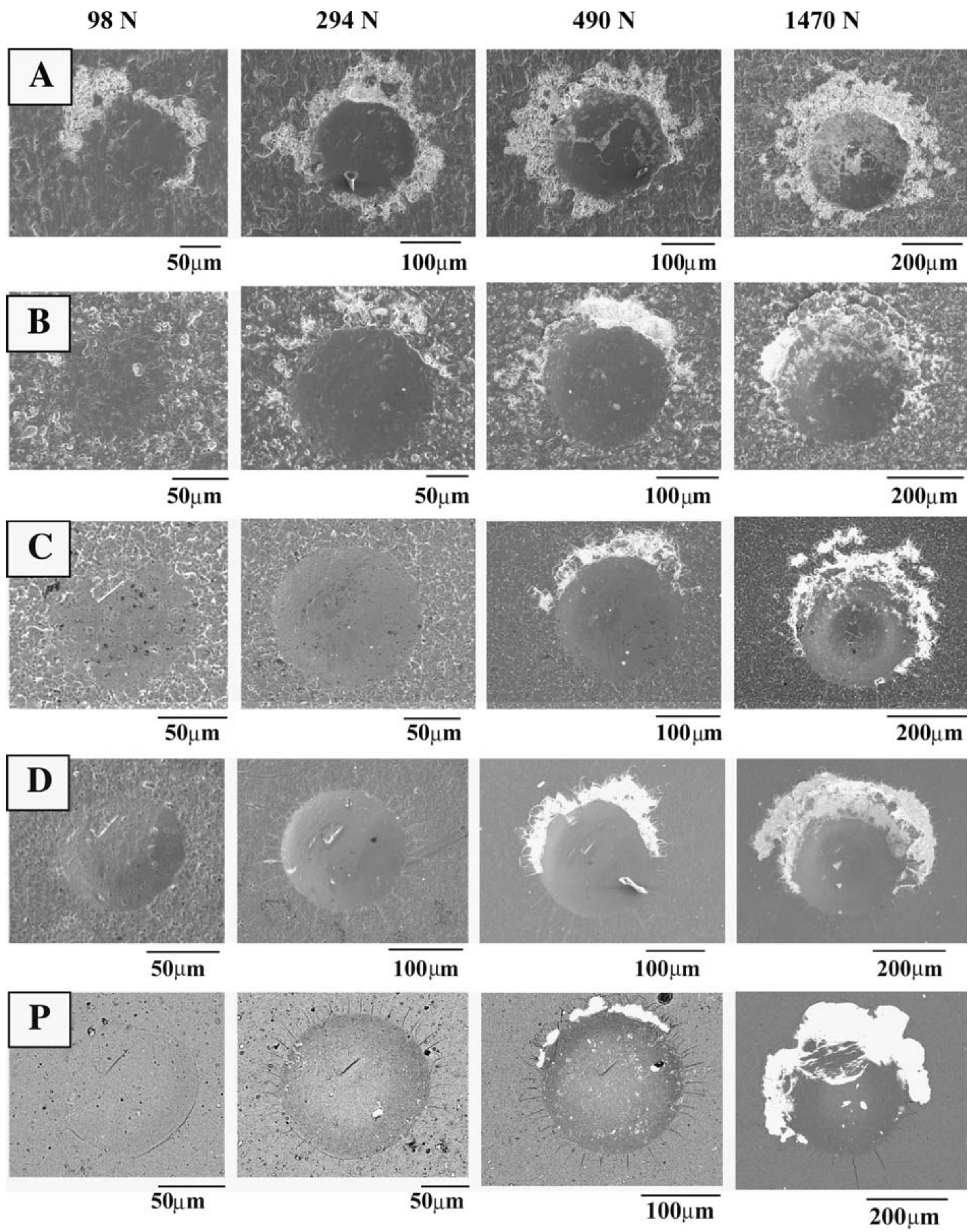

Fig. 3 Indentation adhesion testing: fracture pattern diagram as a function of normal load and surface finish condition

and how it extended as applied load increases was observed to be different depending on the final shaping operation. The latter aspect can be better discerned from the equivalent lateral crack diameter versus the applied load curves (Fig. 4) [11, 12]. The slope of the linear part reflects the ease with which the lateral crack propagated at the interface (similar to the indentation fracture mechanics approach used in bulk ceramics [15, 16], but here it follows an inverse relationship respect to $K^2_{\mathrm{Ic,interface}}$ [10]). It can therefore be used as a qualitative measure of adhesion.

Several other observations may be highlighted as related to both indentation adhesion parameters, P_c and $K_{\mathrm{Ic,interface}}$. For surface conditions corresponding to rough- (A) and finish- (B) EDM, critical loads for partial and discrete spalling, are lower than 98 N and 294 N, respectively. On the other hand, a similar delaminating scenario is only attained for the other EDM surface variants (C and D) for applied loads equal to or higher than 294 N. Such a distinct behavior was also observed with respect to the transition from partial to spread spalling, but at a higher applied load (490 N).

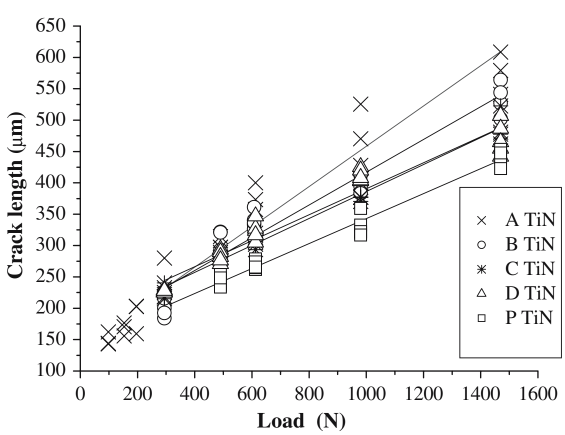

Fig. 4 Lateral crack length–indentation load curves for the different surface finish conditions studied

Regarding the coating delamination behavior exhibited by the control condition P, threshold load values for assessing the above fracture mechanisms were slightly higher than those determined for the EDM surface finish conditions. Based on extensive inspection of a series of more than five indentations for each discrete load level investigated, approximate values for P_c were estimated, either as the mean value or via interpolation (Table 2).

A decrease in the slope of the best-fitting curves for indentation load versus lateral crack length as EDM got finer-executed was observed (Table 2). Considering that lower slopes imply higher interfacial fracture toughness values, these experimental results indicate an increase in adhesion strength with sequential and upgrading EDM up to surface microfinish levels. Although the observed trend is in complete agreement with the results presented above, the indentation toughness parameter associated with the measured slopes allows one to differentiate adhesion strength among all the surface variants investigated, including C, D and P conditions. This observation is in agreement with the general idea of $K_{\mathrm{Ic,interface}}$ being a more discriminating parameter than P_c [11], although accuracy in the latter case would surely be increased as the number of discrete loads used for the study gets larger.

Following the above ideas, a semi-quantitative assessment of the influence of EDM on the adhesion strength of TiN coatings on hardmetal substrates was attempted by the evaluation of the parameter $K_{\mathrm{Ic,interface}}$, on the basis of the original indentation fracture mechanics model proposed by Marshall et al. [16] for the lateral crack system in bulk ceramics, but as adapted by Diao et al. [14] for coated systems. Assuming that the driving force for generating (and propagating under pure mode I) a lateral crack at the interface is given by the residual force at the interface during unloading, Diao and coworkers proposed an expression for evaluating $K_{\mathrm{Ic,interface}}$ as given by

Table 2 Indentation adhesion parameters, effective elastic modulus and effective hardness for each of the surface finish conditions studied

Coated system	P_c (N)	Best-fit slope of crack length–load curve[a] (μm/N)	H_{eff} (GPa)	E_{eff} (GPa)	$K^{b}_{\mathrm{Ic,interface}}$ (MPam$^{1/2}$)
TiN/A	49	0.32	10.9	298	0.02
TiN/B	245	0.26	17.0	308	0.16
TiN/C	294	0.22	17.4	307	0.18
TiN/D	294	0.21	17.1	307	0.18
TiN/P	392	0.20	17.1	308	0.25

[a]Inversely proportional to $K^2_{\mathrm{Ic,interface}}$, from data plotted in Fig. 4

[b]As estimated from P_c, according to Eq. 1

$$K_{\mathrm{Ic,interface}} = \frac{P_c}{kt^{3/2}}\left(\frac{E_{\mathrm{eff}}}{H_{\mathrm{eff}}}\right)^{1/2} \quad (1)$$

where P_c is the critical normal load for the generation of lateral cracking (Table 2), t is the coating thickness, and k is an experimental constant of value about 0.98×10^6 as estimated from indentation cracking data measured on TiN coatings deposited on cemented carbide substrates using a conical indenter [14]. The intrinsic properties of coating and substrate, including the deformation of the latter, are considered in the interfacial fracture toughness assessment through the effective elastic modulus (E_{eff}), as determined from the elastic contact analysis conducted by Diao et al. [14], and effective hardness (H_{eff}), as experimentally determined from Vickers hardness indentation. The corresponding E_{eff}, H_{eff} and $K_{\mathrm{Ic,interface}}$ values for each surface condition investigated are include in Table 2. Two aspects may be highlighted. First, the estimated $K_{\mathrm{Ic,interface}}$ values are of the same order of magnitude as for published data in similar coating-substrate systems [11, 14]. This is a relevant finding since it confirms the suitability of the analytical approach used above for assessing interfacial fracture phenomena. Second, the results substantiate the observed trends that as EDM gets finer execution, interfacial fracture toughness increases up to values close to those exhibited by the control condition P.

Adhesion strength: interface geometry effects

The effective role of a tortuous interface on the adhesion strength of a coated system is difficult to define [17]. On one hand, it may be beneficial in its role on mechanically keying the coating to the substrate and/or accommodating some mismatch in the corresponding thermal coefficients of expansion. On the other hand, it could be detrimental because the unevenness of the coating/substrate interface may entail the generation of normal stresses (whereas they are zero for completely flat surfaces) and existence of stress concentrators at geometrical discontinuities [18]. From this perspective, it is interesting to note that EDM shaping induces a 2-D isotropic surface topography with uniformly distributed microcraters plus a significant level of tensile residual stresses with a maximum near the machined surface and diminishing rapidly with depth [4, 19]. Additionally, as EDM gets finer execution the referred discrete depressions are not only smaller in depth but also less defined in terms of geometry discontinuities, without significant changes in the residual stress state induced by the EDM process. Within this framework, it may be speculated that the observed trends in adhesion strength result from a balance between biaxial mechanical interlocking effects, dependent on the inversely related depth and stress raising severity of the filled-in microcraters (the actual anchoring units), and the effective local stress state at eventual interface sources for inducing spallation. Although a thorough understanding of how stresses are modified in the presence of an uneven interface is beyond the scope of this investigation, the significance of this aspect should be noted, especially considering that relatively high adhesion strength levels are achieved for ‘‘discretely unven’’ EDM surface microfinish conditions. This in spite of the fact that deposition of a layer with compressive residual stresses would be expected to enhance, if the interface were ‘‘flat’’, the EDM-induced tensile residual stress state already existing at the surface substrate [20].

Conclusions

A investigation on the adhesion strength of TiN coatings deposited on electrical discharge machined hardmetals was conducted. Adhesion was evaluated by means of indentation technique with results presented in terms of critical normal load for lateral crack generation, P_c, and interfacial fracture toughness, $K_{\mathrm{Ic,interface}}$. It was observed that adhesion strength increases with sequential and upgrading EDM, reaching levels very close to those exhibited by the same coating on a ground and polished surface condition. EDM-induced effects were mainly characterized in terms of the uneven character of the interface geometry, as compared to the flat surface of the control substrate. As EDM got finer executed, the interface became less wavy in character and narrower in depth with fewer defined discrete microcraters. The adhesion behavior determined is then speculated to result from the compromising EDM influence on mechanical anchoring of the coating to the substrate, as related to depth and stress raising severity of the filled-in discrete depressions, and plausible changes in the local stress state at such geometry discontinuities.

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