Manuscript refereed by Luis Llanes (UPC, Spain)

Residual Stress Behavior of Cemented Carbide Coated with CVD Ti(C,N)/α-Al_2O_3 Multilayers: Effect Of Different Blasting Conditions

Maiara Moreno (Department of Materials Engineering, University of São Paulo, 13563-120, São Carlos, Brazil) maiara.moreno@usp.br; José García (AB Sandvik Coromant, SE-126 80 Stockholm, Sweden) jose.garcia@sandvik.com; Haroldo Pinto (Department of Materials Engineering, University of São Paulo, 13563-120, São Carlos, Brazil) haroldo@sc.usp.br; Manuela Klaus (Abteilung für Mikrostruktur- und Eigenspannungsanalyse, Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, D-12489 Berlin, Germany) klaus@helmholtz-berlin.de; Christoph Genzel (Abteilung für Mikrostruktur- und Eigenspannungsanalyse, Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, D-12489 Berlin, Germany) genzel@helmholtz-berlin.de

Abstract

In this work the effect of different blasting conditions on the residual stress of Ti(C,N)/α-Al_2O_3 coatings deposited on cemented carbide are investigated. Top-blasting using alumina and zirconia micro-particles at different pressures was applied on top of the coated carbides. The residual stress behavior of the WC-Co/Ti(C,N)/α-Al_2O_3 system in a heating cycle (up to 800◦C) was studied *in-situ* using energy dispersive synchrotron X-ray diffraction. The results show that the stress behavior in both coating and carbide can be tailored by optimizing the blasting parameters.

Introduction

Cemented carbides are an important category of composite materials, which combine the hardness of a hard phase, with the ductility of a binder phase [1]. They are used as substrates for coated cutting tools. Hard ceramic thin films deposited by Chemical Vapor Deposition (CVD) or Physical Vapor Deposition (PVD) improve the wear resistance of the cutting tools in machining applications such as milling [2]. Milling is an intermittent machining operation where the insert is subjected to large thermo-mechanical loads, in particular when cooling media is used. The main wear type that leads to failure in milling tools is called comb cracks, which is the result of intermittent mechanical and/or thermal fatigue. The cracks are initiated by the alternating expansion and contraction of the surface of the tool as it is heated and cooled during the interrupted cutting. This process of crack formation and propagation is accelerated by concentration of stresses developed due to mismatches in thermal expansion coefficient between the coating layers and the substrate [3]. CVD coatings exhibit tensile residual stresses after deposition that favor crack propagation. Thus, to retard crack propagation, tensile residual stresses of as-coated CVD inserts can be reduced by post-treatment methods, such as mechanical peening [4]. The process may also contribute to improve the coating adhesion and the performance of coated cutting tools. The top-blasting process may be more (or less) effective depending on the type of particles and the impacting conditions used. The analysis of residual stresses is extremely important in CVD coated cutting tools, because the understanding of how they act on the insert can help to avoid or minimize the appearance of comb cracks. In this study, the residual stresses are evaluated using energy dispersive synchrotron X-ray diffraction to understand the effect of different blasting conditions on the residual stress behavior of Ti(C,N)/α-Al_2O_3 coatings deposited on a WC-Co substrate. Blasting was conducted using both alumina and zirconia micro-particles at different blasting pressures.

Experimental

A Ti(C,N)/α-Al_2O_3 (4µm/4µm) CVD coating was deposited on a WC-6%Co cemented carbide. A thin layer of TiN (0,3 µm) layer was applied on the cemented carbide before the carbonitride deposition. A Ti(C,N,O_x) bonding layer was deposited between the carbonitride and the α-Al_2O_3 layer (Figure 1). Table 1 lists the samples conditions selected for this work.

The XRD experiments using ED synchrotron diffraction were performed at the Material Science Beamline EDDI (Energy-Dispersive Diffraction) of the Helmholtz-Zentrum Berlin at the storage ring BESSY in Berlin, Germany [5]. Residual stress analyses were conducted during a thermal cycle ranging from room temperature (RT1) to 800ºC (typical working peak temperature of cutting tools) and cooling to room temperature again (RT2). The $\sin^2\psi$ method was used to determine the stress values. One of the most important advantages of the $\sin^2\psi$ method is its numerical stability and insensitivity to experimental uncertainties. The analysis of the thermal influence on the residual stress behavior for each material was conducted *in-situ* (heating with an Anton Paar resistance furnace, constant flow of Ar). The fundamental equation of stress analysis using XRD was applied (see Eq.1). The diffraction

elastic constants (DECs) required for the evaluation of the residual stresses and stress depth profiles were evaluated by the Eshelby-Kröner model [6,7], using the respective single crystal elastic constants [8] (Table 2). In the present case the samples do not have compositional gradients within the sub-layers. Thus, the results of the residual stresses do not depend on the variations of elastic and thermal material properties, as well as on the changes of the strain-free d_0-spacing with the depth into the sub-layers. Therefore, an average value over the multiplicity of each *(hkl)* plane is the most adequate choice to determine representative stress values for each layer.

$$\varepsilon_{\varphi\psi}^{hkl} = \frac{1}{2} S_2^{hkl} \sigma_\varphi \sin^2\psi + S_1^{hkl}(\sigma_{11} + \sigma_{22}) \quad \text{(Eq.1)}$$

where $\varepsilon_{\varphi\psi}^{hkl}$ is the lattice strain, $\sigma_\varphi = \sigma_{11}\cos^2\varphi + \sigma_{22}\sin^2\varphi + \sigma_{12}\sin 2\varphi$ - in-plane residual stress component in the azimuth direction φ and S_1^{hkl} and $\frac{1}{2}S_2^{hkl}$ are the diffraction elastic constants, which depend on the investigated reflection *hkl*.

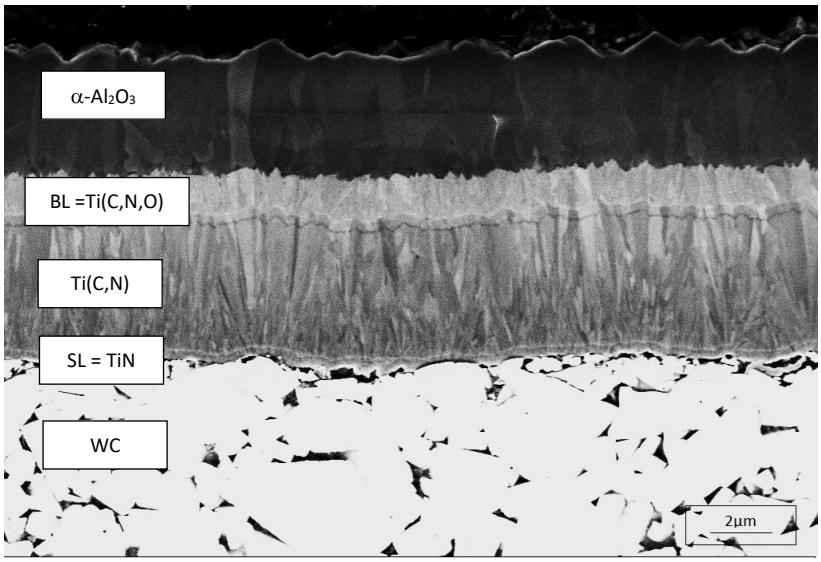

Figure 1: SEM image of coated insert investigated (BL = bonding layer, SL = starting layer).

Table 1: Samples and blasting conditions in WC-Co/Ti(C,N)/α-Al_2O_3 systems investigated.

Name	Blasting Condition
Sample A_NB	No blasting
Sample B_B1.8	alumina - 1.8 bar
Sample C_B2.0	alumina – 2.0 bar
Sample D_SP	zirconia - 5.2 bar
Sample E_SP+B2.3	alumina - 2.3 bar + zirconia - 5.2 bar

Table 2: Diffraction elastic constants for the layers and WC. [6-8]

Component	hkl	S_1^{hkl} (* 10^{-6} MPa^{-1})	$\frac{1}{2}S_2^{hkl}$(* 10^{-6} MPa^{-1})
α-Al_2O_3	012/024	-0.685	3.36
	110	-0.557	2.96
Ti(C,N)	200	-0.425	2.665
	220	-0.465	2.795
WC	001	-0.32	2.01
	100	-0.46	2.74
	101	-0.32	1.95
	110	-0.49	2.7
	002	-0.32	2.01
	111	-0.35	2.17
	200	-0.46	2.74
	102	-0.28	1.82
	201	-0.37	2.31

Results

The residual stress analysis of sample A_NB (non-blasted condition) is shown in Fig. 2. It is observed that the α-Al_2O_3 layer presented high tensile stress at RT1 (600 MPa). At 800°C the residual stress value decreases to +150 MPa. After cooling at RT2 the α-Al_2O_3 layer presents a tensile stress of the same magnitude as RT1. For the Ti(C,N) layer the calculated residual stresses were 600 MPa (RT1), compressive stresses of -150 MPa (800°C) and tensile stress similar to RT1 after cooling at RT2 (600 MPa). The WC phase presents very low tensile stress at RT1, 800°C and RT2 (ranging from 20 to 50 MPa).

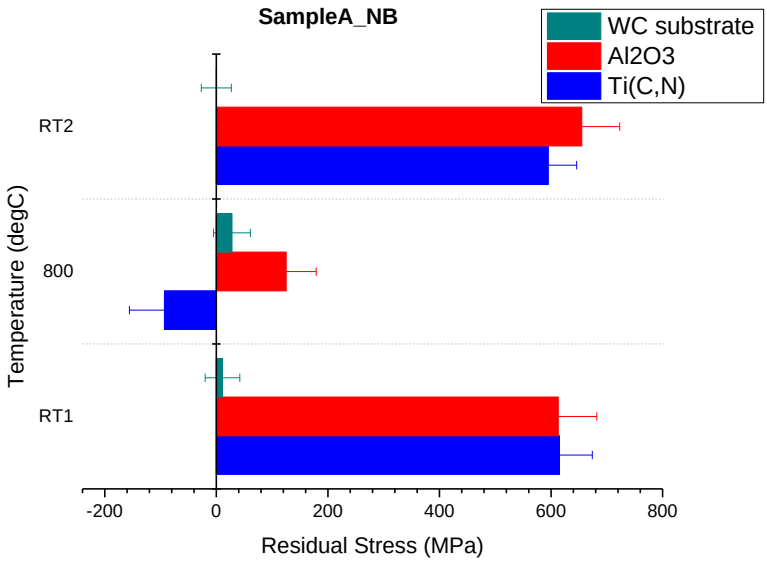

Figure 2: Residual stresses for Sample A_NB.

In Figure 3 the residual stress analysis of sample B in blasted condition (1.8 bar) is shown. The α-Al_2O_3 layer presented high tensile stress at RT1 (500 MPa). At 800°C the residual stress is compressive (-200 MPa). At RT2 the α-Al_2O_3 layer develops a tensile stress of the same magnitude as RT1. For the Ti(C,N) layer the calculated residual stresses were 625 MPa (RT1), compressive stresses of -200 MPa (800°C) and tensile stress at RT2 of the same magnitude as at RT1 (575 MPa). The WC phase presents very low tensile stress at RT1, 800°C and RT2 (between 30 and 10 MPa).

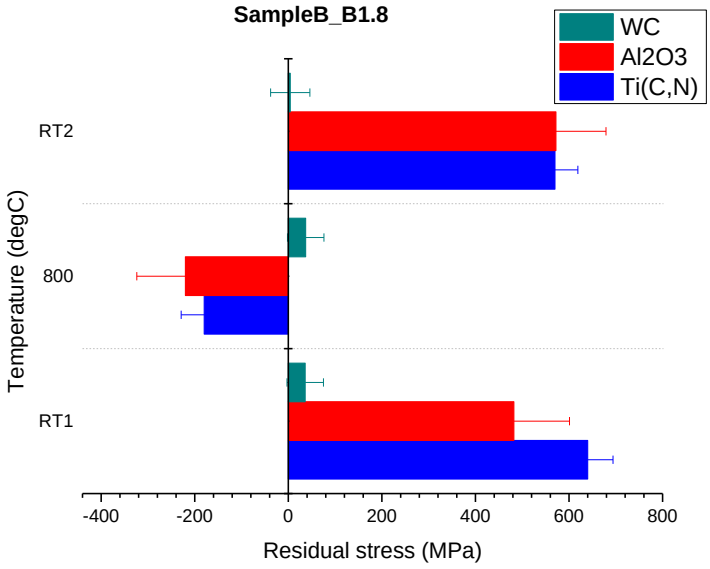

Figure 3: Residual stresses for Sample B_B1.8

The results of increasing the blasting pressure of the alumina particles from 1.8 to 2.0 bar is shown in Figure 4. The α-Al_2O_3 layer presented low tensile stress at RT1 (175 MPa). At 800°C the residual

stress is compressive (-500 MPa) evolving to tensile stress at RT2 (450 MPa). The calculated residual stresses in the Ti(C,N) layer were 600 MPa at RT1, compressive stresses of -150 MPa at 800°C and tensile stress of 650 MPa at RT2. As for the other samples blasted with alumina particles, the WC phase presents very low tensile stress at RT1, 800°C and RT2 (ranging from 50 to 20 MPa).

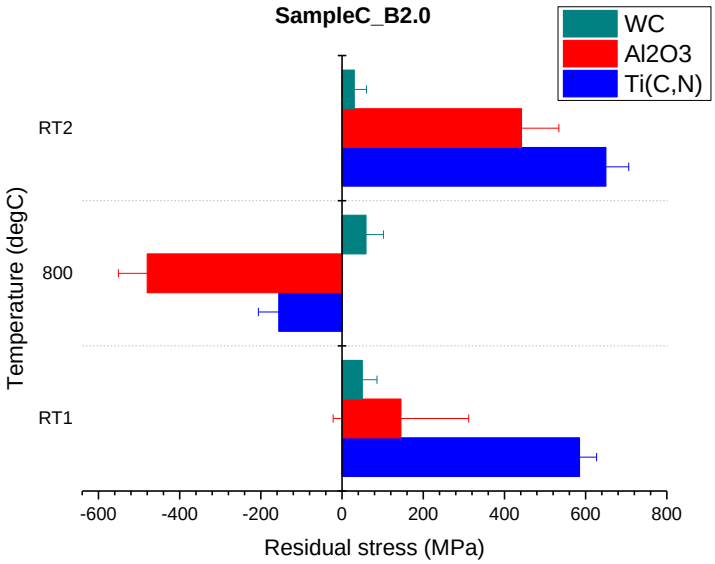

Figure 4: Residual stresses for Sample C_B2.0

In Figure 5 the residual stress analysis of sample D top-blasted with zirconia particles is shown. It is possible to observe that in Sample D_SP the α-Al_2O_3 layer presented tensile stress at RT1 and RT2 of the same magnitude (375 MPa). At 800°C the residual stresses are of tensile type (100 MPa). For the Ti(C,N) layer the calculated residual stresses were 175 MPa (RT1), compressive stresses of -300 MPa (800°C) and tensile stress at RT2 (300 MPa). The WC phase presents for this blasting condition compressive stresses at RT1 (-550 MPa), which reduce at 800°C and RT2 (-75 MPa).

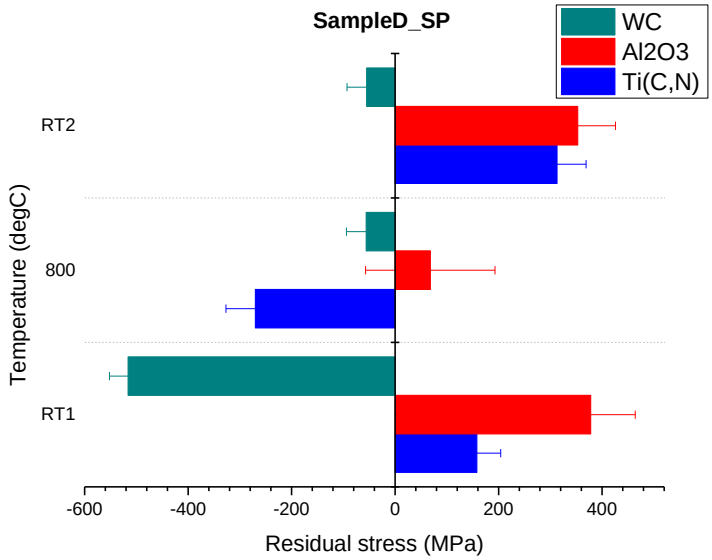

Figure 5: Residual stresses for Sample D_SP

Residual stresses determination in sample E after both blasting with zirconia and alumina particles is shown in Figure 6. The α-Al_2O_3 layer presented similar values of tensile stress at RT1 and RT2 (350 MPa). However, at 800°C the residual stress changed to compressive stresses of -375 MPa. For the Ti(C,N) layer the calculated residual stresses were 175 MPa at RT1, -300 MPa at 800°C and 300 MPa at RT2 The WC substrate presents high compressive stress at RT1 (~ -475 MPa). At 800°C and RT2 the residual stress of the WC phase is almost negligible.

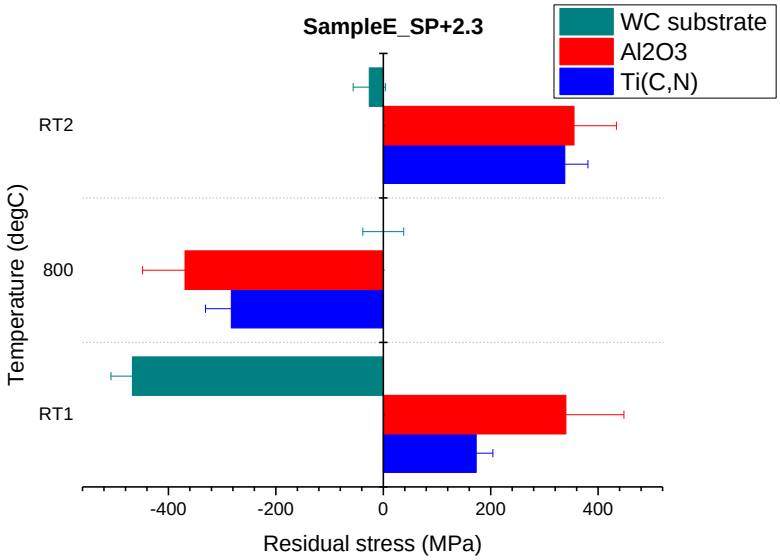

Figure 6: Residual stresses for SampleE_SP+B2.3

Discussion

In this work we have investigated the residual stress behavior of as-coated and top-blasted WC-Co/Ti(C,N)/α-Al_2O_3 systems in one temperature cycle (RT1 - 800°C - RT2). The results of the *in-situ* analysis using high energy synchrotron X-ray diffraction for conditions investigated show particular trends that have been observed before [3,4,9,10]. For example in the as-coated condition the Ti(C,N)/α-Al_2O_3 system presents an alternating cycling behavior of residual stresses, evolving from tensile stresses (+600 MPa) to compressive residual stresses (-100 MPa) at high temperatures. After cooling down at room temperature again, the residual stresses evolve to tensile stresses of the same magnitude as the initial condition (+600 MPa). This result is in agreement with previous works reported by the authors [3][9] and may indicate an elastic behavior in terms of stress evolution for the carbonitride and oxide layer. Interestingly, the residual stress values of the WC phase in the as-coated sample (sample A_NB) are almost of the same magnitude (+30 MPa) during the entire thermal cycle.

Top-blasting with alumina particles influences the initial residual stress of the α-Al_2O_3 layer. The post-treatment process reduces the tensile stresses of the α-Al_2O_3 layer. By increasing the top-blasting pressure up to 2 bar, the initial residual stress is reduced from + 600 MPa to +180 MPa. For this type of blasting conditions, evolution of high compressive stresses is expected, as observed in previous works [3,10], but despite the reduction of tensile stress absolute values, the α-Al_2O_3 layer remains in tensile stress. The main difference between the coating system investigated and the previous works [3,10] is the absence of a TiN thin top layer (which is removed during top-blasting). This is a fundamental difference that may influence the final residual stress behavior and needs further investigations.

Another effect observed is the evolution of higher compressive stress at high temperatures for the α-Al_2O_3 top-blasted layer (up to -500 MPa at 800°C). However, top-blasting with alumina particles does not influence the residual stresses of the Ti(C,N) and WC phase, showing similar values as in the as-coated non-blasted condition. It is known that the blasting effect is confined to very few microns on the top, and even only remaining in the α-Al_2O_3 layer [10].

Top-blasting with zirconia particles has the largest effect on the residual stress of the WC phase. This is in agreement with recent works [4]. Compressive stresses of up to -500 MPa were calculated in the WC phase for the zirconia blasted sample at RT1. This residual stress value is below the expected range (1-3 GPa) but it is certainly influenced by the blasting parameters (pressure, time, particle size, etc). Remarkably, heating the sample at 800°C results in a complete relaxation of the compressive residual stresses of the WC phase. Here it is worth to mention that the time needed to collect the XRD data in the in-situ measurement is 10 min, which may allow for annihilation of defects and relaxation of stresses. In the zirconia blasted sample the Ti(C,N) layer presented reduced tensile stresses at RT1 and higher compressive stresses at high temperatures. However, the α-Al_2O_3 layer residual stress condition is not affected. The influence of the zirconia blasting in the residual stress of the coating layers needs further investigation.

Finally combining both the alumina and zirconia top-blasting results in higher compressive stresses for the WC phase and higher compressive stresses at high temperature for both the Ti(C,N) and the α-Al_2O_3 layer. This condition may be optimum in terms of delaying crack propagation.

Conclusions

The first results of our investigations show distinct residual stress evolution for different blasting conditions in the same WC-Co/Ti(C,N)/α-Al_2O_3 sample. Top-blasting with alumina particles mainly influences the residual stress behavior of the coating system, introducing high compressive stresses in the α-Al_2O_3 layer, whereas blasting with zirconia particles influences the residual stress of the WC phase in the substrate and to a lesser extent the residual stress of the coating layers both at room and high temperatures. These results also indicate that improved tailored residual stress conditions can be achieved by careful design of the combination of post-treatment processes.

Acknowledgements

M. Moreno, H. Pinto and J. García thanks the partial financial support of the Create-Net network project within the Horizon 2020 EU-Program (RISE project N°644013).

References

[1] J. García, V. Collado Ciprés, A. Blomqvist, B. Kaplan. Cemented Carbide Microstructures: A Review. Int. J. Refract. Met. Hard Mater. 80 (2019) 40-68.
[2] Ch. Mitterer, PVD and CVD Hard Coatings, Encyclopedia Comprehensive Hard Materials, Vol. 2 (2014) 449-467.
[3] J. García, M. Moreno, J. Östby, J. Persson, H. Pinto. Design of coated cemented carbides with improved comb crack resistance. In: 19th Plansee Seminar, 2017, Reutte, Tirol.
[4] L. Faksa, W. Daves, W. Ecker, T. Klünsner, M. Tkadletz, C. Czettl. Effect of shot peening on residual stresses and crack closure in CVD coated hard metal cutting inserts. Int. J. Refract. Met. Hard Mater. 82 (2019) 174-182.
[5] Ch. Genzel, IA. Denks, J. Gibmeier, M. Klaus, G. Wagener. The materials science synchrotron beamline EDDI for energy-dispersive diffraction analysis, Nuclear Instruments and Methods, in Physics Research Section a: Accelerators, Spectrometers, Detectors and Associated Equipment 578 (2007) 23–33.
[6] J.D. Eshelby. The Determination of the elastic field of on ellipsoidal inclusion, and related problems. Proceedings of the Royal Society A: mathematical, physical and engineering sciences, vol.241, no.1226 (1957) 376-396.
[7] E. Kröner. Berechung der elastischen Konstanten des Vielkristalls aus den Konstanten des Einkristalls. Zeitschrift für Physik A Hadrons and Nuclei151, no.4 (1958) 504-518.
[8] B. Eigenmann, E. Macherauch. Röntgenographische Untersuchung von Spannungszuständen in Werkstoffen. Teil III. Fortsetzung Matwiss. und Werkstoffen. Heft 3/1995, S.148-160 und heft 4/1995, S.199-216. Materialwiss. Werkstofftechn 27, no.9 (1996) 426-437.
[9] J. Garcia, H. Pinto, E. Ramos-Moore, C. Espinoza, J. Östby, R. Coelho, In-situ high temperature stress analysis of Ti(C,N) coatings on functionally graded carbides by energy dispersive synchrotron X-ray diffraction, Int. J. Refract. Met. Hard Mater. 56 (2015) 27–34.
[10] M. Klaus, Ch. Genzel, H. Holzschuh. Residual stress depth profiling in complex hard coating systems by X-ray diffraction. Thin Solid Films 517 (2008) 1172-1176.