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ANALYSIS OF CRITICAL LOAD OF MULTILAYERED/GRADIENT CrAlSiN PVD COATINGS MEASURED USING SCRATCH TESTS*

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Abstract

In stamping process, sliding between metal sheet and forming tool leads to wear of tool surface. Maintenance of stamping tools involves polishing the tool edge, or, in case of severe damage, replacement of the tool. Hard coatings have been applied on the stamping tool surfaces in order to reduce the maintenance cost causing a great economic impact in metal work industry. In this work, CrAlSiN coatings were applied by PVD onto SRV2W tool steel. The coating toughness was avaliated by the critical load in scratch tests. The following coating parameters were analyzed: 1- substrate surface finishing process; 2- coating architecture (multilayer or gradient); 3- chemical composition of the coating. AlSi concentration varied from the substrate/coating interface to the surface. Coatings were 3 µm thick. Scratch tests were carried out using a Vickers indenter. The applied normal load was varied from 0 to 13 N for a scratch length of 5 mm. In order to study the effect in the toughness, the substrate roughness was controlled by the finishing process before PVD coating. Samples subjected to polishing presented highest critical loads. Gradient coatings presented higher critical loads than those with a multilayer architecture. Coatings with higher concentration of AlSi near the surface supported higher loads before failure.

Keywords: PVD coatings; coating architecture; multilayered/gradient coating; scratch test; coating toughness.

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1 INTRODUCTION

In stamping process, sliding between metal sheet and forming tool leads to wear of tool surface. Related works show that the wear resistance of this kind of tribological contact can be improved by applying PVD coatings on tool surfaces [1]. Thin coatings are used to increase the wear resistance of components in a large number of engineering applications. However, if adhesion of the coating to the substrate is inadequate, the coating may detach prematurely, consequently causing catastrophic failure of the coated part [2].

The CrAlSiN coatings are composed for chromium nitride (CrN) containing aluminium and silicon. This kind of coating caught attention due to its high hardness (> 40 GPa), wear and oxidation resistance [3].

In addition to compositional modification, another approach to achieve multifunctionality is dedicated to coating architecture through multilayering or grading. Hardness is improved by using multilayer structure. Structural gradient coatings have been proven to be effective in reducing crack concentration, improving the adhesion between coatings and substrate [4-6].

The mechanical behavior of CrAlSiN gradient coatings was compared to the multilayered coatings. This investigation presented information about the toughness and failure mechanisms of these films, and contribute to optimize the arrangement of the coatings for a given application.

2 MATERIAL AND METHODS

The samples were made of SRV2W tool steels. The CrAlSiN coatings were applied using the PVD process. In order to observe the effect of the mechanical anchoring of the coatings, samples surface topography was controlled using three different surface finishing processes before coating deposition: Polishing (P); Micro Blasting (MB); and Micro Blasting followed by Polishing (MBP). The *Ra* values of the substrate surfaces were: P – 0.120 ± 0.032, MBP - 0.262 ± 0.025 and MB – 0.541 ± 0.017 µm.

The CrAlSiN coating was deposited by a partner *Platit do Brasil*, using a Platit π300 P + DLC equipment. The temperature used was 350 ºC.

Two approaches were used for the design of the coatings: multilayered coatings and gradient coatings. In both coating architectures, the initial composition (substrate interface) was kept constant at 70% of CrN and at 30% of AlSi, and the surface composition (CrN/AlSi ratio) was varied. More information about those samples are presented in Table 1.

Table 1. Samples features.

Group	Sample	Architecture	Final composition (surface)
1	P1;MB1;MBP1	Multilayer	30% CrN/70% AlSi
2	P2;MB2;MBP2	Multilayer	30% CrN/70% AlSi
3	P3;MB3;MBP3	Multilayer	50% CrN/50% AlSi
4	P4;MB4;MBP4	Gradient	50% CrN/50% AlSi
5	P5;MB5;MBP5	Gradient	30% CrN/70% AlSi

The measurement of micro hardness (50 gf) was performed using a HMV Micro Hardeness Tester with a Knoop indenter. In order to isolate the possible effect of surface finish, only polished samples were analyzed.

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The coating thicknesses were determined by microabrasion tests with a 25 mm ball and 3 µm diamond abrasive in different positions on the sample [7]. Ten tests were made in each group. It was used the values of measured crater diameters *a* and *b*, Fig. 1, and the Eq. (1).

$$t = \frac{b^2 - a^2}{8R} \quad (1)$$

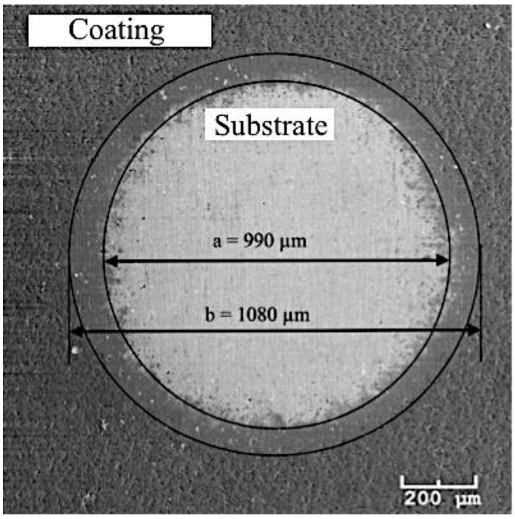
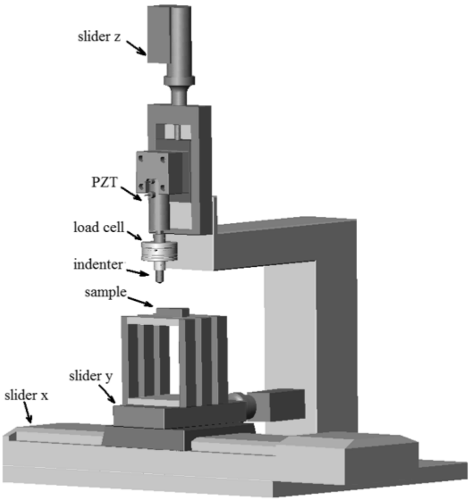


Figure 1. Wear crater made using diamond particles.

The progressive load scratch test was performed on a device specially developed for this purpose [8-10], Fig. 2. Vickers indenter was used. All scratches had 5 mm in length and the normal load varied linearly from 0 to 13 N. The first crack position along the scratch was observed by optical microscopy. This position was associated to normal force curve obtained during progressive scratch test, providing the value of critical load. It was performed three scratches in each sample.



(a)


(b)

Figure 2. Scheme of the equipment (a); Equipment used to make the grooves (b) [8-10].

In order to compare Lc results, a test of hypothesis (t-test) were applied with 95% of reliability.

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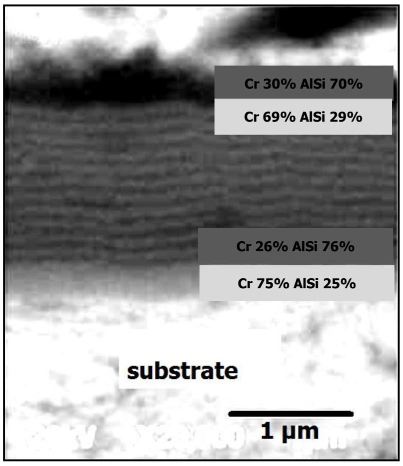
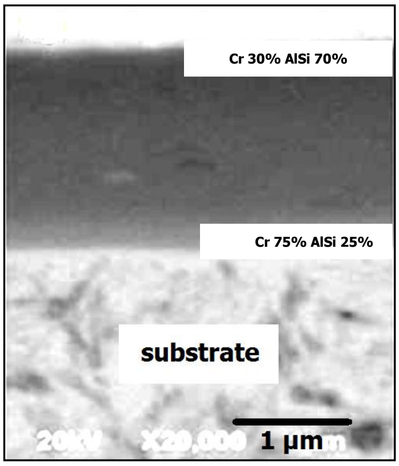
3 RESULTS AND DISCUSSION

3.1 Coating Characterization

Cross sections of gradient and multilayer samples obtained by the PVD process are presented in Fig. 3. In order to highlight contrast between the layers, this image was obtained using scanning electron microscopy in a special combination of secondary electron and backscattered detectors. Fig. 3a shows the CrN rich layer (light) and the AlSi rich layer (dark) intercalated. Fig. 3b presents the gradient structure of the coating, in which the light region close to the substrate is CrN rich and the dark region near the surface is AlSi rich.



(a)



(b)

Figure 3. Examples of cross sections: (a) multilayer coating and (b) gradient coating [11].

The microhardness of the coatings was measured using the Knoop method. The load used was 50 gf. At this load, the substrate hardness made a contribution to the overall hardness value, depending on the coating thickness, so the values should therefore not be used to compare the coatings directly. These results are presented in Tab. 2. The thickness is also shown in the Tab. 2.

Table 2. Hardness and coating thickness.

Group	Thickness [µm]	Hardness [GPa]
1	2.6±0.13	20.9±1.2
2	3.1±0.16	26.9±2.6
3	3.7±0.13	29.9±2.8
4	2.2±0.15	25.6±2.6
5	3.9±0.13	32.3±4.4

Comparing group 2 and 5, the gradient coating hardness was greater than that of the multilayer coating, as presented in Tab. 2. These results may be due to the higher thickness of group 5. The samples in group 5 were 25% thicker than those in group 2. Literature shows similar result for TiAlSiN PVD coatings [2, 12], where the gradient coating had a greater hardness than the multilayered coating.
The smaller amount of AlSi on the multilayer coating surface of group 3 (50% of AlSi) resulted in a greater hardness in comparison with that group 2 (70% of AlSi). This fact was related to the amorphous structure of SiNx that happened in group 2 which was less hard than nanocrystals of CrAlSiN [3, 12].
The lesser hardness of group 1 was associated to the lesser thickness of the coating.

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3.2 Scratch Test

The purpose of this test is to evaluate the critical load which tested coating present the first crack inside the scratch mark. In this case, the value of the critical load is related mainly with the toughness of the coatings [13]. An example of this method is presented in Fig. 4, showing the first crack within the scratch to P2 sample.

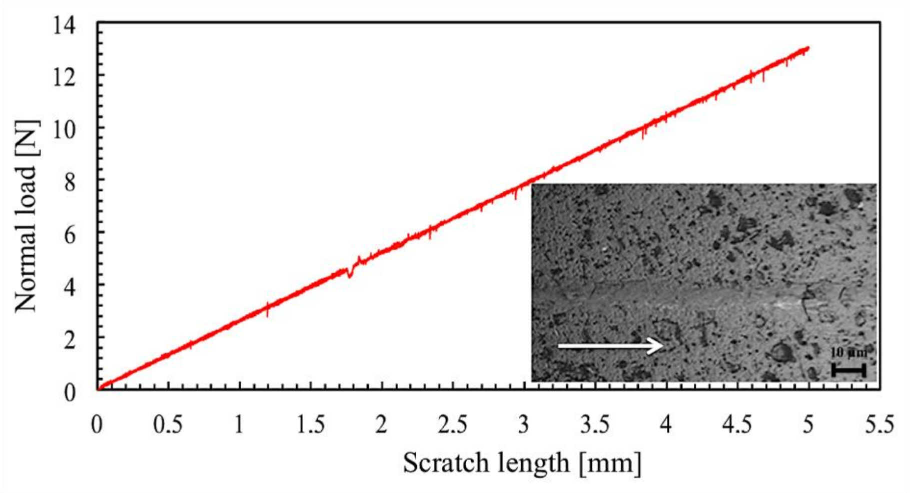
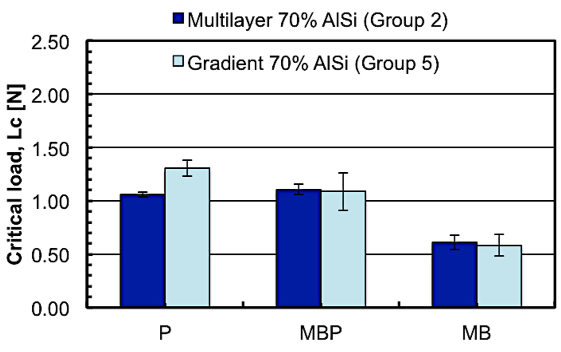
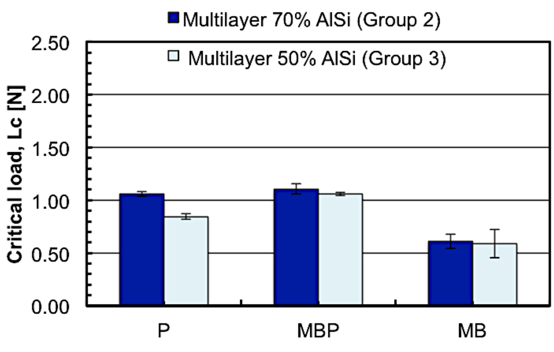


Figure 4. Normal load and tangential load as a function of distance.

The results of critical load, Lc, are shown in Fig. 5. In all cases MB had the lower values of Lc.
The effect of coating architecture is evaluated comparing groups 2 and 5, as presented in Fig. 5(a). Figure 5(b) shows the effect of the final chemical composition of the multilayer coating in critical load. The influence of the coating thickness in the critical load is presented in Fig. 5(c). Figure 5(d) shows the critical load values of groups 1 and 4, where both chemical composition and architecture was changed.
Critical load, Lc, decreased with the increase of the substrate roughness, Fig. 5(a). The P5 (gradient) sample showed a higher value of Lc compared to the sample P2 (multilayer). For MBP and MB samples the critical load was statistically identical, Harry, *et al*. (1999) [14] found similar results for critical load comparing gradient multilayer coatings.



(a)



(b)

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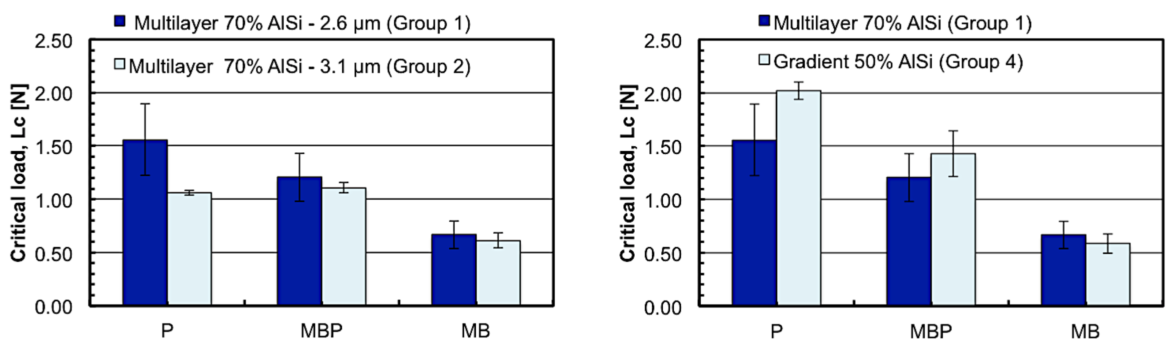
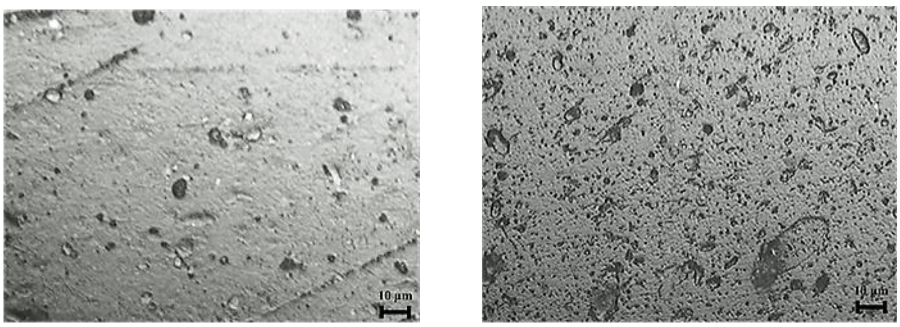


(c) (d)

Figure 5. Critical load, Lc, for all the samples studied as function of surface finishing.

In Fig 5(b), it was observed that, for polished substrate, group 2 showed higher values of Lc. This result was associated to the higher hardness and lower toughness of the multilayer coating with CrN 50% and 50% AlSi (group 3). For MBP and MB substrates the values of Lc were statistically similar.

It was noted in Fig 5(c) that the critical load was affected by the roughness and by the coating thickness. It was also observed that for the MBP and MB substrate the value were similar. For polished substrates the thinner coating presented higher values of critical load in accordance with the literature [15]. This result may be related to the presence of defects (pores) in P2 coating, as shown in Fig. 6 micrographs.



(a) (b)

Figure 6: The sample surface (a) P1 e (b) P2. OM.

The presence of larger pores on the sample P2 as shown in Fig. 6 was related to the deposition process characteristics. It was reported [16, 17] that the density of the coating decreases with increasing thickness due to the presence of defects during PVD process.

Figure 5(d) showed that the P4 sample had the highest value of Lc of the studied samples. It was also observed that the Lc value of MBP and MB samples presented similar values.

Both gradient coatings, with polished substrate, had higher hardness compared to multilayer coatings. Analyzing Fig. 5(a) and 5(d), critical loads of gradient coatings were greater than multilayer coatings. These results indicate that the gradient coatings also presented higher toughness than multilayer coatings. Harry, et al, (1999) [14] reported that the partial detachment of multilayer coatings affected the coating resistance. These authors concluded that the morphology of gradient coatings is more attractive for mechanical applications in comparison to the multilayer coatings [14].

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4 CONCLUSION

The objective of this work was to investigate and to compare the mechanical behavior of CrAlSiN coatings in different architecture (multilayered and gradient), by using scratch tests. We could conclude that:

- The progressive scratch test technique, using Vickers indenter, was capable to evaluate the mechanical properties of the coatings.
- The gradient coatings have higher hardness and toughness than multilayer coatings.
- The increase in the Si content in the multilayer coating surface (group 2) increased the values of critical load.
- Increasing the thickness does not always increase the substrate/coating toughness. Therefore the presence of internal defects resulted from deposition process affected negatively the substrate/coating adhesion.

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