Effect of load and indenter diameter on the amount of film cracks developed during the indentation of coated systems

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Abstract. The deposition of wear resistant thin films represents an usual procedure to improve the tribological behavior of mechanical components and parts. Depending on the application, the fracture toughness of the film plays a key role and different methods were developed to evaluate this mechanical property. Recently, the film fracture toughness was related to the distance between circular cracks generated during the spherical indentation of coated systems with soft substrates. In this work, analyses were conducted to further understand the phenomenon of circular crack formation during indentation, including the effect of load and indenter diameter. Films of chromium nitride (CrN) were deposited onto AA 6061 aluminum substrates through a commercial physical vapor deposition (PVD) process. Coated specimens were later indented by spheres with diameters from 1.6 to 6.4 mm, which applied normal loads from approximately 50 to 500 N. Similar amounts of circular cracks were obtained when an increase in indenter diameter was associated with an increase in normal load.

Keywords: thin films, hardness, circular cracks, ,fracture toughness.

1. Introduction

The deposition of wear resistant thin films on different substrate materials is a common practice to improve the tribological (wear and friction) behavior of different components and parts. For this reason, it is necessary and important to evaluate the coated systems mechanically. A simple method for the characterization of these systems is the indentation test (Weppelmann et all 1996), where indenters with different geometries are used, such as spheres, cones or pyramids (Ma, 1995; Souza, 2001; Hainsworth, 2003). In coated systems, the selection of spherical indenters is frequently preferred over those with pyramidal geometry since the former is associated with a reduction of the stresses in the film. However, the maximum principal stress increases as the diameter of the indenter increases, indicating that the use of larger indenters may not be an effective option to avoid crack development during the indentation process (Gan et all 1996).

The indentation test is also used to understand the mechanisms governing failure in coated systems (Ma, 1995; Weppelmann, 1996; Gan, 1996; Thomsen, 1998; Begley, 1999; Souza, 2001; Karimi, 2002; Abdul-Baqi, 2002; Hainsworth, 2003; Simunková, 2003). This type of study analyzes the development of contact stresses during the process, under which the coated systems can fail due to a loss of film/substrate adhesion, a failure designated as adhesive, or due to the fracture of the film, a failure designated as cohesive (Souza, 2001; Hainsworth, 2003).

In terms of the film fracture toughness, some indentation techniques are based on the propagation of cracks from the corners of pyramidal indentation marks. However, depending on the film properties, cracks are not always observed at the low loads necessary to prevent an influence from the substrate. The fracture toughness has also been evaluated through microindentation methods (Kodali et all 1997), but the loads applied in such cases usually result in penetration depths that are larger than the film thickness, such that the result may be a measurement related to the toughness of the coating-substrate system Alternatively, an instrumented indentation method was proposed, which is based on steps observed on the curves of depth of penetration as a function of applied load (Li, 1997; Li, 1998 e Li, 1999). However, the steps in those curves are not always observed during low load indentations conducted on thin film systems.

As consequence of the indentation, arrangements of circular cohesive cracks may be observed near the perimeter of the impression Fig. (1). This type of cracks is caused by tensile radial stresses present near the indentation edge. In some cases, radial cracks occur both inside and outside the indentation zone (Thomsen, N.B et all 1998) and usually, circular cracks are the preferred cohesive fracture pattern in systems with hard thin films deposited onto a ductile substrate (Begley, 1999; Souza, 2001).

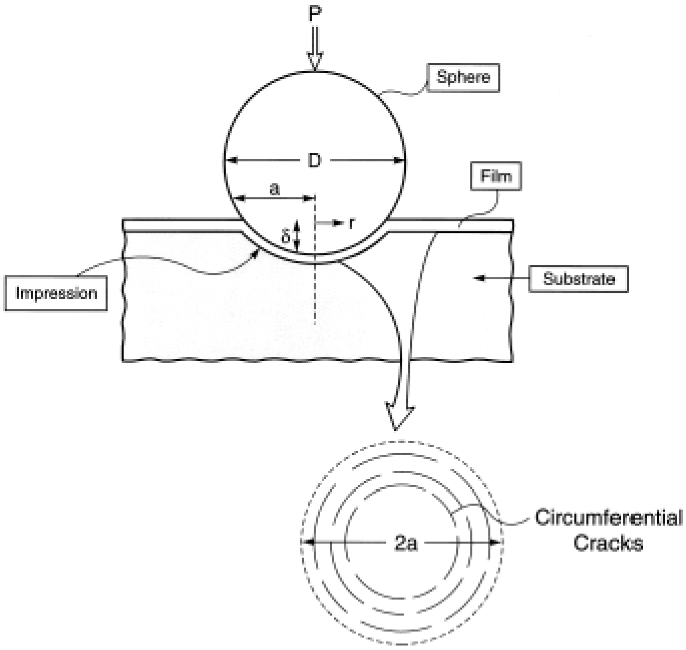


Figure1. Representation of indentation model and circular cracks generated in the inside of the indentation zone (Begley et all 1999).

In many cases, the propagation of circular cracks was associated with the amount of substrate indentation pile-up, which represents the material that moves upward at the indentation corner Fig. (2)

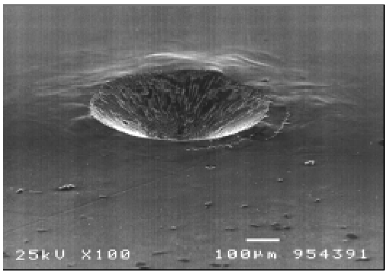


Figure 2. Plastic deformation of the substrate (Pile-up) taking place and extending approximately one indent diameter away from the edge of the indent (Thomsen et all 1998).

Experimental works have also been conducted to study the circular cracks at the contact edge of an indentation (Ma, K.J. 1995; Thomsen, N.B. 1998). However, apparently only little attention was placed on the circular cracks, which may carry important information regarding the characteristics of the film. In a previous work (Souza et all 2001), it was demonstrated that the number and location of circular cracks may be related to several factors, which include the geometry, the friction coefficient between the film and indenter, the substrate-film mismatch in elastic modulus, the level of film residual stresses and the film fracture toughness.

In this work, a series of indentations was conducted using indenters with spherical geometry, which applied normal loads on a system with soft substrate coated with a wear resistant thin film. The main objective was to further understand the mechanical behavior of the coated system under indentation.

2. Experimental Procedure

The specimen substrate was a piece of AA 6061 aluminum with dimensions of 16.6 x 12 x 3 mm^3 (Schematic on Fig. (3a)), which was polished until mirror finish. A film of chromium nitride (CrN) with thickness of approximately 4 μm was deposited onto the aluminum substrate at Brasimet Comércio e Industria S.A, following a commercial physical vapor deposition (PVD) process at low temperature. Figure (3b) presents the characteristics of the film surface, when observed in an optical microscope.

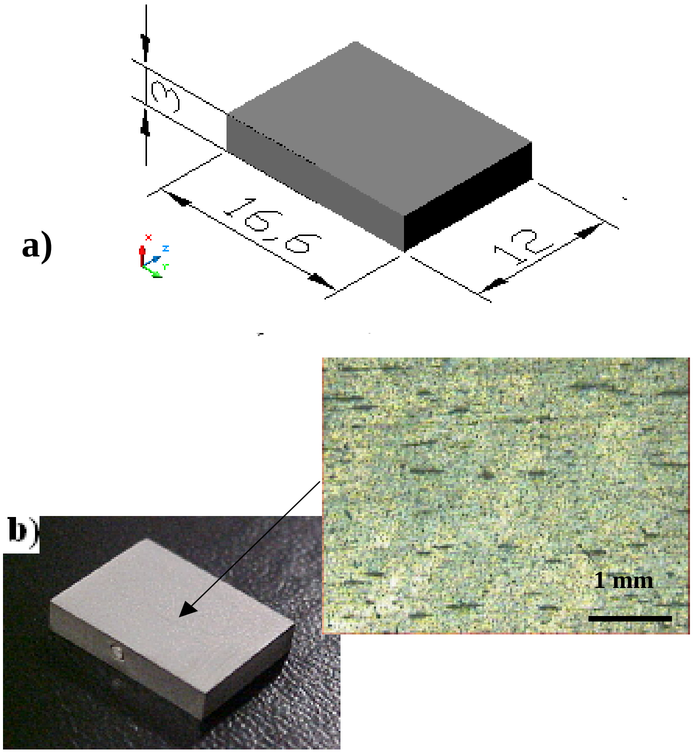


Figure 3. Coated system CrN-Aluminum. a) Specimen dimensions in milimeters b) Superficial aspect of the CrN coating obtained through a commercial PVD process.

The loads used in the indentation test were 9.8, 49, 98, 196.1, 294.2, 490.3 N. Spheres with diameters of 6.3, 3.2, 1.59 mm (1/4, 1/8 and 1/16 inch) were used in the indentations, which are diameters of spheres used in different Rockwell hardness tests. Indentations were conducted in a BUEHLER VMT-7 hardness tester equipment and Figure (4) presents the superficial quality of the spheres used.

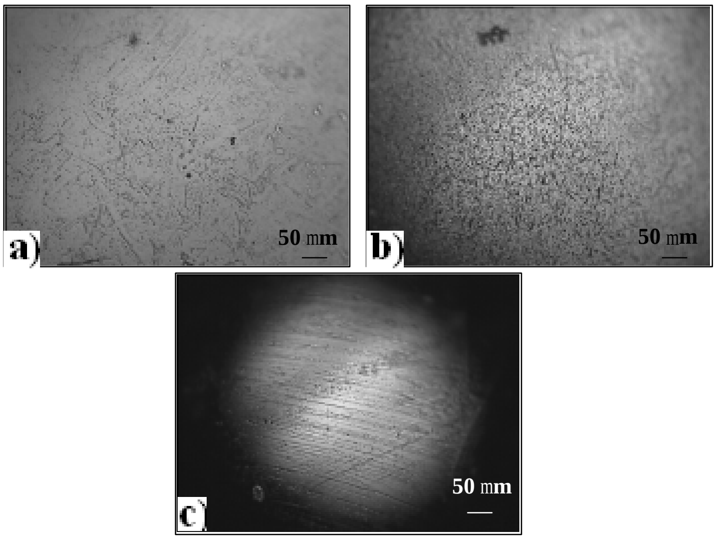


Figure 4. Superficial aspect of the spherical indenter. a) Sphere of 6.3 mm b) Sphere of 3.2 mm c) Sphere of 1.59 mm.

After testing, the indentation marks were analyzed through optical and scanning electron microscopy (SEM). The amount of pile-up in each indentation was evaluated with a roughness equipment SURFCODER IF 1700α. This equipment was set at a roughness measurement configuration, wich allows the measurement of indentation pile-up, but does not allow the measurement of the indentation depth.

3. Results and Discussion

The optical microscopy analysis indicated that, independently of the diameter of the sphere, no circular cracks were observed with loads of 98 N and lower.

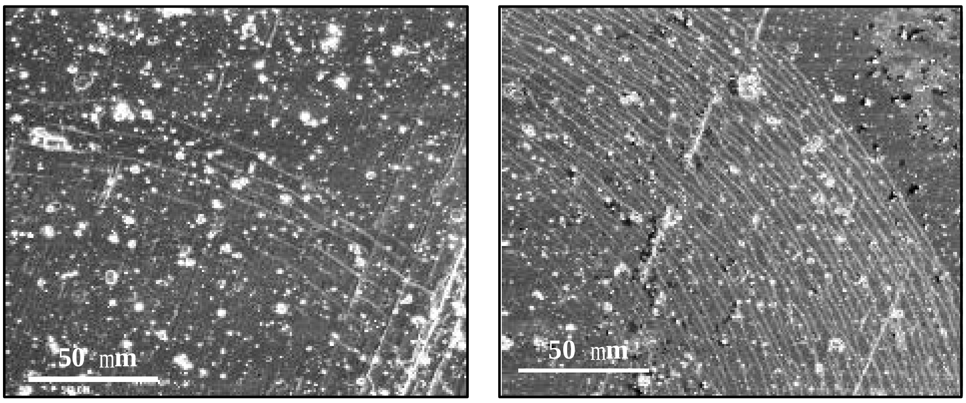
Figure (5) presents the indentation marks obtained with different indenter diameter and with the loads that resulted in circular crack formation. In each of these cases, the number of circular cracks was roughly measured, indicating that a similar amount (from 10 to 15) of cracks was generated for the conditions on the secondary diagonal of Tab. (1).

Figure 5. Indentation marks generated in the coated system during indentation with three spheres (Diameters of 6.3, 3.2, 1.59 mm) and three load levels (196.1, 294.2, 490.3 N).

Table 1. Number of cracks obtained as a function of the load and indenter diameter combinations.

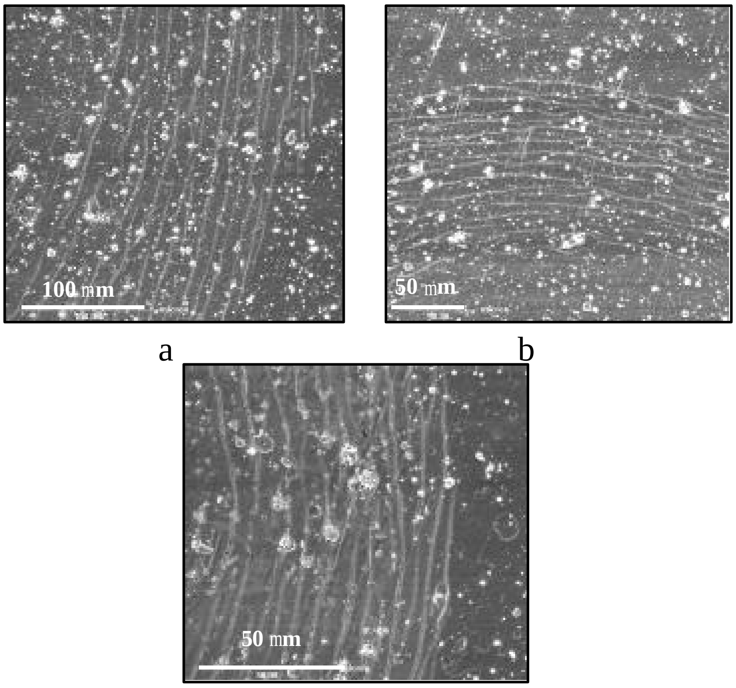
Load (N) \ **Indenter**	**6.35 mm**	**3.17 mm**	**1.59 mm**
196.1	≤5	≤5	10-15
294.2	≤5	10-15	>30
490.3	10-15	>30	>30

Figure (6) shows circular cracks obtained inside the contact region for load-indenter combinations above and below the secondary diagonal of the matrix presented in Tab. (1). For combinations above the secondary diagonal only few circular cracks (approximately 5 or less) were observed (Fig. (6a)). More than 30 circular cracks were observed at the conditions below the secondary diagonal (Fig. (6b)). Figure (7) shows the number of cracks obtained for the load-diameter combinations on the secondary diagonal of Tab. (1).



a) b)

Figure 6. Number of cracks observed by scanning electronic microscopy (SEM) in the inside to the contact area of indentation. a) Indentation doing with indenter of 3.17 mm and load of 196.1N b) Indentation doing with indenter of 3.17 mm e load of 490.3 N.



a b

c

Figure 7. Cracks observed by scanning electronic microscopy (SEM) inside the indentation mark. a) Indentation with indenter of 6.3 mm and 490.3 N b) Indentation with indenter of 3.2 mm and 294.2 N c) Indentation with indenter of 1.59 mm and 196.1 N.

Figure (8) shows the heights of the pile-ups obtained with different load-diameter combinations. As expected, with the same indenter, an increase in normal load resulted in an increase in the height of pile-up. Figure (8) also indicates that pile-up heights were similar for the conditions on the secondary diagonal of Tab. (1). Higher pile-ups were measured below this secondary diagonal and lower pile-ups were found in the conditions above this diagonal. Figure (9) presents differences in pile-up height for conditions with different indenter diameter and the same load.

According to the literature (Weppelmann, 1996; Begley, 1999 e Souza, 2001), film circular cracks observed on the edge of an indentation are associated with the bending of the film. Fig. (10) presents the results of FEM analyses conducted previously (Souza, 1999), in which film element are shown in the original (black) and after indentation displacements (white). In Fig. (10) it is possible to observe that film deformation generates tensile radial stresses at film surface (point c) and compressive stresses at the interface (point d). Following an analogy with a bar that is bended, higher tensile stresses should be expected either when the amount of bending is increased or when the bending curvature is decreased. During an indentation, the amount of bending is mainly controlled by the height of indentation pile-up, and curvature is also affected by indenter diameter. An analysis of the conditions along the secondary diagonal Tab. (1), which had a similar behavior in terms of the amount of circular cracks, indicates that the pile-up height was similar in the three load-diameter combinations. This fact suggests that, when compared to normal load and indenter diameter, pile-up height is the predominant factor in determining the tensile radial stresses and, consequently, the amount of indentation circular cracks.

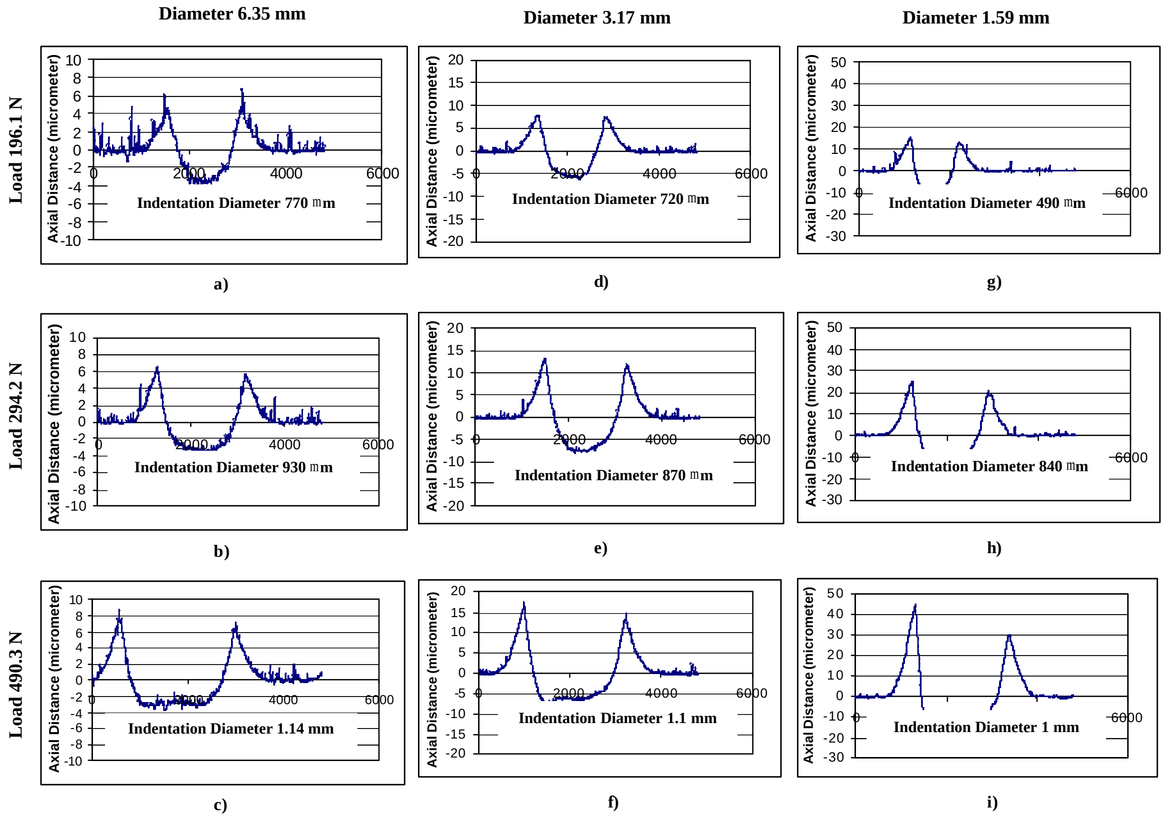
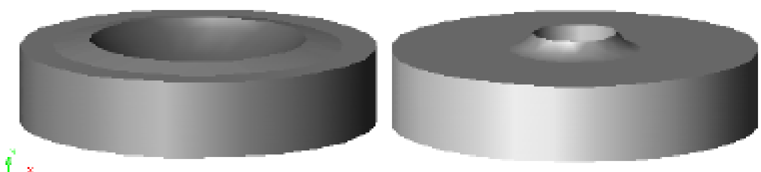
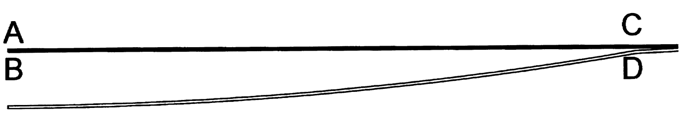


Figure 8. Quantification of pile-up generated in each one of the indenter-load combinations. a) Diameter of 6.35 mm and load of 196.1 N b) Diameter of 6.35 mm and load of 294.2 N c) Diameter of 6.35 mm and load of 490.3 N d) Diameter of 3.17 mm and load of 196.1 N e) Diameter of 3.17 mm and load of 294.2 N f) Diameter of 3.17 mm and load of 490.3 N g) Diameter of 1.59 mm and load of 196.1 N h) Diameter of 1.59 mm and load of 294.2 N i) Diameter of 1.59 mm and load of 490.3 N



a) b)

Figure 9. Representation of pile-up height. Load of 196.1 N a)Diameter of 6.35 mm b) Diameter of 1.59 mm



a

b

Figure 10 Bending of the film. FEM results obtained during the 50 N normal indentation of a system with different wear resistant films and an elastic-plastic 6061 aluminum substrate. Original (black) and displaced (white); (a) Thickness t = 0.6 μm and (b) Thickness t = 4.6 μm (Souza, 1999).

4. Conclusions

This work confirmed that the indentation of coated systems with substrates presenting an elastic-plastic behavior can result in series of circular cracks inside the contact region of the indentation. The number of circular cracks was related to different factors, such as the diameter of the indenter and the applied normal load.

For the load-diameter combinations studied in this work, the height of the indentation pile-up was the most important factor that determined the amount of circular cracks that propagated in each case.

5. Acknowledgements

The authors would like to thank BRASIMET Comércio e Indústria S.A for the help in the deposition of (CrN) thin films. The authors of this work Patricia B. G. de Lamadrid and Eduardo A. Pérez R, acknowledge the São Paulo Research Support Foundation (FAPESP) for the financial support through processes 02/02579-1 and 01/10864-5, respectively.

6. References

Abdul-Baqi, A., Van der Giessen E., 2002, "Numerical analysis of indentation-induced cracking of brittle coatings on ductile substrate", International Journal of Solids and Structures, Vol. 39, pp. 1427-1442

Begley, M.R., Evans, A.G., Hutchinson, J.W., 1999, "Spherical impression of thin elastic films on elastic-plastic substrates", International Journal of Solids and Structure, Vol. 36, pp. 2773-2788

Gan, L., Ben-Nissan, B., Ben-David, A., 1996, "Modelling and finite element analysis of ultra-microhardness ndentation of thin films", Thin Solid Films, Vol. 290-291, pp. 362-366

Hainsworth, S.V., Soh, W.C., 2003, "The effect of the substrate on the mechanical properties of TiN coatings", Surface and Coatings Technology, Vol. 163-164, pp. 515-520

Karimi, A., Wang, Y., Cselle, T., Morstein, M., 2002, "Fracture mechanisms in nanoscale layered hard thin films", Thin Solid Films, Vol. 420-421, pp. 275-280

Kodali, P., Walter, K.C., Nastasi, M., 1997, "Investigation of mechanical and tribological properties of amorphous diamond-like carbon coatings", Tribology International, Vol. 30, pp. 591-598

Li, X., Diao, D., Bhushan, B., 1997, "Fracture mechanisms of thin amorphous carbon films in nanoindentations", Acta Materialia, Vol. 45, pp. 4453-4461

Li, X., Bhushan, B., 1998, "Measurement of fracture toughness of ultra-thin amorphous carbon films", Thin Solid Films, Vol. 315, pp. 214-221

Li, X., Bhushan, B., 1999, "Evaluation of fracture toughness of ultra-thin amorphous carbon coatings deposited by different deposition techniques", Thin Solid Films, Vol. 355-356, pp. 330-336

Ma, K.J., Bloyce, A., Bell, T., 1995, "Examination of mechanical properties and failure mechanisms of TiN and Ti-TiN multiplayer coatings", Surface and Coatings Technology, Vol. 76-77, pp. 297-302

Simunková, S., Bláhová, O., Stepánek, I., 2003, "Mechanical properties of thin film-substrate systems", Journal of Materials Processing Technology, Vol. 133, pp. 189-194

Souza, R.M., 1999, "Finite Element Modeling of contact stresses during the indentation of wear resistant coatings on sosft substrates", Ph. D. Thesis, Colorado School of Mines

Souza, R.M., Sinatora, A., Mustoe, G.G.W., Moore, J.J., 2001, "Numerical and experimental study of the circular cracks observed at the contact edges of the indentations of coated systems with soft substrates", Wear, Vol. 251, pp. 1337-1346

Thomsen, N.B., Fischer-Cripps, A.C., Swain, M.V., 1998, "Crack formation mechanisms during micro and macro indentation of diamond-like carbon coatings on elastic-plastic substrates", Thin Solid Films, Vol. 332, pp. 180-184

Weppelmann, E., Swain, M.V., 1996, "Investigation of the stresses and stress intensity factors responsible for fracture of thin protective films during ultra-micro indentation test with spherical indenters", Thin Solid Films, Vol. 286, pp. 111-121

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