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RESEARCH

PVD Coatings' Strength Properties at Various Temperatures by Nanoindentations and FEM Calculations Determined

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A B S T R A C T

Nanoindentation is usually applied on thin films at ambient temperatures for hardness determination. Recently, instruments for conducting nanoindentation at elevated temperatures have been developed facilitating measurements up to 700 °C. Both indenter and specimen, if necessary, are heated in an inert atmosphere to avoid film oxidations. In the described investigations, nanoindentations were conducted on cemented carbides and high speed steel specimens, coated with various films, up to 400 °C. The obtained results were subjected to statistical analysis to estimate their reliability. Moreover, the results were evaluated by appropriate FEM (Finite Element Method) algorithms for determining the coatings' elasticity modulus, yield and rupture stress as well as hardness at various temperatures. The results reveal a non-linear temperature dependence of the coating properties.

1. INTRODUCTION

The determination of the temperature effect on the PVD coatings' properties is pivotal, among others, for avoiding coatings' overstressing during cutting and for adapting the cutting conditions to the coatings' properties [1,2]. Tensile tests [3] and tribological ones [4] have been applied for characterizing coating strength properties and friction coefficients respectively, at ambient and elevated temperatures. The possibility to conduct "in situ" nanoindentation measurements by appropriate instruments [5,6] has contributed significantly in determining accurately coatings' strength properties in a wide range of temperatures.

The aim of presented studies was to investigate the influence of temperature on the mechanical properties of various PVD coatings. In this context, nanoindentation was conducted at different temperatures between 25 °C and 400 °C. In each coating and temperature case, an appropriate number of nanoindentations were

3. RESULTS AND DISCUSSION

3.1 Nanoindentation measurements at ambient temperature

Nanoindentation results at ambient temperature into the TiAlN coating, up to a maximum load of 15 mN, are exhibited in Fig. 2a. Since the specimens' roughness affects the nanoindentation curves, 30 measurements per nanoindentation experiment were conducted for stabilizing the moving average of the maximum indentation depth [7]. The determined mean load-displacement curve of the examined coating is also displayed in Fig. 2a.

The reliability of these results was checked with the aid of statistical methods, by the standard deviation s and the coefficient of variation CV versus the indention load (see Fig. 2b).

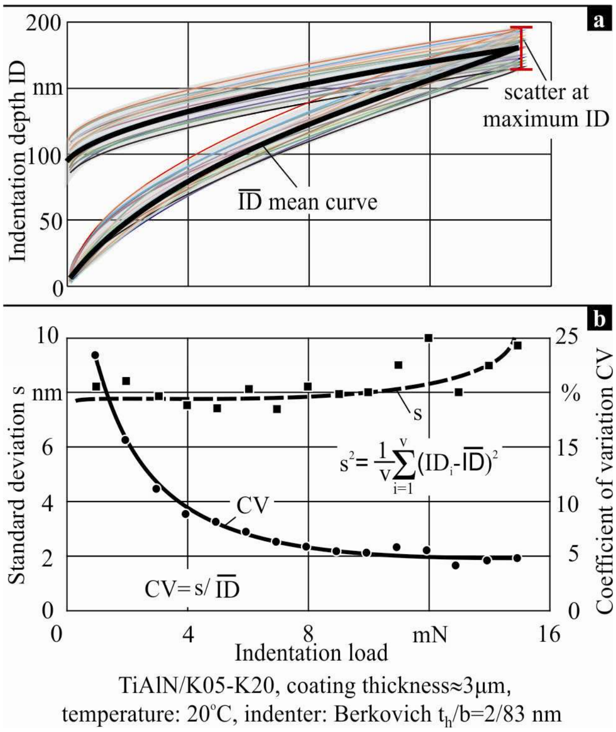

Fig. 2. (a) Nanoindentation results and their mean value's curve, (b) Standard deviation and coefficient of variation of the attained maximum indentation depth at various loads.

The coefficient of variation represents the ratio of the standard deviation to the mean, and it is a useful parameter for comparing the degree of variation of data series, as for example of nanoindentation curves. On one hand, the augmentation of the coefficient of variation CV at low indentation depth restricts the minimum indentation load, at approximately 4mN in the present case, for attaining CV values less than 10%. On the other hand, the standard deviation s growth over a nanoindentation load of approximately 15 mN, sets an upper limit to this load, for keeping the deviation s under ca. 10 nm. At higher indentation loads, this fact might be associated to the bigger volume of the deformed coating material, which includes larger coating structure inhomogeneities. Considering these tendencies, the applied maximum indentation load was adjusted to 15 mN, for obtaining a sufficient results reliability.

3.2 Nanoindentation measurements at elevated temperatures

Further nanoindentations into the TiAlN coating were carried out at a maximum load of 15mN, at various temperatures. The course of the mean indentation depth (ID) at the maximum load versus the temperature is displayed in Fig. 3a. The developed ID scatter versus the temperature, is also shown in the figure. It is visible that the indentation depth depends on the applied temperature. At a temperature of about 150 °C, a reduction of the indentation depth appears, compared to ambient temperature. The indentation depth grows between 200 °C and 300 °C and is diminished again at approximately 400 °C. The later ID diminution is larger, compared to the corresponding one at 150 °C.

In order to evaluate the reliability of these results, the standard deviation as well as the coefficient of variation versus the temperature was determined and both are presented in Fig. 3b. The standard deviation and the coefficient of variation remain at a low level and almost invariable up to 250 °C, whereas a slight increase takes place over a temperature of approximately 300 °C. This can be explained, considering that the indenter is heated locally and the rest of isolated device space remains at room temperature. In this way, an air or inert gas flow slight increase from the device space towards the indenter area can contribute to the indentation depth standard deviation augmentation. Considering the obtained results, from statistical point of view, the nanoindentation is sufficiently reliable up to the temperature of 400 °C.

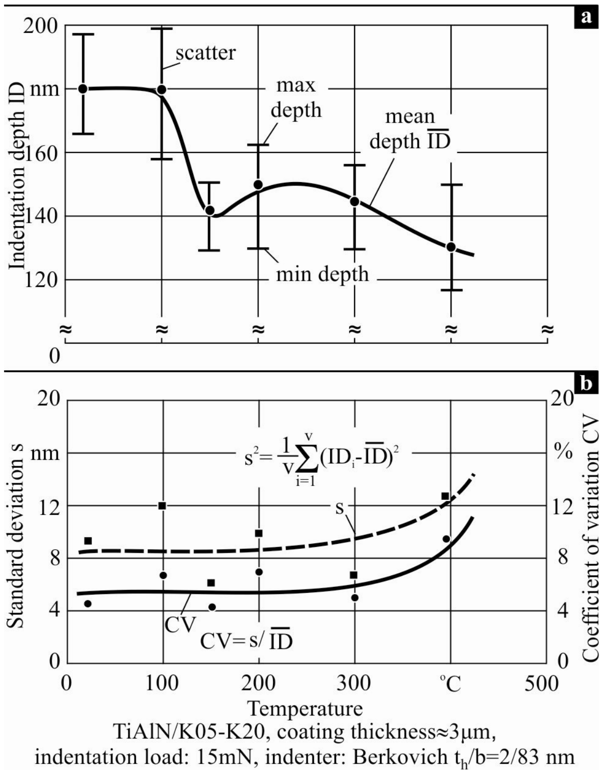

Fig. 3. (a) Maximum indentation depth of the examined TiAlN coating at various temperatures, (b) Standard deviation and coefficient of variation of the maximum indentation depth at various temperatures.

The described changes of the indentation depth versus the temperature can be attributed mainly to dislocations movements, caused by the temperature augmentation. In a polycrystalline material, the crystallographic orientation changes from one grain to the next through a narrow transition zone, or grain boundary, which acts as an effective barrier to slip. Dislocations pile-ups along the active slip planes at the grain boundaries oppose the generation of new dislocations. When the temperature increases, the shear stress developed at the head of the dislocation pile-up can become large enough to cause dislocation movement across the boundary. The dislocation pile-up is mainly responsible for strain-hardening of the material in the early stages of plastic deformation [13]. According to the obtained results, this mechanism is probably active up to a temperature of about 150 °C, since the restricted thermal energy might cause dislocation pile-ups in the grain boundaries, but not significant dislocation movements within the grains, due to the restricted number of dislocations per grain, in the case of nanostructured PVD coatings. Thus, a hardness improvement develops. At temperatures over 150 °C, the dislocation movements are facilitated by higher thermal energy. In this way, stress nucleation is avoided and the coating hardness deteriorates [14]. Over ca. 200 °C to 300 °C, it is assumed that dislocations are arrested at obstacles, owing to solutes arriving by diffusion (dynamic strain ageing) [15] and a further coating strength enhancement occurs. At higher temperatures, in a very fine grained material, the relaxation of residual stresses and of grain boundary sliding are responsible for coating's softening [4, 16].

3.3 Evaluation of nanoindentation results for determining stress strain curves

Employing the "SSCUBONI" algorithm [7], the coating's stress-strain curves at various temperatures were calculated and they are demonstrated in Fig. 4a. The coating's elasticity modulus, the yield and the rupture stress are monitored versus the temperature in Fig. 4b. On one hand, the coating elasticity modulus remains unaffected by the temperature growth up to 400 °C. On the other hand, the yield and rupture stress are not stable when the temperature increases.

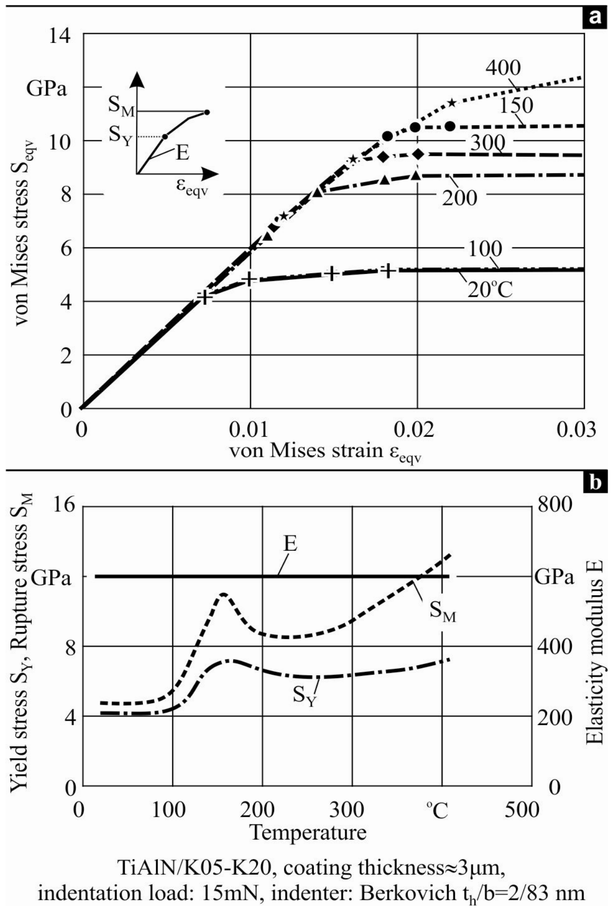

Fig. 4. (a) Calculated stress – strain curves of the examined TiAlN coating at various temperatures, (b) Yield and rupture stress as well as elasticity modulus of the examined TiAlN coating at various temperatures.

More specifically, their non linear courses versus the temperature possess two maxima at approximately 150 °C and at 400 °C.

Impact tests, jointly with further analytical evaluation methods, contributed in estimating the temperature dependent film fatigue endurance stress at literature [17]. A correlation between yield and fatigue endurance stress versus the temperature, by different experimental and analytical methods determined, reveals an impressive convergence.

This tendency is also valid for the Vickers hardness H, the H/E ratio as well as for the ratio H^3/E^2, which are displayed in Fig. 5. All curves have a maximum at approximately 150 °C. Over 300 °C an increasing tendency develops, up to the temperature of 400 °C, due to the described dislocations and solutes movements.

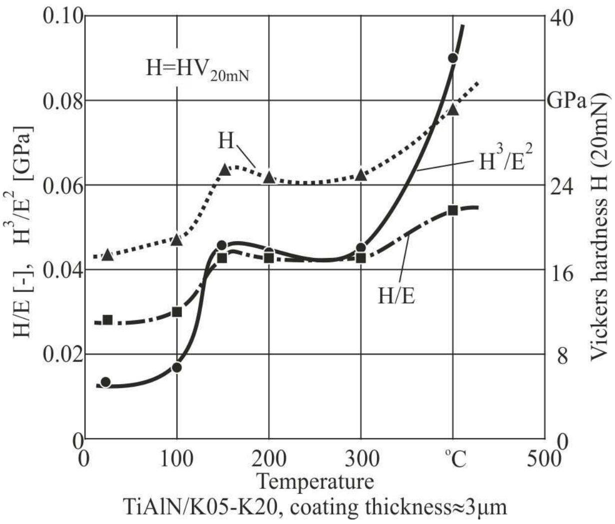

Fig. 5. Hardness and the H/E and H^3/E^2 ratios of the TiAlN coating versus the the temperature.

3.4 Temperature dependent mechanical properties in various coating cases

Nanoindentations at various temperatures between 25 °C and 400 °C were conducted on further PVD films, at a maximum indentation load of 15 mN. The related results are monitored in Fig. 6a.

In the case of the two layer TiAlN/TiSiN film, the maximum indentation depth versus the temperature is not affected by the TiAlN sublayer. This happens, since the stress fields at the indentation load of 15 mN do not exceed a depth of 1.6 μm and the TiSiN top layer thickness amounts to ca. 1.8 μm (see Fig. 6b).

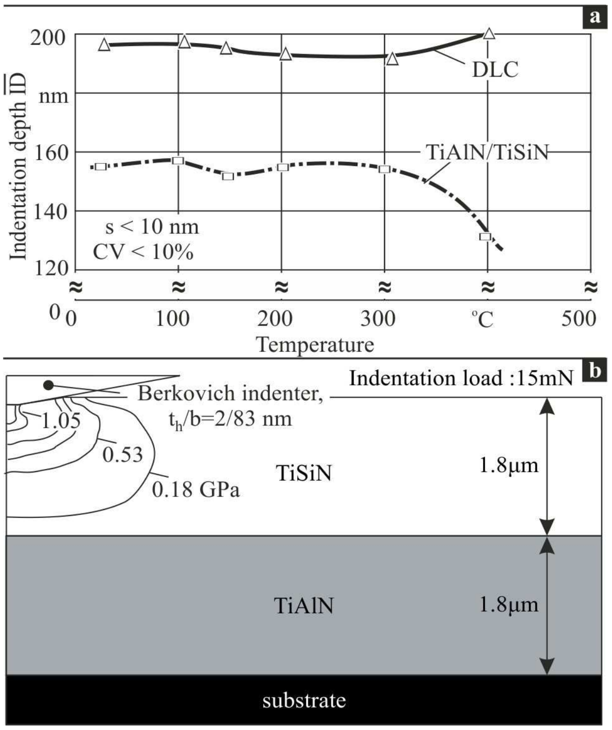

Fig. 6. (a) Maximum indentation depths of the examined films at various temperatures, (b) Stress field in the TiSiN/TiAlN coating at an indentation load of 15mN.

The temperature dependence of the ID, is similar, as in the case of the TiAlN coating, previously described. However, the reduction of the indentation depth at the temperature of 150 °C is less affected, compared to the corresponding one of the investigated TiAlN coating. This can be attributed to the Si content, which reinforces the film structure and hinders the dislocations movements up to the temperature of 300 °C.

Finally, in the case of the DLC coating, the maximum indentation depth remains almost stable in the investigated temperature range. This does not converge with macro-scale experiments using the impact test [17]. A graphitization in the DLC material occurs (transformation from sp3 to sp2 phase), at temperatures between 100 °C and 400 °C, which deteriorates the mechanical properties. Considering the nanoindentation results, the sp3 fraction is not significant in a restricted superficial nano-scale coating region.

The elasticity modulus, the yield and rupture stress of the aforementioned films, were calculated and they are demonstrated in the corresponding diagrams of Fig. 7.

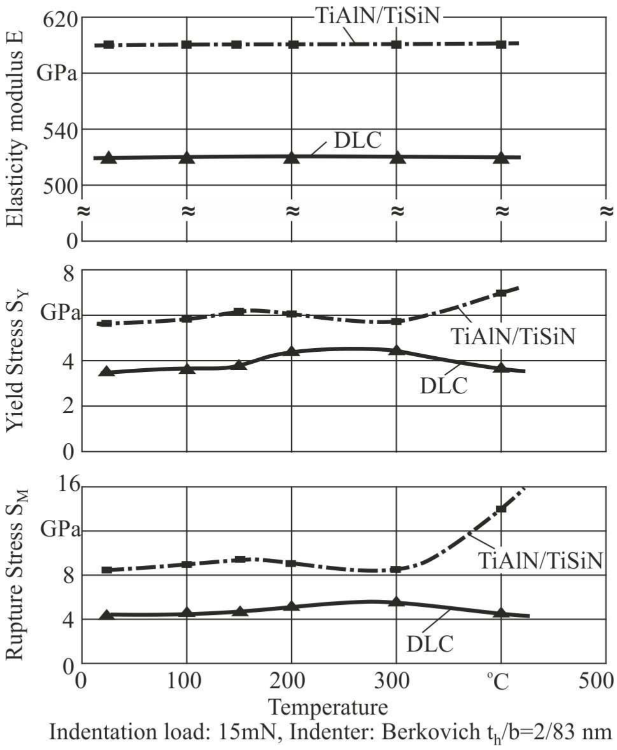

Fig. 7. Temperature effect on the elasticity modulus, yield and rupture stress of the examined films.

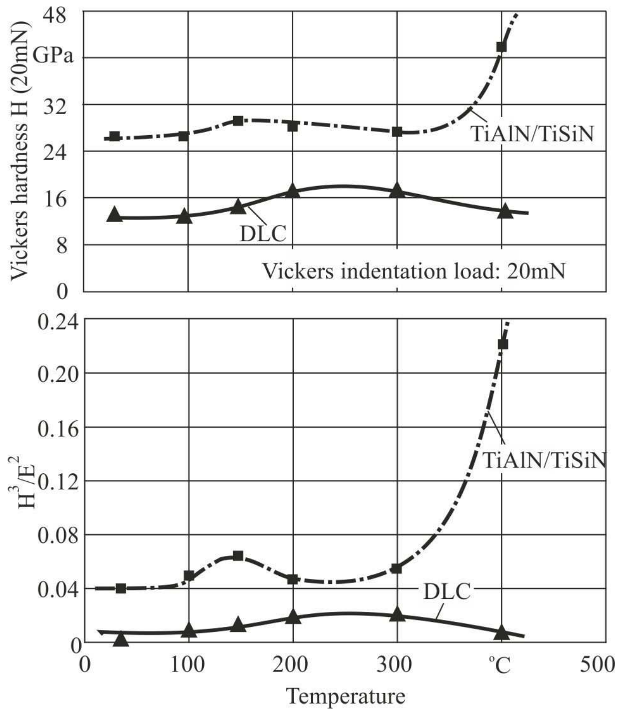

Fig. 8. The hardness and H^3/E^2 ratio versus the temperature of the examined films

According to these results, the elasticity modulus of both examined film materials, remain stable versus the temperature. The yield and rupture stresses of the two layer's coating TiAlN/TiSiN behaves similarly, as in the case of the TiAlN film. Finally, a slight augmentation of the yield and rupture stress of the DLC coating between 200 °C and 300 °C can be observed. The latter tendency is also applicable for the Vickers hardness and for the ratio H^3/E^2 of the DLC coating versus the temperature, which are displayed in Fig. 8. Moreover, in these diagrams, the corresponding data of the TiAlN/TiSiN film are exhibited, demonstrating the same behavior as the TiAlN coating (see Fig. 5).

4. CONCLUSIONS

Nanoindentations were conducted on various PVD coatings at ambient and elevated temperatures. The related results were subjected to statistical analysis for checking their reliability that was found to be sufficient, within a certain range of the indentation load. Moreover, the coatings' strength properties such as elasticity modulus yield and rupture stress as well as hardness at various temperatures were calculated. A non-linear temperature dependence of the coatings' properties was revealed, which is explained by dislocation movements. This effect can be exploited in various practical applications, such as in coated cutting tools.

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