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Nanoindentation of CVD diamond: comparison of an FE model with analytical and experimental data

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Abstract

This paper describes an experimental procedure for the determination of the hardness and the elastic modulus through nanoindentation of a CVD diamond coating using simple analytical formulae. Such tests, performed with a Berkovich indenter, were simulated by finite element analysis. Through the numerical analysis, it was possible to reproduce the load-penetration depth curves and thus, confirm the validity or correct the property calculations. Results show that the predicted property values can be affected by the assumed material strain hardening. By comparing the numerical values with the experimental results, it is possible to characterise, with sufficient accuracy, the material behaviour. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Nanoindentation; Hardness; Elastic modulus; CVD diamond

1. Introduction

CVD technology developed over the last 20 years permits the deposition of continuous coatings on large surfaces (diameter larger than 300 mm [1]) with thickness greater than 3 mm. It is thus possible to take advantage of the properties of diamond [2] in many engineering applications for which a higher resistance to corrosion and erosion is required [3]. However, the high hardness has imposed a limit on the knowledge of the properties of this material. The relatively new test technique of instrumented nanoindention offers a potential route to evaluating the elastic and plastic properties of this material. Initial work shows these properties to be strongly influenced by the quality of the coating and rely heavily on the correct execution of the test and interpretation of the experimental results.

This paper describes an experimental procedure for the determination of the hardness and of the elastic modulus through nanoindentation of a CVD diamond coating with a Berkovich indenter. It also compares these experimental results with analytical and numerical results.

2. Theoretical fundamentals

The elastic modulus can be determined through a procedure originally proposed by Loubet et al. [4] and by Doerner and Nix [5] and subsequently developed by Oliver and Pharr [6]. It is based on the elastic analysis,

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Table 1
Elastic modulus for 10 μm thick CVD diamond on WC at an applied load of 70 mN

Test number	Elastic modulus E_r (GPa)	Elastic modulus E_f (GPa)	Hardness H (GPa)
1	304.9	413.3	18.6
2	462.6	771.6	69.6
3	333.8	468.6	38.2
4	272.3	355.3	34.7
5	491.2	855.1	78.6
6 (pop-in)	346.1	493.3	11.2
7	518.3	941.2	70.7
8	501.7	887.6	41.1
9	400.2	611.8	47.2
10	445.5	725.0	59.4

given in the same table. The calculation was carried out assuming the values of the elastic constants for the indenter are those reported in the literature [2], that is, a Young's modulus of 1141 GPa and a Poisson's ratio of 0.07. The same value 0.07 was assumed for ν_f.

The results for the 10-μm coatings at 70 and 100 mN show an average E_f of 615 GPa and $H = 53 \pm 24$ GPa. Since the residual deformation is relatively small, an error in the evaluation of the area A, or of the effective indentation h_p, would significantly affect the values of E_r and H. In each case, the determined values of E_f and H exhibit a degree of spread analogous to that obtained by Savvides and Bell [13].

The same tests were conducted on 100 μm thick coatings. The average values of the elastic modulus and the hardness for the maximum load of 70 and 100 mN where, $E_f = 775$ GPa and $H = 93.5 \pm 25$ GPa, respectively. The results obtained for the thicker specimens show a slight increase in elastic modulus and a large increase in hardness, possibly reflecting the fact that the specimen was of superior quality with lower defect levels and impurities.

4. Numerical model

The simulation of the nanoindentation test was carried out using NASTRAN, a general purpose finite element code. With the aim to reduce the computational effort and the modelling time, the same simplification introduced in [12] was adopted, namely that of considering the Berkovich indenter as a cone with the vertex angle of 140.6°. Assuming that the specimen was cylindrical, it was possible to take advantage of the axisymmetric geometry and the analysed model consisted simply of one sector of 10°. In section, the indenter is a triangle with a base of 5 μm and vertex angle of 70.3°, while the specimen is a square with a side of 10 μm. The tip of the vertex of the indenter has a radius of 50 nm. The dimensions of the indenter and the specimen were selected in order to limit the dimensions of the problem but also, to eliminate any boundary effects such as those reported in [9] in connection to the extent of the damaged zone.

For simulating the indenter-specimen contact, lines of contact or 'slidelines' [14] were used. In order to be able to apply the procedure described above, it was necessary to adopt the Ramberg–Osgood constitutive law for the specimen material. Moreover, it was assumed that the indenter deforms only elastically. The values of the elastic constants of the indenter were those of the natural diamond, already mentioned in Section 3. For the specimen, values of the elastic modulus equal to 468 GPa and the same value for the Poisson's ratio as that of the natural diamond were used. From the test results on the CVD diamond reported in Section 3, the value of E_f of 468 GPa corresponds to a value of $E_r = 333.8$ GPa and a hardness of 38.2 GPa. Since the ratio H/E_r turned out to be less than 0.16, it was thus possible to determine the elastic limit using Eq. (6). This last value, associated with the value 0.07 of the characteristic plastic strain, permits the determination of, apart from the strain-hardening coefficient, the constant β appearing in Eq. (7). The constitutive relation of the material was entered in the computer program by approximating the σ–ε curve represented by Eq. (7) with a multilinear law.

A resultant load of 70 mN was applied as a uniform pressure on the upper surface of the indenter. The load used is conservative with respect to the value of 250 mN indicated in [7] as at that limit load cracks would form for indenters Vickers and Berkovich. The hardness test was simulated by increasing the load from zero to the value of 70 mN and subsequently returning to zero.

The analyses were conducted for four different values of the strain-hardening coefficient, n, varying from 0.667 to 0.8, keeping the elastic limit constant. For each n, both loading and unloading curves were determined and these are shown in the Fig. 1 along with the corresponding experimental curves. The degree of agreement between the numerically derived and the experimental curves depends on the choice of n. The predicted loading curves appear to be well simulated by the appropriate choice of n. However, the predicted unloading curve is associated with considerable plastic (permanent) deformation, which is not consistent with experimental results. Thus, a different non-linear elastic model for unloading needs to be developed.

5. Conclusions

Simple analytical formulae have been used to generate initial values of material constants from experimental data. These values were subsequently introduced into a finite element simulation for the nanoindentation of CVD diamond coatings. Comparisons between the simulated and experimental load–indentation curves have been used to indicate the corrections required to the material model.

The Ramberg–Osgood law was used to describe the material behaviour. The effect of the strain hardening coefficient on the determination of the load indention curve and consequently on the hardness and relative elastic modulus was assessed. By continuous comparison with experimental data it was possible to calibrate the model and describe, with sufficient accuracy, the material behaviour.

The simulation permitted the reconstruction of the residual stresses and effective profile of the indentation. Using contact elements it is easy to include friction between the contacting surfaces. This was tried and found to have a low influence on the load-displacement curve.

Variation of the hardness and elastic modulus was assessed for various applied loads using the finite element model. The load at which the substrate properties become important has yet to be determined.

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