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Multiple cohesive cracking during nanoindentation in a hard W-C coating/steel substrate system by FEM

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ABSTRACT

Stress evolution and subsequent cohesive cracking in the hard and stiff W-C coating on steel substrate during nanoindentation have been investigated using finite element modelling (FEM) and eXtended FEM (XFEM). The FEM simulations showed that the maximum principal stresses in the studied system were tensile and always located in the coating. They evolved in several stages. At indentation depths below 15% of the relative indentation depth, the maximum principal tensile stresses of ~3 GPa developed at the top surface of the coating along the indenter/coating interface. At relative depths range 15–60%, the maximum tensile stresses of ~6–8 GPa concentrated under the indenter tip in the coating along the interface with the substrate. At relative depths exceeding 60%, the maximum stresses gradually increased up to 10 GPa and they were located in the sink-in zone outside the indent as well as below the indenter tip. The first and subsequent cohesive cracks developed when the maximum tensile stresses in the sink-in zone at the top surface of the coating (and at the coating/substrate interface under the indenter) repeatedly reached the ultimate tensile strength of the coating. The hardness profile as well as cohesive cracking is controlled by the deformation of the substrate defined by the ration of the yield stresses of the coating and substrate. Very good correlation between the experimentally obtained cracks and multiple cracks predicted by XFEM confirmed the ability of the applied modelling in the prediction of fracture behavior of the studied coating/substrate system.

1. Introduction

The measurement of hardness of thin coatings by nanoindentation or instrumented indentation technique remains a challenging theoretical and experimental task due to an inevitable influence of the substrate. The interactions of the substrate and coating during nanoindentation are usually described by the dependencies of the hardness and indentation modulus on indentation depth called hardness and indentation modulus depth profiles. Their shape depends on the ratio of the mechanical properties of both constituents: hard and stiff coatings on a softer ductile substrate exhibit maximum at small (<10% of coating thickness [1–5]) relative indentation depths, and then the values gradually decrease to asymptotically approach to the properties of the substrate. The situation is reverse in the case of a softer coating on hard and stiff substrates [4]. To extract the data corresponding to the coating from that of the composite coating/substrate system, several theoretical models applicable to the experimentally obtained depth profiles have been developed. These models are based on the analytical approach supported by a statistically sufficient set of experimental measurements [1,2,6,7], on the contact mechanics [8,9] or on a finite element modelling (FEM) [10–19]. All the theoretical models contain fitting parameters. Relatively recent comparison of such models indicated that the reliability of the obtained values is inversely proportional to the number of fitting parameters [19]. The result is that the simplest Jőnsson-Hogmark's model [1] with only two parameters provided higher reliability compared to the models with more parameters.

Numerical methods of nanoindentation in coated systems are based on finite element modelling (FEM) verified vs. the experimental nanoindentation curves [10–19]. FEM with some modifications can predict not only the nanoindentation behavior but also cohesive and adhesive cracking in the coatings. It requires input of either critical stress or critical energy as the criteria for crack nucleation and growth [20]. Then, the interfacial toughness [21,22] and fracture toughness of the coatings can be calculated [23,24]. The nucleation and propagation of the cohesive cracks is usually calculated within the eXtended FEM (XFEM) [25]. The XFEM is even able to determine fracture toughness of the coatings [17,18,26]. In our recent work on W-C coatings on steel substrate

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[18], gradual evolution of the maximum stresses in different areas with regard to the indenter position and coating/substrate and a single cohesive crack development in the sink-in zone were demonstrated, when the ultimate tensile strength of W-C coating was achieved in this zone. Based on the comparison of the experimental load – indentation depth curves with the pop-in attributed to the first crack formation and iteratively adjusted XFEM parameters, the fracture energy per unit area $G = 38.1\ \mathrm{J\,m^{-2}}$ and the fracture toughness $K_{IC} = 3.52\ \mathrm{MPa\,m^{1/2}}$ of the W-C coating were obtained [18].

Unfortunately, the nanoindentation experiments performed by both experimental and theoretical modelling on coating/substrate systems are influenced by a large number of additional factors involving indenter tip radius [27] as well as the ratio of the elastic and plastic limits of the coating and substrate [10,27,28], surface roughness [29], type of deformation behavior (pile-up or sink-in) [30], residual stresses [31] etc., besides single or multiple cracking [18,32] and delamination. The possibilities for the elimination of all these factors experimentally are limited and a computational approach seems to be the only way to quantify the influences of the above mentioned factors on nanoindentation.

According to our knowledge, the formation of multiple cohesive cracks was not investigated by FEM up to now. Therefore, and as a continuation of our previous works [18], the goal of the current study is to investigate the evolution of multiple cohesive cracks in the hard and stiff coating deposited on softer and more compliant substrate during nanoindentation by means of standard finite element modelling combined with extended FEM. The study was performed on W-C coating which exhibits an interesting combination of hardness, elastic modulus and tribological properties depending on the deposition conditions [34–39]. The FEM results were correlated with the real nanoindentation measurements on W-C coating/steel substrate system.

2. Experimental procedure

2.1. W-C coating deposition

The details of the deposition conditions for the preparation of the studied W-C coatings were described earlier [18]. Briefly, the substrates were thermally treated construction steel (12050) discs (diameter 25 mm, thickness ~3 mm) polished by diamond slurry to R_a ~10 nm. The depositions were carried out after plasma cleaning of the substrates in Cryofox Discovery 500 (Polyteknik, Denmark) system at the working pressure of 0.5 Pa. At first, around 50 nm thick Ti bond layer was deposited using DC magnetron sputtering. Subsequent W-C coatings with thickness around 1 μm were obtained after 34 min of deposition from the WC target using High Power Impulse Magnetron Sputtering (HiPIMS) source at 500 W average power, duty cycle $\alpha = 3\%$ (frequency f = 200 Hz and impulse length of $\tau = 150\ \mu s$) with the addition of 2 sccm of C_2H_2 into Ar atmosphere.

2.2. Nanoindentation

The nanoindentation tests were carried out on a nanoindenter (model G200 Agilent, USA) using a diamond Berkovich tip in a load controlled tests (up to 50, 350 and 400 mN) and in a continuous stiffness measurement (CSM) mode with the frequency 45 Hz and amplitude 2 nm up to the constant indentation depth of 1500 nm with the constant strain rate of $0.05\ \mathrm{s^{-1}}$. Special attention was paid to the sharpness of the indenter tip because of the possibility of high wear of the diamond tip in the case of multiple indentations of very hard coatings. Fig. 1 illustrates the profile of the diamond tip employed in the current tests obtained by scanning electron microscopy (SEM). The tip wear up to 77 nm occurred, which resulted in the tip curvature of 1.15 μm. Such a blunted tip was used also for modelling to obtain fair comparison with the experiment. The final hardness and indentation modulus depth profiles were obtained on a matrix of 4 × 4 indents by averaging individual curves of up to 16 tests. The Poisson's ratios of the steel substrate and W-C coating were assumed to be $\nu = 0.3$ [62]. Further details of the measurements conditions can be found in [18].

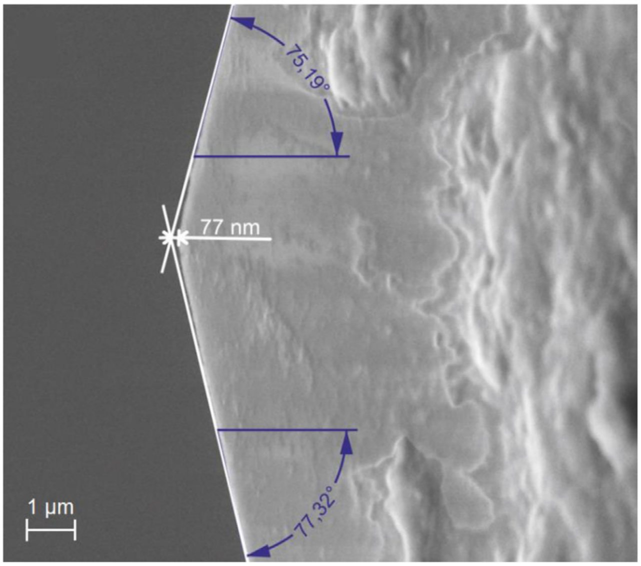

Fig. 1. The profile of the diamond indenter tip used in the nanoindentation experiments. The curvature radius of the indenter correspond to R = 1.15 μm.

The structure of W-C coatings, indent topography and coating cracking and cross sections were investigated using the scanning electron microscope (model Auriga Compact, Zeiss, Germany) combined with the focused ion beam (FIB).

2.3. Finite element modelling

Nanoindentation in the W-C/steel coated systems was simulated using ABAQUS Finite Element Method (FEM) software combined with the eXtended Finite Element (XFEM) module. Because full three dimensional (3D) modelling of nanoindentation with standard Berkovich tip is time consuming, the much faster two-dimensional (2D) simulations of the nanoindentation with an ideal conical indenter tip with a semi-angle of 70.3° were preferred. This choice was justified by two facts: conical indenter exhibits the same contact area to depth ratio as in the Berkovich tip; the FEM results between 2D and 3D simulations on corresponding indenters exhibited differences within 3%. Such differences were considered sufficiently small and 2D modelling representative also for the 3D case. Three dimensional calculations were therefore limited to the visualization of the spatial distribution of principal stresses and cracks and for comparison with the real nanoindentation tests. The tip curvature of the indenter used in FEM was intentionally set to 1.15 μm in agreement with the tip used in the experiments (see Fig. 1).

The FEM calculations were carried out assuming an isotropic elastic – perfectly plastic behavior and von Mises yield criterion for the plastic flow. The Poisson's ratio $\nu = 0.3$ was set constant for both materials; the input parameters for standard FEM included only Young's moduli of both substrate ($E_s = 210$ GPa) and coating ($E_c = 300$ GPa) and yield stresses $Y_s = 1.1$ GPa and $Y_c = 9.0$ GPa, respectively. Roller-boundary conditions were applied along the axis of symmetry and fixed-boundary conditions were used at the

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