Surface & Coatings Technology 237 (2013) 135–141

Contents lists available at ScienceDirect

Surface & Coatings Technology

journal homepage: www.elsevier.com/locate/surfcoat

Fatigue strength of diamond coatings' interface assessed by inclined impact test

K.-D. Bouzakis [a,b,*], G. Skordaris [a,b], E. Bouzakis [a,b], S. Makrimallakis [a,b], S. Kombogiannis [a,b], O. Lemmer [c]

[a] Laboratory for Machine Tools and Manufacturing Engineering, Mechanical Engineering Department, Aristoteles University of Thessaloniki, Greece
[b] Fraunhofer Project Center Coatings in Manufacturing, in Centre for Research and Technology Hellas in Thessaloniki, Greece and in Fraunhofer Institute for Production Technology in Aachen, Germany
[c] CemeCon AG, Germany

ARTICLE INFO

Available online 25 September 2013

Keywords:
NCD coatings
Inclined impact test
Thermal residual stresses
Interface fatigue strength

ABSTRACT

The inclined impact test is an efficient method for characterizing the fatigue strength of interfaces of nanocrystalline diamond coatings (NCD). During this test, an oscillating oblique load induces repetitive shear stresses into the region between film and its substrate. Inclined impact tests were conducted on NCD coated specimens with a thickness of ca. 5 μm at forces up to 850 N and 1.5 million loading cycles. The related imprints were evaluated by confocal microscopy measurements and EDX micro-analyses. Dependent on the applied load, after a certain number of impacts, damages in the film interface region develop resulting in coating detachment. In this way, residual stresses of the film are released leading to its lifting (bulge formation). The bulges are destroyed by further repetitive impacts and the coating is totally removed. The geometry of the developed NCD coating's bulges can be effectively described by appropriate Finite Element Method (FEM) supported calculations. Based on the attained impact test results, Woehler-like diagrams were developed for monitoring the fatigue failure or endurance of NCD coatings at various impact conditions. Employing such diagrams, the fatigue strength of NCD coatings can be assessed. According to the attained Woehler-like diagrams, a threshold load of 730 N exists for the film delamination by fatigue in the investigated case.

1. Introduction

Multilayer nanocrystalline diamond coatings (NCD) on cemented carbide inserts are very efficient in machining non-ferrous materials like Al–Si alloys [1–4]. NCD coatings on hardmetal tools are characterized by elevated mechanical properties and hardness, however by high residual stresses too [5]. It has to be pointed out that residual stresses in NCD films are associated with numerous reasons such as epitaxial crystal differences as well as the significantly smaller thermal expansion coefficient of the diamond coating compared to its cemented carbide substrate. The latter parameter leads to compressive stresses in the film structure during the cooling of NCD coated specimens from CVD process temperature to the ambient one [4]. A key factor for ensuring a long life-time of NCD coated tools, especially in heavy duty applications, is the film bonding on its substrate. Among others, the bonding region of the film has to possess sufficient fatigue strength for withstanding successfully the repetitive cutting loads in the case of milling operations. Prior to the NCD coating deposition on cemented carbide substrates, appropriate treatments are conducted aiming at increasing the carbide surface roughness, depleting cobalt from film-substrate interface region and deactivating the non adhesive cobalt surface. Such procedures are commonly based on selective chemical etching [2,3]. The nucleation of the diamond is favored by the rough carbide surfaces. Another option to immobilize Co is by producing stable cobalt compounds, i.e. silicides and borides, which are quite stable under diamond deposition conditions. Selective etching of Co with various acids offers a wide range of different pre-treatment methods. The thickness of the etched zone is important, because the remaining porosity in the cemented carbide results in reduced layer adhesion [2].

Various techniques have been developed for evaluating the film adhesion such as Rockwell [6] and scratch tests [7]. In the literatures [8] and [9], using the inclined impact test, the interfacial toughness can be quantified by the ratio of the tangential to normal film-substrate interface stiffness and the critical shear failure stress respectively. A comprehensive study of the different adhesion testing techniques along with analytical models describing the coating delamination are introduced in [10] and [11]. Moreover, Kamiya et al. [12,13] have developed an appropriate experimental setup supported by a FEM model. This model calculates the interfacial toughness in terms of a critical energy release corresponding to a critical load for crack propagation.

The present paper describes the application of inclined impact test for assessing the fatigue strength of NCD coatings' interface. As already mentioned, due to the repetitive impacts, the film-substrate bonding is damaged during this test, and the coating residual stresses are released leading to its lifting and subsequent failure.

* Corresponding author at: Laboratory for Machine Tools and Manufacturing Engineering, Department of Mechanical Engineering, Aristoteles University of Thessaloniki, GR 54124 Thessaloniki, Greece. Tel.: +30 2310 996021, +30 2310 996079; fax: +30 2310 996059.
E-mail address: bouzakis@eng.auth.gr (K.-D. Bouzakis).

0257-8972/$ – see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.surfcoat.2013.09.030

2. Experimental details

CVD diamond coatings were deposited by a CC800/9Dia CEMECON coating machine on cylindrical cemented carbide specimens of HW-K05 ISO specifications (see Fig. 1) [14]. The coatings were produced by the hot filament method. The filament and substrate temperatures were 2000 °C and 900 °C respectively. The total pressure was 30 mbar, the carbon to hydrogen ratio equal to 1%, the coating rate 0.5 μm/h and the gas flow 2 l/min. The total coating time amounted to ca. 20 h. The film thickness was approximately 5 μm with multilayer nanocrystalline structure. The deposition time and the overall cooling time were approximately 10 h and 9 h respectively. The content of Co in the surface region of the substrate was reduced by chemical pretreatment. The cemented carbide substrate used is characterized by a low content of Co-binder phase (<6%). In Fig. 1a, the specimens' geometry and their roughness Rz, Rt and Ra are displayed. The substrate mechanical strength properties determined by analytical evaluation of nanoindentation measurements are shown in Fig. 1b. These properties were determined before the substrate etching. The monitored nanoindentation curve represents the mean value of 50 measurements for excluding roughness effects on the results' accuracy [15]. After approximately twenty measurements, the moving average of the maximum penetration depth is stabilized. The standard deviation at the maximum indentation depth amounts to approximately 10. This parameter decreases by diminishing the coated specimen roughness [15]. According to investigations presented in [16–18], the nanoindentation is a reliable technique to determine mechanical properties of various materials.

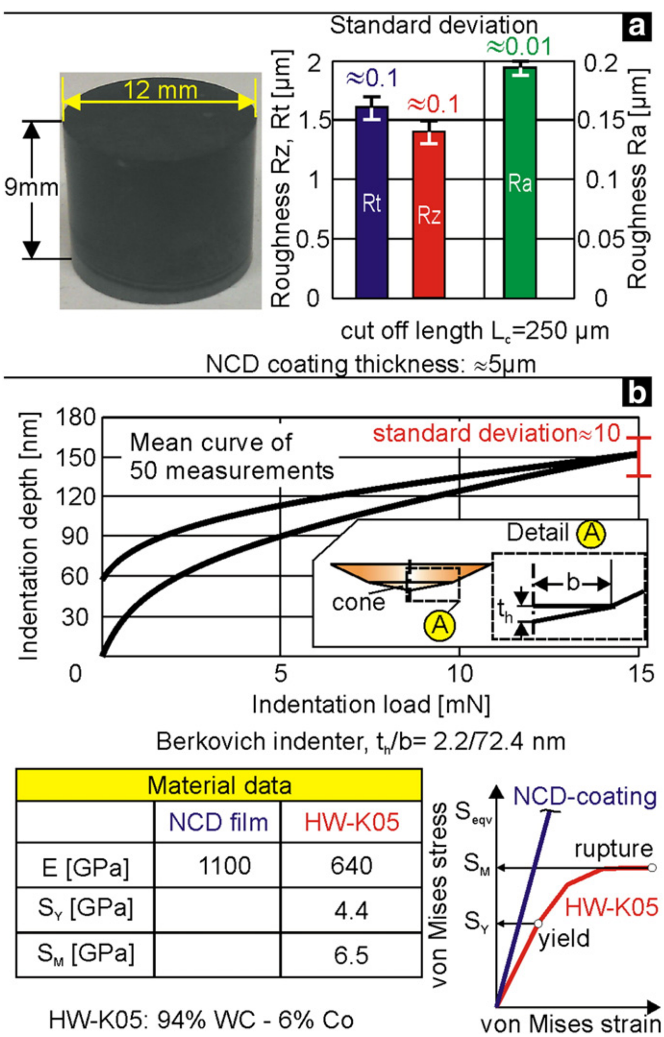

Fig. 1. (a) Coated specimens' geometry and roughness. (b) Mechanical properties of the applied materials.

The composition and structure of the NCD coated specimens were investigated by scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDX). A Joel JSM-840 scanning electron microscope (SEM), with EDX analysis facilities, was applied for conducting such investigations. Fig. 2 shows a SEM micrograph of a cross-section of the investigated NCD coated specimens. In the same figure, indicative EDX analysis results along the scan path in the coating and substrate are presented. According to these results, only carbon was detected in the coating region, whereas in the substrate mainly tungsten and cobalt exist. In the SEM micrograph, the film's surface elevation heights correspond to the roughness values, already described in Fig. 1.

The NCD films' adhesion fatigue strength was assessed via inclined impact tests at various loads and cycles. The load inclination angle was 15° and the ball diameter 5 mm (see Fig. 3a). At the start of a new test, an unused region of the carbide ball surface was employed. The impact tester device was designed and manufactured by the Laboratory for Machine Tools and Manufacturing Engineering of the Aristoteles University of Thessaloniki in conjunction with CemeCon AG. The load signal duration t_d and impact time t_e are displayed in Fig. 3b [19]. These were practically constant in all carried out experiments. The applied force increases progressively up to a set maximum value. The coating and substrate stresses, calculated by the methods described in [8], are demonstrated in Fig. 3c at the loads of 250 and 750 N. The colors correspond to certain ranges of the von Mises stresses according to the monitored scales at the figure bottom. When the maximum force is reached, the ball velocity is nullified, its direction changes and the indenter motion becomes upwards. According to the calculated stresses, the coating and the substrate are elastic

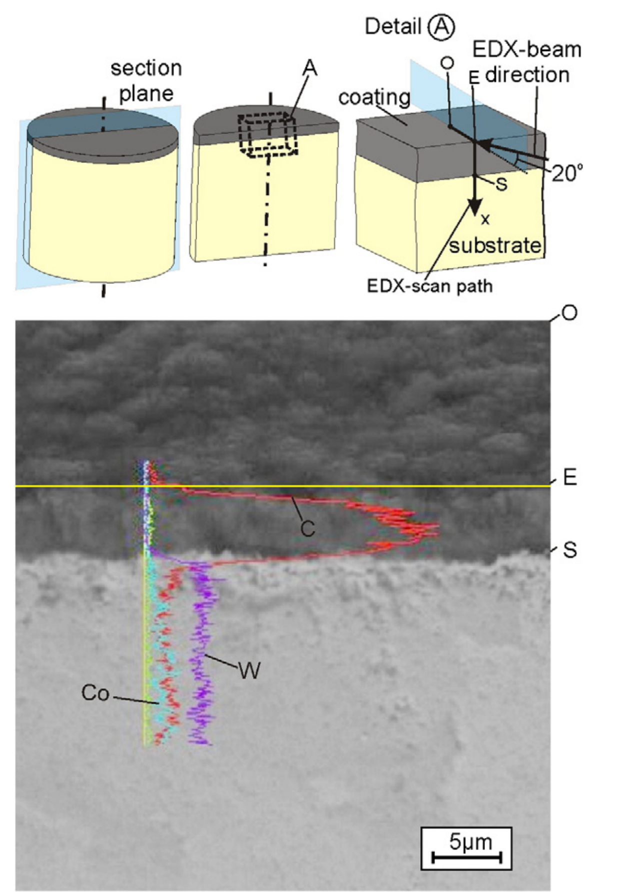

Fig. 2. SEM micro-graph and C, W and Co concentrations on a cross-section of the NCD coated specimens based on EDX analyses.