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Experimental and simulative strain field investigation of nano- and microscratches on nanolaminated (Cr, Al)N coating

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ABSTRACT

Micro- and nanoscratches were made on a nanolaminated CrN/AlN coating under different loads and velocities. The remaining vertical strain fields under the scratches were determined by measuring the bilayer periods in cross-fracture images using scanning electron microscopy. These results were compared with the remaining vertical strain fields obtained from scratch simulations utilizing the strain-rate-dependent material properties examined in preliminary work. The effect of the friction coefficient and residual stress on the strain and stress fields was investigated using simulations. The experimental and simulated strain fields show good quantitative agreement for the nanoscratches. Furthermore, the appearance of fractures and microcracks under the scratches could be correlated with strong shear stresses in simulations during indenter overrun. This method and the results obtained enable the stress–strain fields of coatings under dynamic mechanical loads to be determined quantitatively. Additionally, the possibility of determining failure criterions using scratch tests enables the simulative prediction of a coating's resistivity under real loading conditions.

1. Introduction

The chromium aluminum nitride coating system (Cr, Al)N is a promising hard coating system used to protect tools against adhesion and wear, for example, in plastic product manufacturing. Recently, considerable research on the effect of the deposition process, chemical composition and micro- or nanostructure on the coating's mechanical properties has been performed, and decisive improvements in the coating system have been made [1–3]. The coating's elastic properties with respect to their dependence on chemical composition [4], phase structure and orientation as well as layer thickness [5] have been investigated.

Recently, methods for obtaining the plastic flow curves of thin films based on nanoindentation and finite element method (FEM) simulations have been developed, and different coating systems have been analyzed. In preliminary work, the author determined the effect of the chemical composition and grain structure [6] of $Cr_xAl_{x-1}N$ coatings on plastic flow behavior. Furthermore, the strain-rate dependence of plastic flow was analyzed for pure and nanolaminated coatings [7], demonstrating a strong dependency of the material's strain rate hardening on grain size. Bouzakis et al. [8] investigated the temperature dependence of plastic flow for nanostructured TiAlN using a method similar to that used in this work and showed a nonlinear dependence of the plastic flow behavior on temperature, whereas the elastic properties remained constant.

Knowledge of the effects of chemical composition, grain structure and deposition parameters on the elastic–plastic properties of coatings facilitates the design of optimized coatings for different applications and substrate materials. Furthermore, the quantitative measurement of the elastic–plastic properties of a coating enables simulation of the coating's mechanical behavior under specific loads and the determination of the resulting strains and stresses. In this manner, the critical stresses inducing cracks or delamination of the coating can be determined.

In this work, the ability to determine materials' stress–strain fields under scratches using different types of indenters, loads and velocities based on simulations was investigated. Herein, the simulation results are compared with experimentally determined strain fields under the same scratch test parameters. The experiments and simulations were performed on a nanolaminated CrN/AlN coating, the strain-rate-dependent material properties of which were determined in preliminary work [7].

2. Experimental setup

2.1. Coating deposition

The coating analyzed in this study was deposited in a CC800/9 HPPMS unit from CemeCon AG, Würselen, Germany, equipped with two HPPMS Sinex 3.0 power supplies made by Chemfilt Ionsputtering AB. The substrate material was THM12, a tungsten carbide containing 6% Co and 2% TiC/TaC. The substrate sample was coated using a pure

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chromium (99.9% pure Cr) target and a pure aluminum (99.5% pure Al) target. The nanostructure of the CrN/AlN nanolaminate was synthesized using the two pure targets opposite to each other and running the deposition process with a single rotation type of the sample at 0.3 rpm. The duration of deposition was set to three hours. The coating was identical to that used to determine the material properties [6,7].

2.2. Material characterization

The indentations made to calculate the flow curves and the nanoscratches were performed using a nanoindenter XP (MTS Systems Corporation, Oak Ridge, Tennessee, USA). The indentations used to determine flow curves of the coating were made using a spherical indenter with a radius of approximately 10 μm, and the nanoscratches were made with a Berkovich diamond. The process by which these flow curves were determined is described in detail in [7].

The nanoscratches were formed by scratching with one edge of a three-sided, pyramidal Berkovich tip through the sample surface (Fig. 1). Scratches measuring 10 mm in length were produced under four different loads (10, 40, 160 and 640 mN) and at two different velocities (0.5 and 5 μm/s). To model the form of the indenter tip for simulation, the Berkovich indenter geometry was analyzed using a laser scanning microscope (Keyence VK-9700, Neu-Isenburg, Germany).

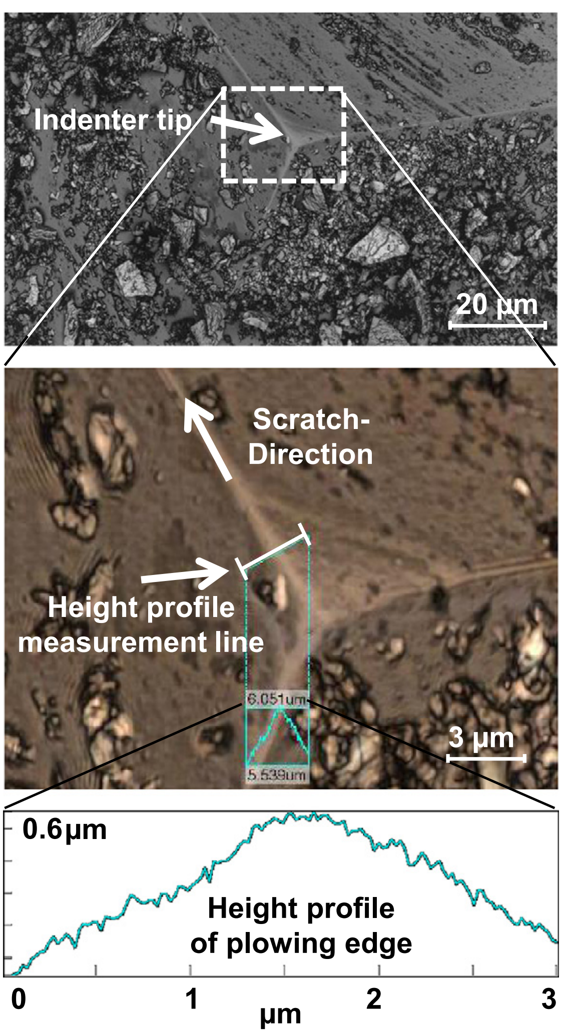

Fig. 1. Laser intensity (above) and laser-color mixed (middle) image of the Berkovich scratch tip. The graph at the bottom shows the rounding of the plowing edge.

Furthermore, microscratches measuring 10 mm in length were made using a CSM LSRH Revetest with a spherical diamond tip with a radius of approximately 200 μm. The microscratches were applied under loads of 2, 10 and 40 N and at a velocity of 1 mm/min.

All the scratches were analyzed on a cross-fractured sample using a ZEISS DSM 982 Gemini scanning electron microscope (SEM). The thicknesses of the CrN–AlN bilayers were determined in the deformed and undeformed areas using high-resolution images.

2.3. FEM simulations

The nanoscratches were simulated using the FEM program Abaqus 6.12 based on a geometric model of the indenter and the sample. The Berkovich indenter was modeled as a rigid body. The tip and edges of the ideal Berkovich geometry were rounded based on the results obtained by laser microscopy, resulting in a tip radius of 500 nm and an edge radius of 350 nm. The entire model was meshed using four-node linear tetrahedron elements. The mesh element size at the indenter tip was 50 nm. At this element size, no noteworthy changes in the simulation results could be observed with increasing mesh density. The sample model was 50 μm wide, 30 μm long and 10 μm thick. The sliding conditions between the indenter and sample were modeled to be frictionless because the real sliding coefficient was not known. The effect of sliding with different friction coefficients was exemplarily studied. The coating thickness was set to 3.75 μm. The simulation was performed in two steps. In the first step (the loading step), the load on the indenter was increased to the specific vertical force within 6 s. In the second step (the scratch step), the indenter was moved with a constant vertical load and defined velocity on the sample surface over a distance of 20 μm. The sample was modeled with a very fine mesh in the scratch area (element size of 200 nm) and an increasingly coarser mesh with increasing distance from the scratch. The microscratch model diverges from the nanoscratch model mainly in terms of the indenter and sample geometry. The sample was modeled as a 200 μm thick, 300 μm wide and 1500 μm long body, and the indenter was modeled as a sphere with a radius and height of 200 μm and 10 μm, respectively.

In preliminary work, the flow curves of the nanolaminated coating were determined for strain rates of 0.05 s^{-1}, 0.25 s^{-1} and 1.25 s^{-1} [6,7]. The substrates' material properties are presented in [6,9] and were determined for a strain rate of 0.05 s^{-1}. The maximum strain rate values in the substrate do not exceed 0.15 s^{-1} for both micro- and nanoscratches in simulations. Since the strain rate of substrate's flow curve determination and the maximum strain rate in simulation don't differ much, no strain-rate dependency of the substrate material was implemented in the model. To implement the strain-rate dependency of the coating material in Abaqus, the 0.05 s^{-1} flow curve was used as a reference and implemented in the program as a series of stress–strain value pairs. The flow curves of higher strain rates in simulation were calculated by increasing the flow stress values of 0.05 s^{-1} strain rate using a strain-rate-dependent factor. This factor was calculated for strain rates of 0.25 and 1.25 s^{-1} as the ratio of the mean stress value for the first 20% of strain between the 0.25 and 1.25 s^{-1} strain rate flow curves, respectively, and the 0.05 s^{-1} strain rate flow curve. The strain rate factor and the flow curve value pairs were linearly interpolated and constantly extrapolated. This implementation created small deviations in the modeled and measured flow curves at higher strain rates because the shape of the experimental flow curve changed with increasing strain rate. The determined flow curves and flow curve values implemented in the material model are listed in Table 1.

3. Results

3.1. Determination of nanoindenter geometry

After performing the nanoscratches, the Berkovich indenter used was examined by laser microscopy (Fig. 1). The rounding of the tip