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Identification of critical load for scratch adhesion strength of nitride-based thin films using wavelet analysis and a proposed analytical model

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ABSTRACT

The use of Acoustic Emission (AE) signal of scratch test to evaluate the thin films' adherence led to unavoidable large uncertainties due to the problematic determination of the frequency band and the improper installation of the acoustic sensor, especially for coatings thinner than 100 μm. In this paper, thin film–substrate adherence in high-speed steel is evaluated by Wavelet Analysis (WA) of the force–displacement curve in micro-scratch tests performed on the samples. The measured load displacement signals of deposited thin films were used as inputs for the wavelet module. We propose an analytical model combined with wavelet analysis, which is based on experimental data. Scratch adhesion strength could be identified by applying wavelet technique to the measured force displacement data to detect the location of the critical load during micro-scratch testing for TiSiN coating on high-speed steel. An analytical model is also proposed to predict the adhesion stress and to support the wavelet analysis technique where the stress is difficult to be assessed. Thin films were studied by optical microscopy, micro scratch testing machine and X-ray diffraction. The WA results are compared with model predictions in order to establish indication of scratch adhesion strength aimed at improving the manufacturing process.

1. Introduction

A nano-composite TiSiN coating is a combination of a hard transition metal nitride (TiN) nano-crystal and an amorphous interfacial phase of Si_3N_4 [1,2]. It has attractive mechanical properties such as high hardness, oxidation resistance and adhesion strength [3–7] that is used in cutting tool, among other things [8]. In an application where the process involves sliding contact between at least two materials, scratch test is a practical way to evaluate the material performance and mechanical behavior of the system. The scratch testing can be done by applying certain loads upon an indenter, which is moved horizontally, or vertically onto the coated surface until a coating failure occurs. Failure of thin coating under scratch is usually triggered by initiation and propagation of cracks on the coating substrate interface and consequent flaking of the coating material from the substrate [9,10].

Some computational models have been used including Finite Element Analysis (FEA), Molecular Dynamics Analysis (MDA) and Smoothed Particle Hydrodynamics (SPH) to monitor damage initiation and crack propagation during scratch testing [11–13]. For example, FEA simulation technique was utilized to study displacement and stresses during scratch but has differed 38% with the experimental data [11]. Meanwhile, MDA and SPH are also insufficient to meet requirement for the application in practical engineering problems [12,14].

Acoustic emission wavelet analysis—on the other hand seems to be a good technique for assessing the coating adherence failure at phase interfaces [15]. However, this technique does not use the measured force–displacement of the scratch test. Rather it needs an acceleration sensor to be fixed at proper distance away from the starch indenter to detect the elastic wave signal. Practically, determination of the location and frequency band as well as installation of the acoustic sensor imposes unavoidable large uncertainties of the obtained results especially for coatings lower than 100 μm. According to the current literature, it is difficult to have an analytical model which includes the effects of three types of friction i.e. sliding, adhesion and ploughing friction in one model for scratch adhesion strength.

In another word, there is no comprehensive analytical model for scratch test that interprets the nonlinear relationship between friction components, and interaction between coating thickness and surface roughness with adhesion strengths. That's why our proposed wavelet analysis uses real experimental data, which includes all effects of the

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interacting parameters in order to investigate the combined effects of the interaction between film thickness, surface roughness, indenter and friction.

In this manuscript, to avoid large errors from acoustic sensors especially for those coatings lower than 5 μm, Wavelet Analysis (WA) was done on the measured force–displacement signals. In other words, the detection of the critical load location during micro-scratch testing for TiSiN coating on high speed steel is now depending on more accurate values.

The measured load–displacement signals of deposited TiSiN coating are used as inputs for WA instead of the acoustic signal that suffers from unavoidable large errors. In addition, an analytical model will be developed to predict coating adhesion strength where it is difficult to access or get information from WA.

2. Sample preparation and characterization

2.1. Deposition process

The TiSiN thin films were coated on high speed steel (HSS-AISI (M3:2)) substrates using a multi-target magnetron sputtering physical vapor deposition (PVD) of high purity Ti and Si targets which coupled to the DC and RF power source, respectively. Both targets were fixed above the substrate holder at about 125 mm distance. The substrates, [2 × 2 × 2] cm cube, were ground and polished to a mirror finish of surface roughness, $R_a < 0.05$ μm. The substrates were ultrasonically cleaned in acetone and subsequently cleaned with distilled water and dried with nitrogen gas before placing in the PVD sputtering chamber.

The experiments were conducted by varying four parameters i.e. the RF power, DC power, N_2/Ar gas flow rate ratio and deposition time. The deposition process was initiated by evacuating the chamber to 2.67 mPa. Then argon gas was purged into the chamber to facilitate plasma formation for etching possible oxides/contaminations from the targets. The top surface of the substrates was also etched by applying negative substrate bias of 100–150 V. A Ti interlayer deposition was deposited which then followed by TiSiN thin film coating. The deposition of TiSiN was initiated by purging in N_2 gas into the chamber. The working pressure was maintained in the range of [0.93: 1.19] Pa during the coating process. The substrates were taken out after coating when the temperature in the chamber was at room temperature and then kept in a dry box for further investigation.

2.2. Material characterization

The microstructure analysis of the deposited thin film was carried out using a field emission scanning electron microscopy (FESEM) model (Zeiss Auriga). Typical microstructure and morphology images are illustrated in Fig. 1.

Micro-scratch testing of the TiSiN coating was performed using a Micro Test system (Micro Materials Ltd., Wrexham, UK). A diamond probe with a nominal radius of 25 μm was drawn across the surface of the coating at 1.20 μm/s. Pre-processing is necessary to remove the effect of roughness, topography, slope, and instrument bending on the data according to [16]. A typical scratch test and failure point Lc_2 is indentified in Fig. 2. Three stages in failure mode of typical PVD coating were identified in the scratch test. LC_2 is marked as '4' whereby the delamination is obviously seen in the micro image.

The scratch procedure involved three sequential scans over 1000 μm scan distance using a multi-pass wear test mode in the instrument's software. The three scans were (1) an initial topography scan of 2.00 mN constant load, (2) a scratch scan where the applied load was ramped after 50 μm at 3.00 mN/s to the maximum load of 1000 mN and (3) a final topography scan over the scratched track at 2.00 mN load. At least three tests were carried out on each sample.

The coating thickness was measured using the Micro Test System as a surface profile meter scanning with a minimum load of 2.00 mN (sufficiently low that no wear occurs at this load) across boundaries of the coated and uncoated regions. The uncoated region was exposed when the tape-covering portion of top surface of substrate was removed. The difference in the step height between these regions provides the coating thickness. An average value from at least three measurements was taken on each sample.

The crystal structure of the deposited TiSiN coating was characterized by an X-ray diffraction pattern (XRD) model (Siemen D500) with Cu Kα (1.54 A) radiation in a [θ:2θ] scan mode. The XRD with initial angle of $2\theta = 10–100°$, a step size of $\Delta\theta = 0.10°$, and step time of Δt 3 s. The XRD pattern is plotted in Fig. 3. The macrostrain was determined using XRD line broadening analysis by employing an approximation method [17] followed by Rietveld analysis [18,19] using HighScore Plus developed by PanAnalytical company.

The strain and size analysis was performed using line profile analysis. Williamson-Hall plots were prepared to quantify the broadening. Then $B^*\cos\theta$ was plotted against $\sin\theta$ for (111), and (022) corresponding to $2\theta = 36.8$, and 61.96°, respectively. The information on how to calculate the macrostrain are extensively discussed and explained in

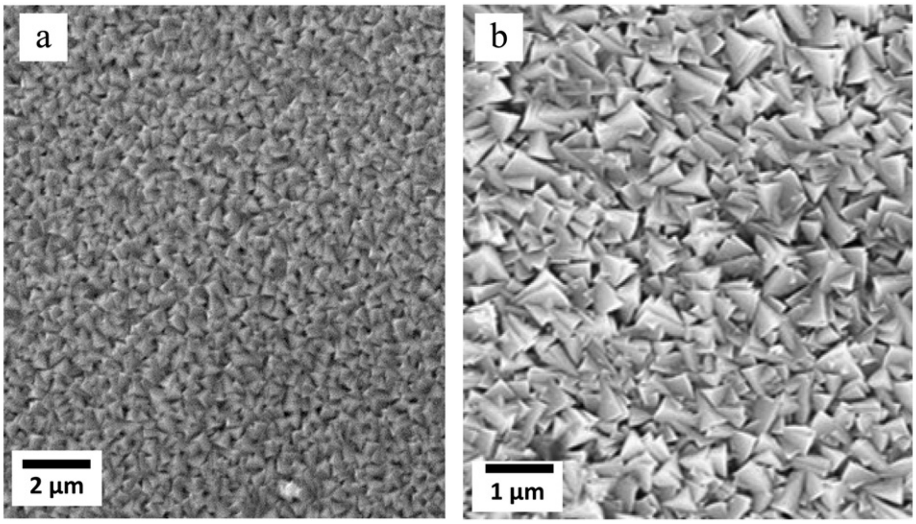

Fig. 1. FESEM micrographs of a typical sample.