Topographical features in micro and nano scale scratch tracks on CNx coatings

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INTRODUCTION

Carbon nitride (CNx) coating has the potential to be the toughest of all composite coatings due to the β-C_3N_4 phase[1]. The CNx phase has been shown to improve fracture and wear resistance in coatings[2]. The micro and nano mechanical properties of CNx coating are critical in the context of its numerous uses, such as protective wear resistant coatings on tools and write heads of storage devices, bio implants, and so on. CNx coatings have mostly been deposited using various PVD and CVD processes and static Nanoindentation has been performed on the CNx coatings previously[3]. Sliding nanoindentation tests on the other hand is an effect means of studying properties related to adhesion, wear and abrasion and are employed on thin coatings to test its usability for a specific purpose [4-9]. It is practically difficult to quantify coating adhesion from the load-depth plot and requires visual inspection of the scratch region as well. Minute features related to film failure can be present inside the scratch as well as adjacent regions which requires proper inspection and analysis. CNx coatings deposited on Si (100) underwent scratch tests in both micro and nanoscale the results of which were analyzed thoroughly showing some novel results.

MATERIALS & METHODS

The plasma enhanced chemical vapour deposition (PECVD) using N_2/C_2H_2 precursor gases was used in depositing CNx films on Si(100) substrates at 400°C substrate temperature for 2 hrs. The deposition chamber was initially pumped down to 10^{-5} mbar by turbo molecular pump prior to inletting the precursor gases. A flow rate of 5 sccm was maintained for C_2H_2 prior to the inflow of nitrogen gas. The sliding nanoindentation (MTS, USA) tests were performed using a Berkovich indenter and the optical microscopic images of the scratch track was captured. Micro-scratch tests were done using a Rockwell C indenter to see the wear and abrasive response.

RESULTS & DISCUSSIONS

Fig 1a shows the scratch track on CNx coatings having a width of **45µm**. performed by a Rockwell C micro indenter. Wear can be seen to have taken place confined to an intra scratch track of lower width (**15 µm**). Hence two different zone of the spherical indenter having different tip diameter are in action with the smaller one responsible for wear as shown in the inset. Formation of radial cracks originating from the larger zone can be seen. The radial cracks were not linear and had curvature associated with them. These cracks emerged due to the release of accumulated plastic strain due to the advancing crack. Cracks also emerged from the inner track at higher loads. Image analysis of the different regions is shown separately (**Fig 1 b, c, d**) Ploughing, interface sliding, and pulling the coating occur as the indenter advances towards the scratch under increasing load. Four loading effects—friction force, geometrical deformations, bulk plasticity concentration, and residual stresses—combine to produce the stress field in the coated surface. **(Fig 1e)**[10].

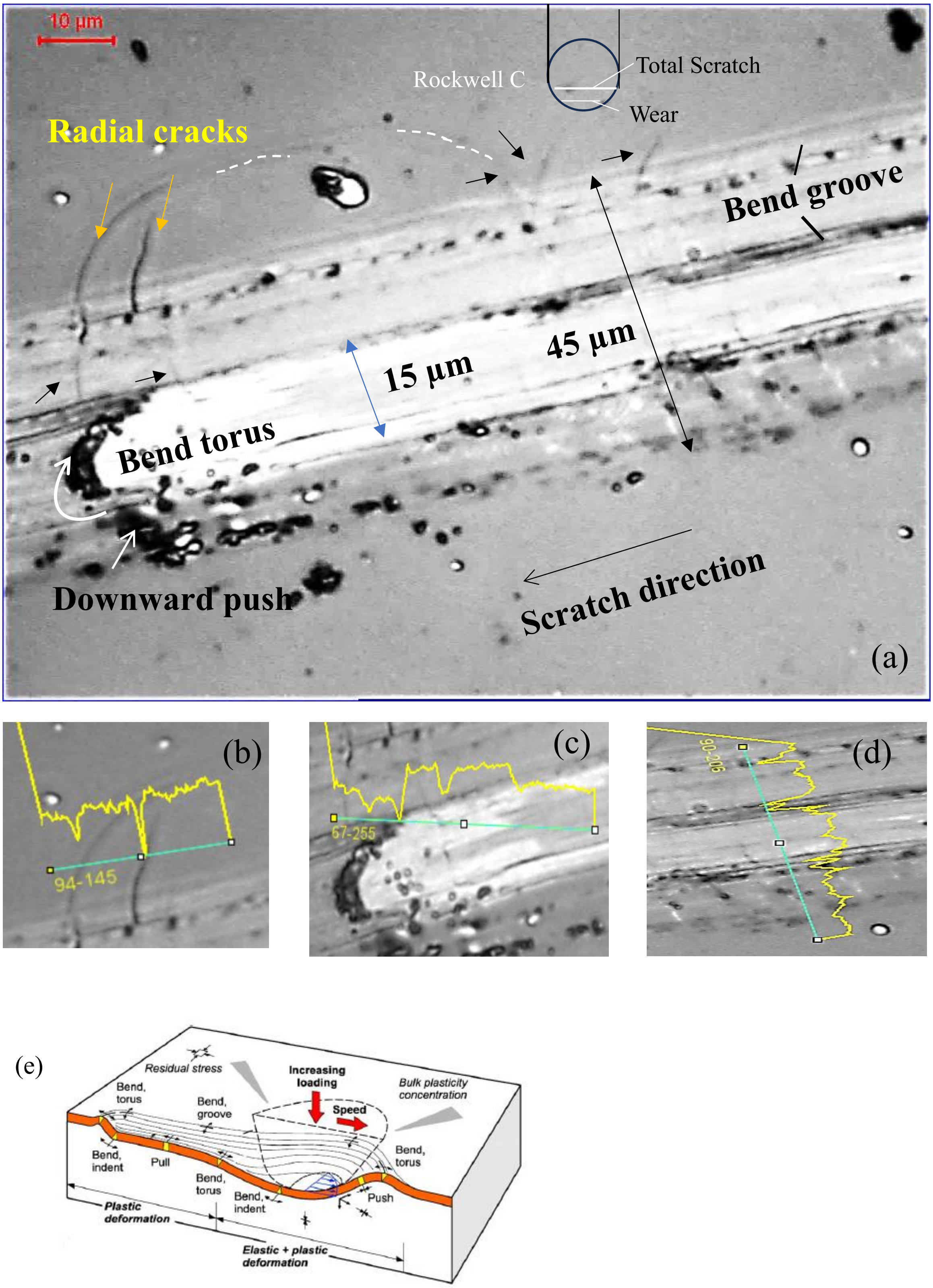

Fig 2: Wear track from sliding indentation on CNx coatings on Si (100) substrates and linear profiles of the different failure features (e) the stress field in the coated surface resulting from four loading effects: friction force, geometrical deformations, bulk plasticity concentration, and residual stresses (**reproduced with permission** [10])

The adjacent radial cracks were not same in depth with the one nucleating closer to the internal track had less intensity. The reason behind is the accumulated strains are formed in the bend grooves due to interaction with the bend torus. The bend torus is more intense as one moves further away from the direction of scratch and is also the reason for the curved nature of the radial cracks. The bend torus also caused a downward push of the coating into the substrate. The coating removal (wear) was similar in the inner and outer cracks for lower loads, but quite distinctive as the loads increased.

Nano scratch involved pre-scan, scratch and residual profile as shown in **Fig 2**a. The sliding indents were done with a Berkovich indenter, with a speed of **50 µm/s** to a maximum load of **100 mN** on a **600 μm** scratch track. The change in the nature of the displacement curves were tallied with the scratch track image (**Fig 2b**) and were found to consists of three loads at which the films failed cohesively (Lc^1), adhesive failure (Lc^2) and radial crack formations (Lc^3) obtained as **55, 75 and 85 mN** respectively.

The magnified images for different regions are shown in **Fig 2 (c, d, e)** which showed a wedge spallation followed by cohesive failure and formation of bending grooves and lastly adhesive failure. The plasticity percentage in the failure was much than the previous case due to the use of sharper and smaller 3-sided pyramidal Berkovich indenter as compared to Vickers indenter. The cross-sectional profile of the scratch is given in **Fig 2g** showing a penetration of **25 nm** for a maximum load of 100 mN applied. The centralized pile up was about **5 nm.** Absence of any major brittle failure indicated the films to have high fracture resistance.

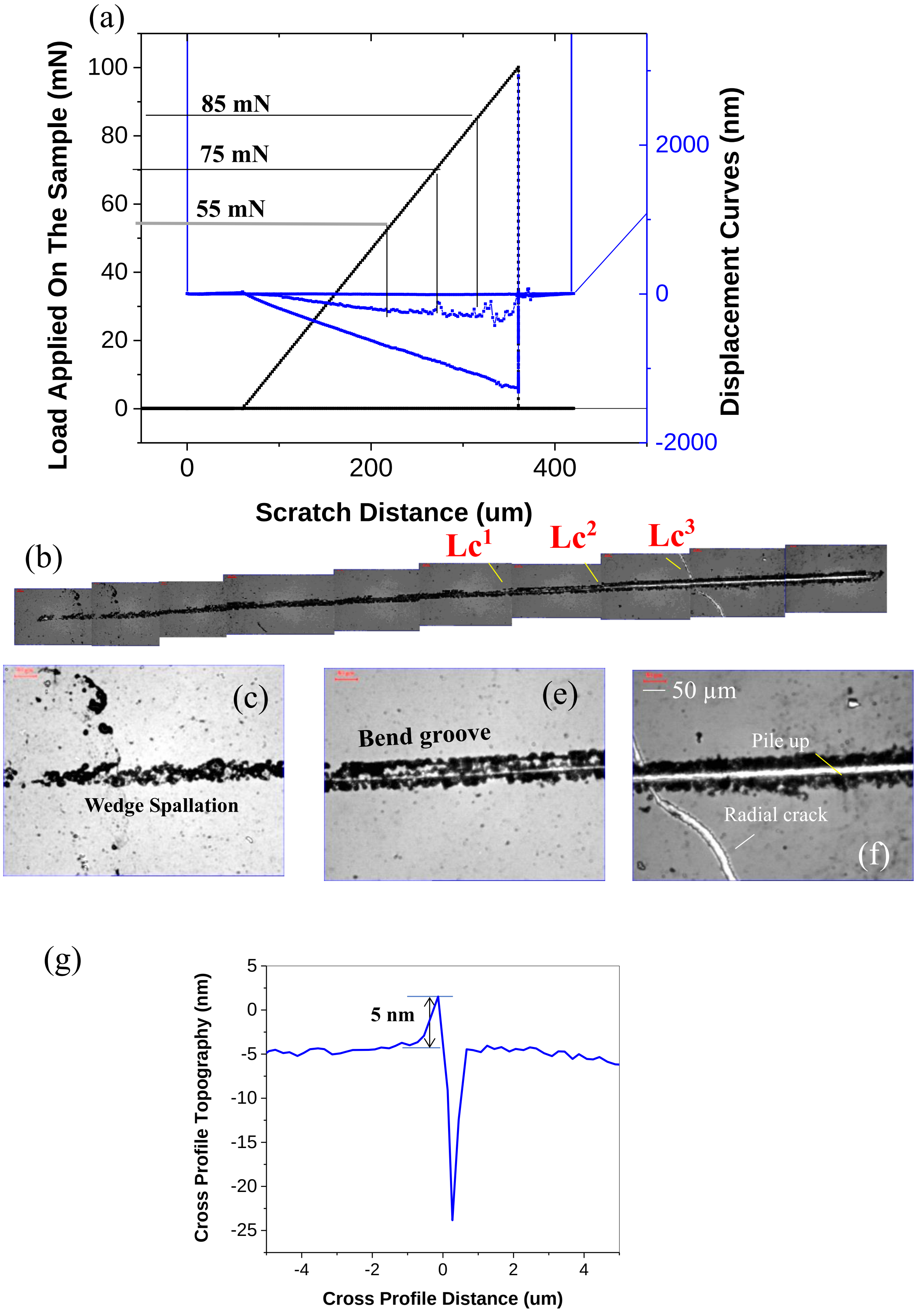

Fig 2: Nano scratch test performed on CNx coatings deposited on Si substrates with (a) the 3-stage profiles (b) optical image of the scratch track with (c, d, e) selected magnified area and cross-sectional profile.

CONCLUSIONS

CNx coatings deposited on Si substrates showed good adhesion along with wear and fracture resistance. No major brittle failure was observed in the micr0 and nano scale tests performed. However, a higher proportion of plastic deformation was found due to higher coating sharpness in case of Berkovich indenter.

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DECLARATIONS

Compliance with Ethical Standards

The manuscript has not been submitted in parallel either in full or partially to any other journal.

Conflict of interest

There is no conflict of interest among the authors

Research Data Policy and Data Availability Statements

Data shall be provided on request

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