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A simple method for residual stress measurements in thin films by means of focused ion beam milling and digital image correlation

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ABSTRACT

Hydrogenated amorphous carbon (a-C:H) coatings contain substantial residual stresses which particularly influence the failure behaviour of the coating system. However, measuring their residual stresses with conventional methods is not trivial. In this work, a simple method is presented to determine residual stresses of thin films locally by using stress relaxation tests by means of focused ion beam (FIB) milling and digital image correlation (DIC). It is shown, that the H-bar geometry, as it is used for TEM lamella preparation, is especially suitable for such measurements. Displacements due to relaxation of residual stresses are tracked using DIC and the stresses are obtained by correlation with finite element analysis. The method is applied to determine residual stresses of two a-C:H coatings processed with different deposition processes and parameters. It was found, that the coatings differ in their mechanical properties as well as in their residual stress state. The proposed method offers therefore a simple way for analysing residual stresses in thin amorphous coatings.

1. Introduction

Diamond-like carbon coatings show high residual compressive stresses in the magnitude of a few hundred MPa up to some GPa [1] which influence the performance of the coating and their adherence to the substrate. Thus, knowing the residual stress state quantitatively is a crucial point for understanding the damage behaviour of such coatings. Common X-ray methods for determining residual stresses are hardly applicable, since these coatings are amorphous and usually only a few microns in thickness.

A method based on focused ion beam (FIB) milling and digital image correlation (DIC) presented in the literature [2] enables the local measurement of residual stresses of thin films regardless of whether they are amorphous or crystalline. Thereby, material is removed by FIB milling to form a specific geometry, where the material in the vicinity relaxes its internal stresses. The displacement can be measured with DIC and allows reconstruction of the residual stresses by comparing the relaxation displacements with finite element analysis (FEA). In the literature groups experimented with different milling geometries like circular holes and rectangular trenches [3–5] as well as a pillar geometry [6]. These specific geometries represent common relaxation states, which are exemplarily shown in Fig. 1a) and b) for a coating under residual compressive stresses together with the corresponding relaxation deformation. However, these geometries are disadvantageous in certain aspects of the FIB milling or the stress analysis. Therefore, a new geometry of the shape of an H-bar is proposed for stress measurement, to overcome these disadvantages (see Fig. 1c).

Methods such as the rectangular trench or the circular hole, which use a half infinite continuum for stress determination, relax only at the milled areas and this results in a complex, non-linear displacement and strain gradient (see Fig. 2). In order to correct for rigid body movement either the displacement at a certain position has to be zero or the displacement field needs to be symmetrical. For these geometries, it is difficult to find a reference position on the sample which is not relaxing. Further, the symmetry of the cut can only be used at low magnification where two milled edges are visible in the micrograph, thus hindering a high spatial resolution. For most of these geometries no simple or analytical solution exists to calculate the residual stresses and a FEA is necessary. On the other hand with the pillar milling a very small volume is tested which relaxes homogeneously and thus results in a symmetric, linear displacement gradient and constant strain field, from which the rigid body movement can easily be removed by using the symmetry of the relaxation field (cf. Fig. 3). Furthermore, a simple equation to calculate the stresses is available [6]. A circular shape allows evaluation of all directions in two dimensions and so the principal stresses and axes in a plain stress state can be determined. Next to the small displacements a further drawback of this geometry is its relative complex shape for the FIB milling. In order to obtain a precise circular pillar without rounded edges at the top a high quality FIB technique is required, which is especially a problem for microscopes with raster-based milling engines. Further, the shape of the milling geometry influences the stress analysis. Rectangular or line shaped geometries are sensitive to specific directions and so only stresses in these directions can be analysed.

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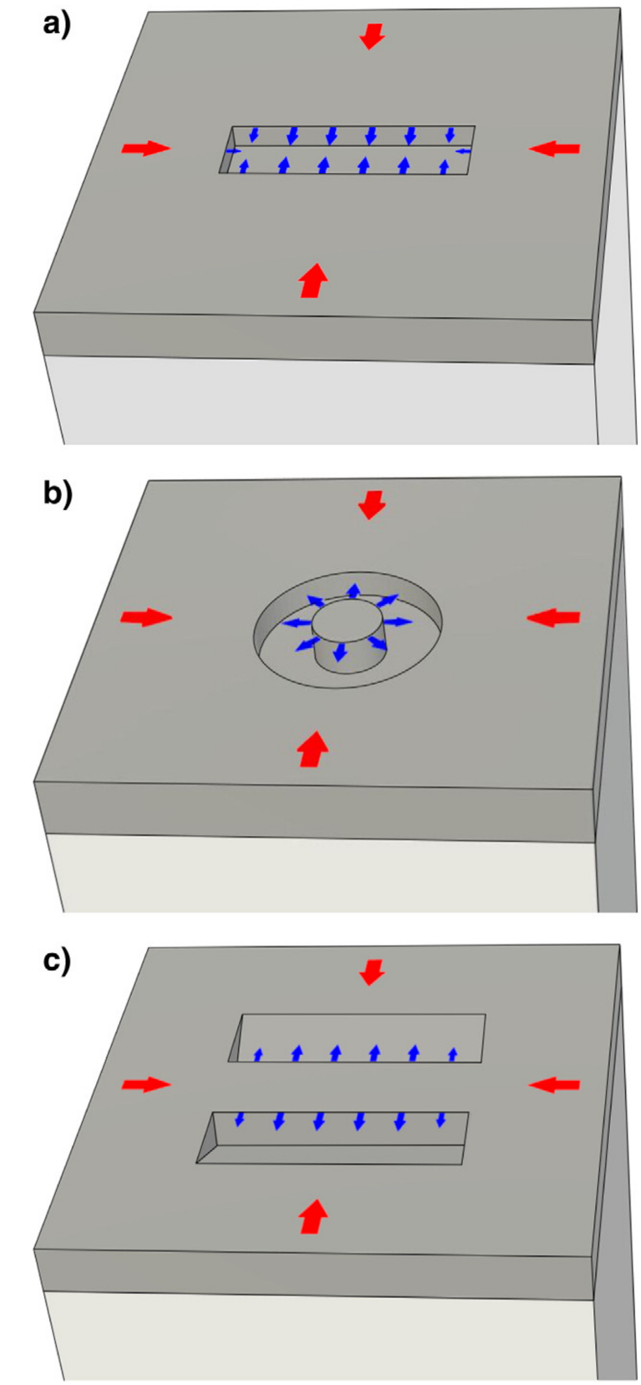

Fig. 1. Schematically shown are different relaxation geometries and their deformation (blue) due to relief of compressive stresses (red): a) slot milling after Kang et al. [2] b) pillar milling after Korsunsky et al. [6] and c) proposed H-bar geometry.

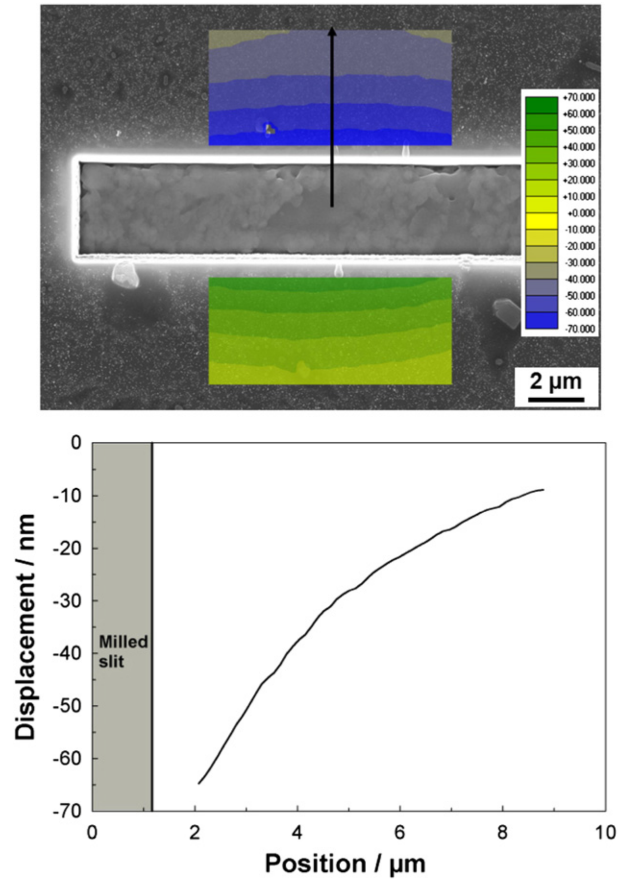

Fig. 2. Displacement gradient (in nm) of slot milling of an a-C:H coating system on steel and corresponding displacement plot along the black arrow.

Slot and pillar milling were exemplarily applied to a hydrogenated amorphous carbon (a-C:H) coating system. A complex displacement gradient was found at the edge for the slot geometry (cf. Fig. 2). The pillar geometry results in a linear homogeneous expansion in all directions. Fig. 3 shows the symmetric displacement gradient in one direction of the pillar. In comparison higher displacements are found for the same coating system at the milling edge of the slot geometry, enabling therefore a more accurate displacement measurement at low residual stress levels. It should be noted however, that the maximum displacements for the pillar geometry depend also on the pillar diameter. Larger pillar diameters would result in a larger maximum displacement.

In this work a similar approach with a different milling geometry is proposed. Material is removed by FIB milling to form an H-bar geometry, as it is used for TEM lamella preparation (see Fig. 1c). This geometry combines some of the before mentioned advantages of slot and pillar geometry. DIC is employed to measure the expansion or contraction of the bar due to stress relief. The displacement is correlated with a finite element analysis in order to reconstruct the residual stresses. The implementation of this method is demonstrated on two hydrogenated amorphous carbon coating systems showing different mechanical properties.

2. Experimental details

2.1. Hydrogenated amorphous carbon coating

2.1.1. System 1

Polished steel plates (AISI D2) were coated with about 1.8 µm a-C:H in a Balzers BAI 830 PVD/PECVD (physical vapour deposition/plasma enhanced chemical vapour deposition) equipment. A well adherent coating was achieved by firstly depositing a sputtered columnar Cr film as an adhesion layer followed by an adjacent ramp layer showing a chemical gradient to the carbon rich a-C:H coating. The ramp layer is deposited in a combined PVD/PECVD process, with processing temperatures in the range of 200 °C. The overall thickness of the adhesion and ramp layer is between 500-700 nm. Further details on the deposition process and the microstructure of the investigated coating system can be found elsewhere [7].