**In Situ** Nanoscale In-Plane Deformation Studies of Ultrathin Polymeric Films During Tensile Deformation Using Atomic Force Microscopy and Digital Image Correlation Techniques

Xiaodong Li, Weijie Xu, Michael A. Sutton, and Michael Mello

**Abstract**—The local, nanoscale deformation behavior of ultrathin polyethylene terephthalate (PET) and polyethylene naphthalate (PEN) films used as substrates in magnetic tapes was studied by atomic force microscopy (AFM) and digital image correlation (DIC) techniques. A custom-designed tensile stage was integrated with the AFM to perform uniaxial tension tests on the polymeric films in situ where the film surfaces were imaged simultaneously by AFM. The surface features on the PET and PEN films were used as reference patterns for the DIC processing. To improve the accuracy of the AFM imaging system for the application of the DIC method, a simple, cost-effective experimental procedure was established. Axial and transverse strain fields and Poisson’s ratio maps with a spatial resolution of 78.13 nm were constructed via processing the AFM images of unstretched and stretched samples with the DIC software. Results from the AFM studies indicate that the deformation in both PET and PEN is nonuniform at the nanoscale. The nanoscale deformation mechanisms are discussed in conjunction with the structure of the PET and PEN films.

**Index Terms**—Atomic force microscopy (AFM), deformation, digital image correlation (DIC), magnetic tapes, polymeric films.

I. INTRODUCTION

R**ECENT** developments in science and engineering have advanced our capability to fabricate and control materials/structures on the scale of micro/nanometers, and have brought problems of material behavior on the micro/nanometer scale into the domain of science and engineering [1]–[5]. A precise characterization of the mechanical properties of micro/nanosstructures is required to use them as structural and/or functional elements in devices. In particular, full-field data regarding the local deformation response of complex material systems such as polymeric nanocomposite materials is greatly needed to understand how changes in local structure relate to the nanoscale response, damage evolution and resulting flaw progression. For example, the design, manufacturing, and reliability testing of high-density magnetic tapes require diagnostic and characterization methods that can be scaled to detect and locate nanoscale deformation variations. It is believed that local deformation in magnetic tapes is one of the major root causes for magnetic signal loss and distortion [6].

The extremely small structural features of polymeric nanocomposite materials impose a tremendous challenge to many existing testing and measurement techniques for quantitative measurement of deformation at the nanoscale. Polyethylene terephthalate (PET) and polyethylene naphthalate (PEN) with nanofillers as reinforcements are currently the most widely used polymeric substrate materials for magnetic tapes [8], [9].

Thinner substrates and higher areal densities (track density × linear density) are required to meet the demand for advanced magnetic storage devices with high volumetric densities, especially for computer data storage tapes. Since the mechanical properties of a magnetic tape substrate play a critical role in determining the performance and failure behavior of the tape [6]–[10], and thereby control the track spacing variation [10], it is important to understand the dimensional stability of the tape substrate, which accounts for 90% of the total tape thickness.

To enhance their mechanical properties, nanofillers are widely used as reinforcements in both PET and PEN matrices [8]. For high areal and track densities, deformation due to mechanical, thermal, and hygroscopic effects must be minimal during storage on a reel and use in a drive [8], [9]. For instance, when a magnetic tape slides against its magnetic head, nanoscale asperities such as debris particles at the tape–head interface may cause local deformation, thereby leading to magnetic signal distortion and loss. If the substrate of a magnetic tape deforms excessively, then the head cannot read or misreads information stored on the tape [9]. Various long-term reliability problems including uneven tape-stack profiles (or hard bands), mechanical print-through, instantaneous speed variations, and tape stagger problems result from local deformation and damage in the substrate. Although the mechanical and tribological properties of magnetic tapes and their substrates such as elasticity modulus, strength, creep resistance, and scratch and wear resistance have been well studied, the local deformation behavior is still, to a large extent, unknown. This limits further development of magnetic tapes.

The digital image correlation (DIC) technique developed in the early 1980s has been widely used to measure local deformation of solid surfaces such as local displacement and local
strain [11]. Displacement resolution of the DIC method relies heavily on the metrology tools that are used to determine surface topology, as these features are used to quantify the surface displacement field. Thus, the repeatability of the image-capturing instrument will have a significant effect on the accuracy of the resulting displacement field measurements. Traditionally, the DIC method utilizes an optical imaging system to obtain a characteristic pattern for use in estimating surface displacements. In such cases, the method is diffraction limited to spatial resolutions greater than 1 μm. To extend application of traditional DIC approaches to nano paradigm, a nanoscale surface measurement tool is needed. Atomic force microscopy (AFM) and scanning tunneling electron microscopy (STM) have been widely used to measure the nanometer-scale topography of solid surfaces [4], [5], [12]–[14]. The AFM technique offers digital data storage, ability to postprocess data to obtain a range of spatial resolutions, and use of incoherent illumination sources, providing a base for integration with currently available DIC operation platforms to map the local deformation variation at very small scales [15]–[17].

In this paper, we report, for the first time to our knowledge, quantitative in situ nanoscale deformation measurements for magnetic tape substrates subjected to uniaxial tensile loading, imaged by AFM and analyzed using the DIC technique. A simple, cost-effective experimental procedure was established to improve the accuracy of the AFM imaging system for the application of the DIC method to experimental nanomechanics. Following this procedure, the in situ tensile testing was integrated with AFM imaging so that DIC could be performed to map the axial and transverse deformation as well as the Poisson’s ratio for both PET and PEN films at the nanoscale. Experiments were carried out in situ where the polymeric films were stretched and the surfaces were imaged by AFM. The surface morphology change and deformation mechanisms during tensile loading are discussed in conjunction with the structure of the PET and PEN films.

II. EXPERIMENTAL

A. Test Samples

The PET and PEN films used in this study are 6.4 and 4.7 μm in thickness, respectively. They are used for advanced magnetic tapes, especially computer data storage tapes. Fig. 1 shows the chemical formulas of PET and PEN films. Both films were manufactured by a biaxial drawing process and have identical hydrocarbon backbones indicative of polyester materials. PET contains a single benzene ring in each repeating unit, whereas PEN contains a naphthalene ring that is slightly more rigid [8], [9]. However, naphthalene groups have high mobility from ambient temperature to 60°C. Typical crystallinities of the PET and PEN films are 40–50% and 30–40%, respectively. Strips of samples that are 4 mm wide were cut from a 300 mm × 300 mm sheet along the longitudinal direction (LD). The strips are then glued onto the grips of the tensile stage for tensile testing and AFM imaging.

B. In Situ Tensile Testing and AFM Imaging

A custom-built tensile stage (NanoSolutions Technology Inc., West Columbia, SC) was used to perform in situ tensile tests on the PET and PEN films under an AFM (D3100; Digital Instruments, Santa Barbara, CA). A stepper motor driven linear stage was used to apply uniaxial loads on the polymeric film samples along the LD. The tensile stage has a load resolution of 10 mN and a displacement resolution of 1.6 μm. All tensile tests were conducted at a constant strain rate of 4 × 10⁻⁵/s. AFM tapping mode with a 2–5 nm radius silicon tip was used to scan the film surface to achieve higher lateral spatial resolution and less topographical distortions due to tip–sample interactions. Images of the same control area, which was chosen at the center of the sample, were taken before and after stretching the sample with aid of the attached optical microscope.

C. DIC Processing

The AFM images obtained before and after stretching the polymeric film sample were processed using a Vic-2D 4.4 DIC package (Correlated Solutions, Inc., Columbia, SC). Two-dimensional surface displacement and strain distribution field maps were constructed by comparing the AFM images of the same area before and after deformation.

For more detailed information about DIC, please see [11], [12], and [15]–[17].

III. RESULTS AND DISCUSSIONS

A. Stress–Strain Relationship

Both PET and PEN were stretched uniformly during the test, exhibiting both elastic and plastic deformation regimes. No necking was observed before failure. Fig. 2 shows the stress versus global strain curves for the PET and PEN films obtained by using the measured far-field loading, grip displacements and initial cross-sectional area of the films. The elastic modulus for
each sample was calculated from the slope of the initial linear portion of its stress–strain curve. The elastic moduli of the PET and PEN films obtained from our experiments are 2.3 GPa and 3.3 GPa, respectively. The PEN exhibits higher elastic modulus and yield stress than the PET, while PET shows much higher failure strain than PEN (65% for PET and 25% for PEN). The measured mechanical properties for PET and PEN are in good agreement with previously reported values [8], [9].

B. Calibration of the Accuracy of the AFM Imaging System Using DIC

To determine if the AFM is a suitable imaging tool for the application of the DIC technique to experimental nanomechanics, the accuracy of the AFM imaging system used in this study must be calibrated. A typical AFM image takes several to tens of minutes to scan. During the scanning period, many factors affect AFM accuracy: AFM scanner drift; nonlinearity due to time and temperature dependent behavior of the piezo materials; relative movements between the scanner and the sample; and the sample and the in situ tensile testing stage environment change inside the AFM system affecting viscoelastic behavior of the test materials [18]. Among these factors, nonlinearity and hysteresis of the AFM system contribute the most to the “artificial” image motions. Because of difference in the material
properties and dimensions of each piezoelectric ceramics element, the AFM scanner exhibits more movement per volt at the end of a scan line than at the beginning, i.e., the relationship of movement versus applied voltage is nonlinear which causes the forward and reverse scan directions to behave differently. The nonlinearity in \( x \) and \( y \) directions may induce image distortions that increase “artificial” strains. The nonlinearity in the \( z \) direction may lead to inaccurate height measurements. This will greatly decrease the accuracy of the DIC method since the relative contrast of the height images will be used for the correlation between different images. This nonlinear relationship can be partially corrected during the calibration routine by applying a nonlinear voltage in real-time to produce a linear scan in \( x \), \( y \), and \( z \) in both trace and retrace scan directions for the AFM system used in this study. The self-heating of the piezoelectric ceramics in turn causes the hysteresis effect.

In this study, the AFM system linearity in \( x \), \( y \), and \( z \) directions was calibrated strictly according to the manufacturer’s manual. The baseline linearity accuracy of the AFM system was calibrated by continuously taking AFM images under the same AFM control parameters on an unstrained sample using the “auto scan” function of the AFM control software. The obtained images were correlated with the immediately previous images using DIC software. Fig. 3 shows representative AFM height images during continuous scanning and imaging under the same AFM control parameters on unstrained PET and PEN, respectively. The artificial displacement, which was obtained by marking the same particle with an arrow in a series of 256 pixel \( \times \) 256 pixel AFM images of the same scan area of 20 \( \mu \text{m} \times 20 \mu \text{m} \), is 20 pixels (1.6 \( \mu \text{m} \)) in \( x \) direction and 20 pixels (1.6 \( \mu \text{m} \)) in \( y \) direction for the PET film and 1 pixel (0.08 \( \mu \text{m} \)) in \( x \) direction and 3 pixels (0.24 \( \mu \text{m} \)) in \( y \) direction for the PEN film, respectively. Fig. 4 shows the average artificial axial and transverse strains from the comparison between the consecutive images by DIC. For the PET film the average artificial axial and transverse strains at the beginning of the 20 \( \mu \text{m} \times 20 \mu \text{m} \) AFM imaging tests are \(-0.05\%\) and \(0.4\%\), respectively. For the PEN film the average artificial axial and transverse strains at the beginning of the 20 \( \mu \text{m} \times 20 \mu \text{m} \) AFM imaging tests are \(0.05\%\) and \(0.6\%\), respectively. Such artificial axial and transverse strains result from the nonlinearity and hysteresis effects discussed in the previous paragraph. After

Fig. 6. AFM images and the corresponding axial strain \( (\varepsilon_{xx}) \) and transverse strain \( (\varepsilon_{yy}) \) fields obtained from the DIC for the PET film at 0%, 2%, 4%, and 6% applied global strains.
Fig. 7. AFM images and the corresponding axial strain ($e_{xx}$) and transverse strain ($e_{yy}$) fields obtained from the DIC for the PEN film at 0%, 2%, 4%, and 6% applied global strains.

During the in situ AFM tensile testing, images obtained from the same area are needed. In general, this can be achieved using zoom and offset functions in the AFM control software. It is necessary to test the accuracy of the AFM imaging system under the conditions of changing the AFM control software parameters. These experiments were carried out as follows: 1) take images of an unstrained sample; 2) manually move the sample using the stage controller; and 3) take AFM images and locate the same area by adjusting the control software offset parameters. After processing these images using the DIC software, it was found that even a small offset value such as 0.5 μm in $x$ or $y$ direction would cause a strain error of 1% or even higher. Such errors were unstable and did not follow any patterns. Jin et al. [20] obtained similar results. So it is obvious that we cannot rely on the software control parameters to obtain AFM images of the same area. This problem can be solved through circumventing the “upper part” i.e., the AFM control software parameters by moving the “lower part” i.e., the sample stage manually. With aid of the attached optical microscope and the stage controller, we were able to locate the same area with a deviation of ±5 pixels in both $x$ and $y$ directions for a 256 pixel × 256 pixel image of the scan area of 20 μm × 20 μm. With this order of image overlap, the artificial strain errors in both axial and transverse directions were within ±0.5% range, which was still acceptable for applied strains larger than 2%. Such errors could be further minimized by using an advanced stage positioning system with nanometer resolution while the resolution of the current positioning system was only 1.6 μm. Another concern is that the particles, which were used as a reference pattern on the sample surface, could be deform differently when strained. For a 256 pixel × 256 pixel image of 20 μm × 20 μm scan area, however, difference in deformation among the particles inside
this area was much negligible. Thus, it is possible to achieve the baseline accuracy provided that we could obtain exactly overlapped images before and after stretching the test sample.

The artificial strains were the average values over the DIC calculation area of 14 μm × 14 μm. The DIC calculation can obtain the full-field displacement and strain distribution over the same area. After extensive experiments, we found that there existed a polynomial fitting relationship between the average artificial strains and the relative level of the overlapping of the compared images, i.e., the relative x and y direction displacements for the same particle in the respective images. Due to limited space, the detailed results are not presented in this paper. Thus, it is possible to subtract the error values from the calculated DIC average strains according to the overlapping level of the respective images even though the expected strain errors of ±0.5% would be within acceptable range. However, for the displacement and strain distributions, simple or even point to point subtraction would not eliminate the errors. As mentioned previously, the errors were relatively small compared to the applied strains larger than 2%. In this study, such errors were not subtracted.

For comparison, the respective AFM phase images of the test samples were also used for the DIC processing. It was found that these images produced almost the same or a little better results than the height images. This was not surprising considering that the phase images had more contrast (which would facilitate the image correlation processes) than the height images for some materials without distinctive surface features. However, for the AFM system used in this study, the phase images were not always as stable as the height images. Therefore, the height images were used in the study.

The general resolution of the DIC technique for optical systems is stated as being ±0.01 pixel. For a 256 pixel × 256 pixel image, the resolution in the form of strain is ±0.04%, which is much smaller than the strain errors caused by the AFM imaging system.

C. Mapping Axial and Transverse Strains and Poisson’s Ratio Using DIC

As noted previously, the accuracy of deformation measurement of the AFM/DIC technique depends on both the AFM...
scanning resolution and the DIC processing sensitivity [17]. In this study, a scan area of 20 μm × 20 μm was imaged by AFM using 256 pixel × 256 pixel sampling points for both PET and PEN films. Therefore, the spatial resolution is 78.13 nm/pixel. Interpolation algorithms were used in the DIC processing to improve the accuracy of the matching for each subregion [21], [22].

For comparison to the globally applied strains, the average axial strains obtained by DIC over a 14 μm × 14 μm subregion centered in the scan area are shown in Fig. 5. The average axial strain values obtained from the 14 μm × 14 μm scan size are in good agreement with the globally applied strains for both PET and PEN material systems.

Figs. 6 and 7 show the AFM images and the corresponding full-field axial strain (ε_{xx}) and transverse strain (ε_{xy}) fields obtained from the DIC for the PET and PEN films. It can be seen from the AFM images in Figs. 6 and 7 that the PET and PEN films exhibit clearly visible surface features which provide a nanoscale pattern that can be used as natural markers for the DIC processing. The axial and transverse strain fields obtained by using DIC to compare stretched and unstretched AFM images of the film samples clearly indicate that the deformations in both PET and PEN are nonuniform at the nanoscale. This is not surprising for polymeric nanocomposite materials that have viscoelastic characteristic and various structural components (polymer matrix and reinforcements) with different load carrying capabilities. It is also noted that the measured strain fields changed across the scan area in a nonrepetitive, wave-like manner as the sample was stretched. These variations in measured strain are believed to be associated with the localized response of different nanoscale structural features in the samples, resulting in highly localized strain variations of up to 150% of the average strain measured in the PET and PEN films. The high local strain variation is believed to be one of the major obstacles on the road to achieve a volumetric density of 1 terabyte per cubic inch in a linear magnetic storage device.

For illustrative purposes, Fig. 8 shows 3-D plots of the axial and transverse strain fields and Poisson’s ratio maps for the PET and PEN films. The 3-D plots clearly show that the axial and transverse strains change with increasing applied global strains. The PET film exhibits a smooth development process and small variations in strain, whereas the PEN film shows a large strain variation and localization of strain near the center of the scan area when the strain extended into the plastic regime.

Poisson’s ratio ν is defined as the negative ratio of transverse strain over axial strain during uniaxial loading in the elastic de-
formation regime [8]. The PET and PEN films used in this study are soft and thin, therefore conventional displacement measurement techniques such as strain gauges cannot be applied. Experimental determination of Poisson’s ratio of a polymeric film remains a great challenge. Using DIC, the Poisson’s ratio map can be constructed via the negative transverse strain divided by the axial strain, as shown in Fig. 8. It should be noted that the calculation of Poisson’s ratio does not require the measurement of applied global load and displacement since the local ratio of measured strains at each load level suffices. As shown in Fig. 8 for nominal applied strain of 2%, the average Poisson’s ratio values for PET and PEN are 0.04 and 0.8, respectively. These values do not match those measured using the laser scan micrometer [9], which indicates that the extracted information from the AFM images in our study is extremely localized so that the Poisson’s ratio could be different from the macroscopic scale value.

D. Nanoscale Deformation Mechanisms

Fig. 9 shows the AFM images of the PET film under the applied strains of 2.5%, 2.5%, and 3.0%. Three characteristic points, labeled as points A, B, and C in the AFM images in Fig. 9, were chosen to investigate the deformation mechanism. Taking point A as a reference point, one can see that points B and C were stretched away from point A, and rotated around point A. The rotation mechanism has been found in many nanostructured materials such as nanostructured Ni [20], and is believed to be one of the major deformation mechanisms in nanomaterials that help deformation occur and reduce stress concentration. The material rotation in the PET and PEN films might result from their viscoelastic characteristic that affects local deformation.

IV. CONCLUSION

A successful integration of in situ tensile testing, AFM imaging, and DIC processing has been achieved to map the strain fields and Poisson’s ratio variation of the PET and PEN films at the nanoscale. A simple and cost-effective procedure has been established to improve the accuracy of the AFM imaging system for the application of the DIC techniques. The measurement resolution can be further improved by increasing AFM sampling points and by reducing scan size. The combined AFM/DIC method used in this study can be satisfactorily utilized for characterizing the nanoscale deformation of polymeric films and should find more use in practical applications.

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