NANODAC – A METHOD FOR FRACTURE MECHANICAL CHARACTERIZATION ON THE NANOSCALE

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ABSTRACT

With the development of micro- and nanotechnological products such as sensors, MEMS/NEMS and their broad application in a variety of market segments new reliability issues will arise. The increasing interface-to-volume ratio in highly integrated systems and nanoparticle filled materials and unsolved questions of size effect of nanomaterials are challenges for experimental reliability evaluation. To fulfill this needs the authors developed the nanoDAC method (nano Deformation Analysis by Correlation), which allows the determination and evaluation of 2D displacement fields based on scanning probe microscopy (SPM) data. In-situ SPM scans of the analyzed object are carried out at different thermo-mechanical load states. The obtained topography-, phase- or error-images are compared utilizing grayscale cross correlation algorithms. This allows the tracking of local image patterns of the analyzed surface structure. The measurement results of the nanoDAC method are full-field displacement and strain fields. Due to the application of SPM equipment deformations in the micro-, nanometer range can be easily detected. The method can be performed on bulk materials, thin films and on devices i.e microelectronic components, sensors or MEMS/NEMS. Furthermore, the characterization and evaluation of micro- and nanocracks or defects in bulk materials, thin layers and at material interfaces can be carried out.

1 INTRODUCTION

Scanning probe microscopy (SPM) has become a powerful imaging tool for submicron surface analysis and nanoscopic structures. Profile measurements as well as lateral pitch measurement, e.g. for semiconductor lines, have been established. Regarding deformation measurements at material defects or micro cracks most of the published approaches are qualitative of semi-quantitative. Examples are in-situ straining experiments on magnetic thin films carried out by Bobji and Bhushan [1] or the evaluation of crack propagation in NiAl single crystals which was analyzed by Göken et al.[2] Recently, research on the combination of atomic force microscope (AFM) images and digital image correlation (DIC) algorithms proofs the ability to measure deformations on the nanoscale. The authors of the paper made use of AFM equipment for deformation field measurement [3-6]. Chasiotis/Knauss [7,8] and Soppa [9] used similar approaches to measure strain fields from AFM micrographs. In this paper the underlying basic principles of the DIC method and the stability and reproducibility will be discussed. The application of AFM based image correlation on micro- and nanomaterials will be shown by a crack analysis of a thermoset polymer material.

2 NANODAC PRINCIPLE

Digital image correlation methods on gray scale images were established by several research groups. Examples from different fields of application can be found in various publications, e.g. in [3-7,10-12]. In previous research the authors developed and refined different tools and equipment in order to apply scanning electron microscopy (SEM) images for deformation analysis on thermo-mechanically loaded electronics packages. The respective technique was established as microDAC, i.e. micro Deformation Analysis by means of Correlation algorithms [11].

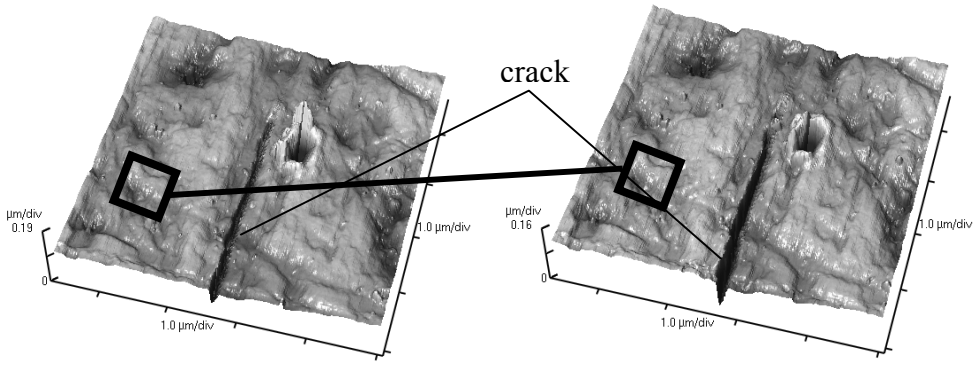

Figure 1: AFM topography image of a crack in a thermoset polymer material for different crack opening displacements, scan size 4.6 × 4.6µm; inside the black rectangles typical topographic features are visible, which can be tracked by DIC; (the indentation near the crack tip is a indentation caused by a cantilever approach)

The microDAC technique is a method of digital image processing. Digitized micrographs of the analyzed objects in at least two or more different states (e.g. before and during/after mechanical or thermal loading) have to be obtained by means of an appropriate imaging technique. Generally, the utilized cross correlation algorithms can be applied to images extracted from a variety of sources such as SEM or laser scanning microscopy (LSM). The basic idea of the underlying mathematical algorithms follows from the fact that images commonly allow to record local and unique object patterns, within the more global object shape and structure. These patterns are maintained, if the objects are stressed by thermal or mechanical loading. Figure 1 shows examples of AFM topography images taken at a crack tip of a polymeric material. Markers indicate typical local patterns (i.e. topographic structures) of the images. In most cases, these patterns are of stable appearance, even if severe load is applied to the specimens so that they can function as a local digital marker for the correlation algorithm.

For enhancement of resolution a so-called subpixel analysis is implemented in the utilized software. The result of the two-dimensional cross correlation in the surroundings of a measuring point primarily gives the two components of the displacement vector. Applied to a set of measuring points (e.g. to a rectangular grid of points with user defined pitches), this method allows to extract the complete in-plane displacement field. These results can be displayed in the simplest way as a numerical list which can be post-processed using standard scientific software codes. Commonly, graphical representations such as vector plots, superimposed virtual deformation grids or color scale coded displacement plots are implemented in commercially available or in in-house software packages. Fig. 2 shows two typical examples of graphical presentations for the results at an AFM image.

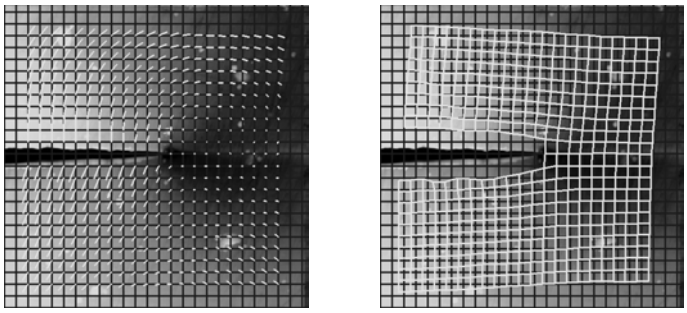

Figure 2: Digital image correlation results derived from SFM images of a crack tip, scan size 15 × 15µm; (left) image overlaid with user defined measurement grid and vector plot; (right) image overlaid with user defined measurement grid and deformed measurement grid; displacement vector and deformed grid presentation are enlarged with regard to the image magnification.

3 CRACK EVALUATION

Cyanate ester resins and their modifications which are typically used in microelectronic applications show a high modulus of elasticity but poor resistance to fracture [13]. Due to the miniaturization in electronic packages micro material testing of such material systems will be a key for successful design of microelectronic packages. Therefore this type of material is chosen for crack evaluation experiments.

3.1 Experimental set-up

A simple specimen configuration is selected to demonstrate the fundamental approach. With a compact tension (CT) crack test specimen as shown in Fig. 3 Mode I (opening) loading of the crack tip is enabled. The CT-specimen is loaded with the force *P* by a special tension/compression testing module, which can be utilized for in-situ SEM and SPM measurements. Figure 3 shows the CT-specimen and parts of the loading device under the SPM.

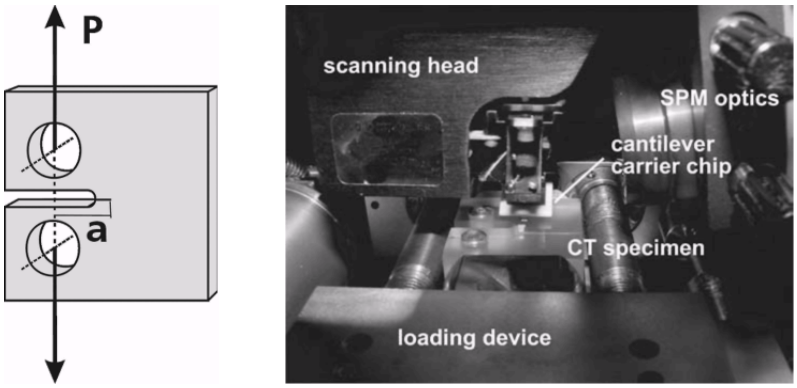

Figure 3: (left) Compact tension (CT) specimen; (right) In-situ loading under the AFM.

For the deformation measurements AFM non-contact topography scans are taken directly at the crack tip before and after loading. Afterwards the DIC algorithms are applied to the set of images and displacements are determined at predefined grid nodes. Figure 4 (left) shows the results in form of a crack opening displacement field, u_y, extracted from the AFM scans at the cyanate ester resin CT-specimen.

3.2 Crack opening displacement analysis

A straightforward approach for crack evaluation in the AFM is the technique of crack opening displacement (COD) determination. In order to extract the mode I stress intensity factor K_I crack opening displacements, u_y^u and u_y^l, are measured along both the upper and lower crack boundaries.

If determined by linear elastic fracture mechanics they must equal to:

$$u_y^{u,l} = \pm \frac{K_I}{2\mu}\sqrt{\frac{x}{2\pi}}(k+1) \qquad \text{for } x \le 0 \qquad (1)$$

$$u_y^u = u_y^l = 0 \qquad \text{for } x > 0 \qquad (2)$$

where μ, is the shear modulus and k is a function of Poisson's ratio, ν; k = (3-4 ν) for plane strain and k = (3- ν)/(1+ ν) for plane stress. Taking the square of the difference of upper and lower displacements, we obtain a linear function of the x-coordinate or 0, depending on the position relative to the crack tip:

$$\left(\frac{u_y^u - u_y^l}{2}\right)^2 = Cx \qquad x \le 0 \qquad (3)$$

$$= 0 \qquad x > 0$$

For the equation above, the crack tip is set at location x = 0. The crack tip location on the real specimen can be found at the interception of a linear fit of the curve Cx with the x-axis. The slope C allows to estimate the stress intensity factor K_I, which is a measure of the crack tip load. It is given by:

$$K_I = \frac{E}{1+\nu}\frac{1}{k+1}\sqrt{2\pi C} \qquad (4)$$

where E is the Young's modulus.

The discussed analysis is applied to the displacement field measurements presented in Fig. 4 (left). The results of the linear fit according to Eqn. 3 are shown in Fig. 4 (right).

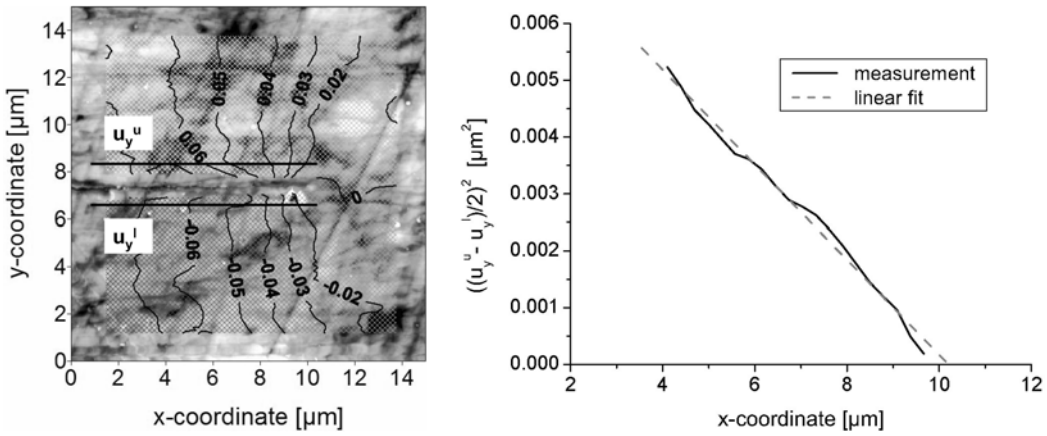

Figure 4: (left) AFM image of crack tip area (size: 15 × 15µm) with overlaid displacement results in *y*-direction, u_y , lines for the upper and lower crack face are included; (right) evaluation of slope *C* for the calculation of the stress intensity factor K_I.

The determined value for K_I with the application of Eqn. 4 equals to 0.056 MPam$^{1/2}$ which is about 1/10 of the critical stress intensity factor for the cyanate ester resin. The value calculated from the applied loads is 0.085MPam$^{1/2}$. The comparison shows that the value calculated from the specimen loading is 1.5 times higher than the value extracted from the crack opening field. Possible reasons or error sources for the deviation are:

- the value of the Young's modulus is assumed too low for this CT specimen,
- the crack is not fully loaded at the surface of the specimen, due to the fact that the crack front is a relatively straight line normal to the specimen surface,
- the plane strain crack field solution is not accurate or is not completely predominating at the surface of the specimen.

4 SUMMARY

In this article the principle of DIC-based displacement measurements at in-situ loaded structures under the AFM is successfully applied to crack tip evaluation of a polymer material. The measurements were carried out at a commercially available scanning probe microscope equipped with specially designed loading stages. The presented nanoDAC method is suited for in-situ thermomechanical measurements of MEMS and sensor components. Material data such as fracture properties, Young's modulus, coefficient of thermal expansion, Poisson's ratio can be determined.

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