Nanoscale Resolution Deformation Measurements at Crack Tips of Nanostructured Materials and Interface Cracks

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ABSTRACT

The trend towards the application of nanoparticle filled materials in the aerospace and automotive electronics sectors have led to a strong need in material characterization on the micro and nano scale. Another challenging task is the development and evaluation of interface concepts of biological structures to microelectronic materials such as polymers, metals, ceramics and semi-conducting materials. To fulfil these needs new strategies for reliability assessment on the submicron scale are essential. Under this prerequisite Scanning Probe Microscopy (SPM) serves as the basis for the development of the nanoDAC method (nano Deformation Analysis by Correlation), which allows the determination and evaluation of 2D displacement fields based on SPM data. In-situ SPM scans of the analyzed object are carried out at different thermo-mechanical load states. The images are compared utilizing grayscale cross correlation algorithms. This allows the tracking of local image patterns of the analyzed surface structure. The derived results are full-field displacement and strain fields. Due to the application of SPM equipment deformations in 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 mechanical characterization of material interfaces can be carried out with highest precision.

Keywords: nanoDAC, nanodeformation, AFM, SPM, deformation measurement

1 INTRODUCTION

With the demands for low cost electronics stacking and packaging of ICs is essential for the design of microelectronic systems. In addition to the IC and module level the integration on system level is achieved by embedded active and passive components. Regardless of the integration level thermo-mechanical challenges have to be solved due to the fact that material interfaces become even more important. Another challenge in electronic packaging is the integration of multiple device technologies such as digital, RF and MEMS, optoelectronics on the same packaging platform. Loading such structures thermally

and/or mechanically means to stress the structure within submicron and nano-scale volumes caused by severe material mismatch. Therefore, actual loading causes local stresses and strains due to different material properties such as coefficient of thermal expansion (CTE), Young's modulus or time depended viscoelastic or creep properties. The smallest existing material imperfections or initial micro/nano-scale defects can grow under stress and strain and can finally cause failure of the device. Due to these facts efforts have to be made to gain a better understanding of the material responses at submicron and nano scale and at material interfaces. The way to achieve this aim is the combination of displacement and strain measurements on the micro-and nano-scale with modeling techniques such as finite element analysis or molecular modeling. Under this prerequisite Scanning Probe Microscopy (SPM) serves as the basis for the development of the nanoDAC method (nano Deformation Analysis by Correlation), which allows the determination and evaluation of 2D displacement fields based on SPM data.

From the experimental point of view measurements of thermo-mechanically induced deformations and strains at the nano scale can be carried out by state of the art nanotechnological microscopy. Research on the combination of atomic force microscope (AFM) images and digital image correlation (DIC) algorithms proofs the ability to determine nanodisplacements at microelectronic components and MEMS. The authors of the paper made use of AFM equipment for deformation field measurement [1-4]. In this paper the underlying basic principles of the digital image correlation (DIC) method will be presented. The application of nanoscale displacement measurement technique on micro- and nanomaterials will be shown by a crack analysis of a thermoset polymer (cyanate ester thermoset) material.

2 NANODAC PRINCIPLE

Digital image correlation methods on gray scale images were established by several research groups. 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, which

means micro Deformation Analysis by means of Correlation algorithms [5]. 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, images extracted from a variety of sources such as SEM or laser scanning microscopy (LSM) can be utilized for the application of digital cross correlation. 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. In the case of atomic force microscopy (AFM) topography images structures (patterns) are obtained by the roughness of the analyzed object surface. Figure 1 shows examples of AFM topography images taken at a crack tip of a polymeric material.
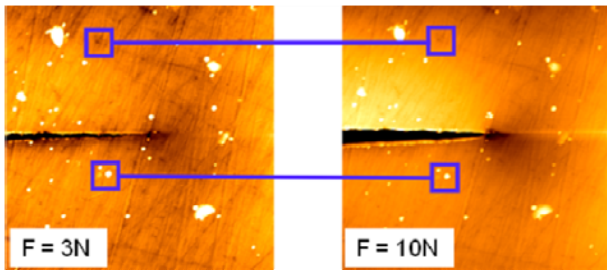

Figure 1: AFM topography scans [15 µm × 15 µm] at a crack tip of a polymer CT (compact tension) specimen; the scans are carried out at different load states.

Markers indicate typical local patterns (i.e. topographic features) 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. The cross correlation approach is the basis of the DIC technique. A scheme of the correlation principle is illustrated by Fig. 2.
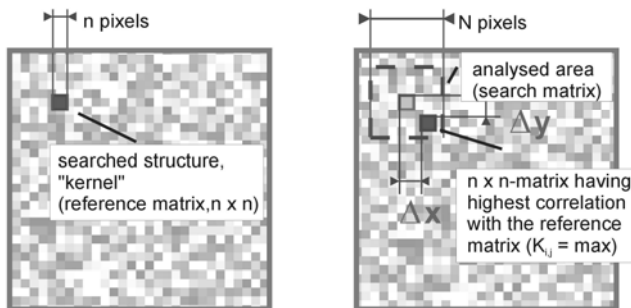

Figure 2: Displacement evaluation by cross correlation algorithm; (left) detail of a reference image at load state 1; (right) detail of an image at load state 2 [6].

Images of the object are obtained at the reference load state 1 and at a different second load state 2. Both images are compared with each other using a cross correlation algorithm. In the image of load state 1 (reference) rectangular search structures (kernels) are defined around predefined grid nodes (Fig. 2, left). These grid nodes represent the coordinates of the center of the kernels. The kernels themselves act as gray scale pattern from load state image 1 that have to be tracked, recognized and determined by its position in the load state image 2. In the calculation step the kernel window (n × n submatrix) is displaced inside the surrounding search window (search matrix) of the load state image 2 to find the position of best matching (Fig. 2, right). This position is determined by the maximum cross correlation coefficient which can be obtained for all possible kernel displacements within the search matrix. The described search algorithm leads to a two-dimensional discrete field of correlation coefficients defined at integer pixel coordinates. The discrete field maximum is interpreted as the location, where the reference matrix has to be shifted from the first to the second image, to find the best matching pattern. For enhancement of resolution a so-called subpixel analysis is implemented in the utilized software [6]. The two-dimensional cross correlation and subpixel analysis 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 a user defined pitch), this method allows to extract the complete in-plane displacement field. 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 [7, 8]. Finally, taking numerically derivatives of the obtained displacement fields $u_x(x,y)$ and $u_y(x,y)$ the in-plane strain components ε and the local rotation angle ρ are determined.

For images originating from scanning probe microscopy (SPM) techniques the described approach has been established as so-called nanoDAC method (nano Deformation Analysis by Correlation) [1]. This method is particularly suited for measurement of displacement fields with highest resolution focused on MEMS/NEMS devices and micro and nano-structural features of typical microelectronics materials.

3   CRACK EVALUATION

3.1   Experimental set-up

In a typical nanodeformation measurement session in-situ AFM scans of the analyzed object are carried out at different thermo-mechanical load states as shown in Fig. 1. In the illustrated case an AFM topography signal serves as the image source. It is also possible to use other SPM imaging signals such as Phase Detection Microscopy or Ultrasonic Force Microscopy. The AFM scans are taken at

the vicinity of a crack at a compact tension (CT) crack test specimen, Fig. 3. The CT-specimen is loaded with the force *F* by a special tension/compression testing module so that a Mode I (opening) loading of the crack tip is enabled. Figure 3 shows the CT-specimen and parts of the loading device under the AFM.
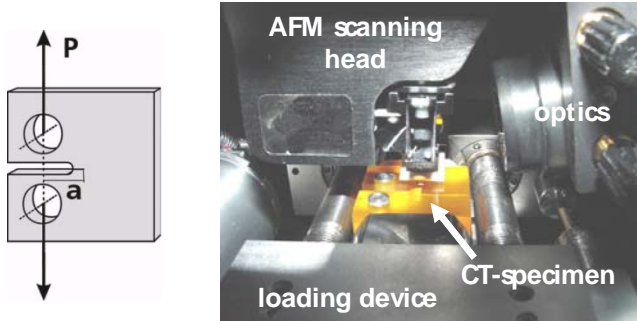

Figure 3: (left) Compact tension (CT) specimen; (right) In-situ loading under the AFM.

For images of the discussed loading of a thermoset polymer CT-specimen as given in Fig. 1 an extracted vertical (crack opening) displacement field is illustrated in Fig. 4.
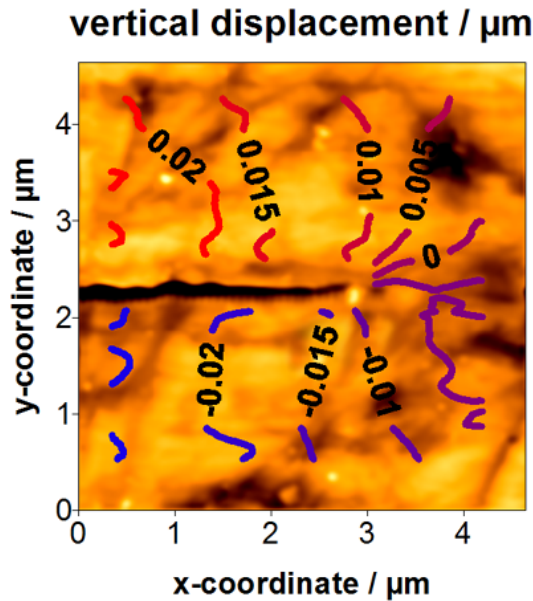

Figure 4: Crack opening displacement field in vertical (*y*)-direction [μm] determined by means of nanoDAC; in the background of the contour lines an AFM topography scan is illustrated.

Due to the application of SPM equipment deformations in the micro-, nanometer range can be easily detected. Currently the accuracy of the nanoDAC method for displacement field measurement is 1 nm for scan sizes of 2 μm, where the accuracy is determined by the thermo-

mechanical stability of the SPM system. Details on the effect of thermal drifts and typical SPM related stability issues are discussed in [9]. In addition this reference shows compensation strategies for such error sources. The measurement technique 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 nano-cracks or defects in bulk materials, thin layers and at material interfaces can be carried out. An example of the determination of crack parameters based on nanoDAC displacement fields is shown in the following section.

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 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:

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

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

where μ, is the shear modulus and k is a function of Poisson's ratio, ν; $k = (3-4\nu)$ 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:

$$\left(\frac{u_y^u - u_y^l}{2}\right)^2 = Cx \quad \text{for} \quad x \leq 0 \quad (3)$$

$$= 0 \quad \text{for} \quad x = 0$$

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 estimating the stress intensity factor K_I, which is a measure of the crack tip load. It is given by:

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

where *E* is the Young's modulus.

The discussed analysis is applied to the displacement field measurements presented in Fig. 5. The results of the linear fit according to Eqn. 3 are shown in Fig. 6.

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.

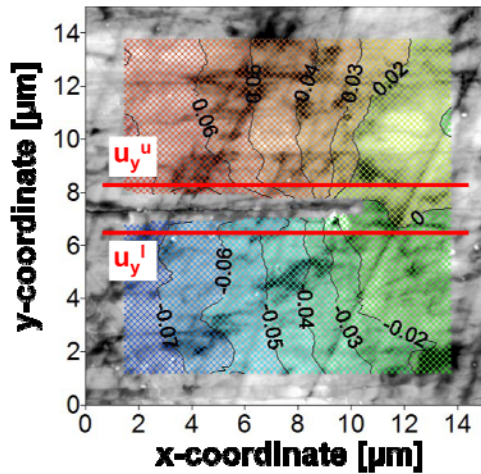Figure 5: AFM image of crack tip area (15µm × 15µm) with overlaid displacement results in y-direction u_y; lines for the upper and lower crack face are included
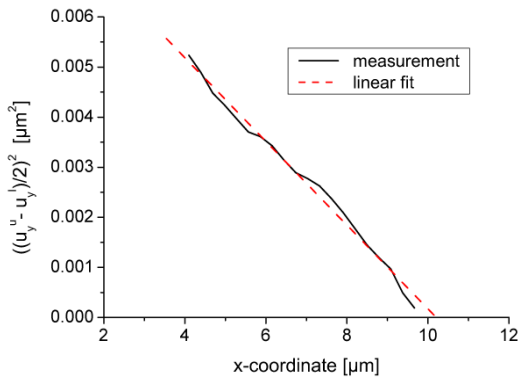

Figure 6: Evaluation of slope C for the calculation of K_I

4 CONCLUSIONS

The principle of digital image correlation 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 SPM 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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