

# Nanoscale Deformation Measurements – Concepts for Failure and Reliability Assessment at the Nanoscale

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## ABSTRACT

The paper presents two methods for deformation measurement at the nanoscale level. The first method is based on Scanning Probe Microscopy (SPM) in combination with Digital Image Correlation (DIC). The technique 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. The second approach for nanoscale deformation measurements is the so-called fibDAC (FIB, Focused Ion Beam) method. It provides the classical hole drilling method for residual stress measurement for the nanoscale region. The ion beam of the FIB station is used as a milling tool which causes the stress release. With the combination of fibDAC and finite element analysis stresses of silicon microstructures of MEMS devices or at other pre-stressed materials or surface coatings can be determined. Both presented methods can be applied for experimental reliability evaluation in microelectronics packaging, MEMS and NEMS. In addition residual stress determination at ultrathin layers and at microstructural features of bulk materials can be approached.

**Keywords:** nanoDAC, fibDAC, nanodeformation, residual stress measurement

## 1 INTRODUCTION

“Nanoreliability” is a name to describe a branch of research activities taking into account effects arising from the nanoscale new for reliability analysis and lifetime estimation. An advanced nanoreliability approach requires new tools for local stress and strain evaluation taking into account structural effects and physics of failure concepts combined with those continuum-based calculations which are suited to describe the essential procedures of the dominant failure mechanisms sufficiently well. Strain fields are coupled with thermal effects, diffusion phenomena, vibrations and various kinds of local changes of structure (defects, defect interactions etc.). Most of these phenomena have been shown to be related with internal stresses. That’s why an advanced reliability analysis based on local deformation field characterization also requires an

incorporation of local intrinsic stress analysis. By means of X-ray diffraction some information will be obtained, but very often this is not sufficient enough because local strains on a submicrometer scale have to be taken into account.

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.

## 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.

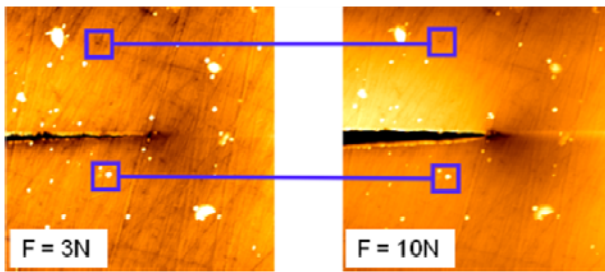


Figure 1: AFM topography scans [ $15\ \mu\text{m} \times 15\ \mu\text{m}$ ] at a crack tip of a polymer CT (compact tension) specimen; the scans are carried out at different load states.

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.

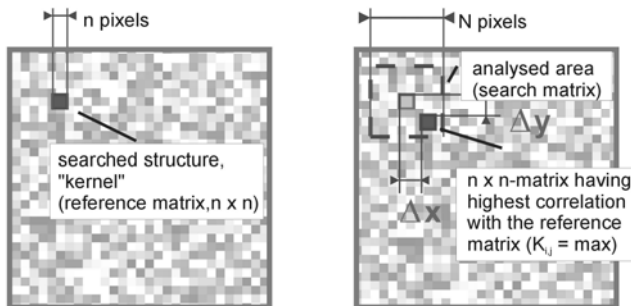


Figure 2: Displacement evaluation by cross correlation algorithm; (left) detail of a reference image at load state 1; (right) detail of an image at load state 2 [6].

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 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 ( $n \times n$  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  $\varepsilon$  and the local rotation angle  $\rho$  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

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.

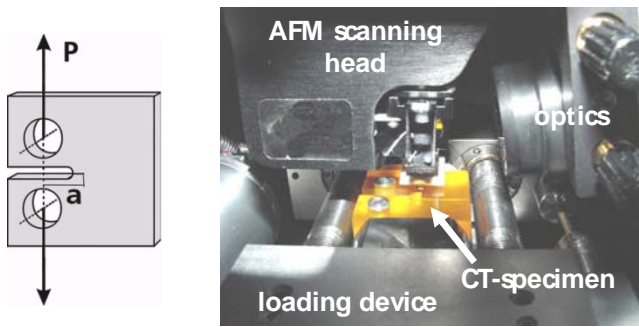


Figure 3: (left) Compact tension (CT) specimen; (right) In-situ loading under the AFM.

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.

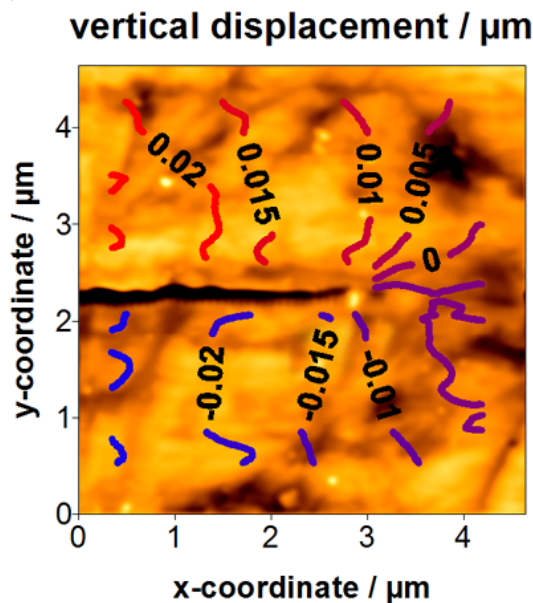


Figure 4: Crack opening displacement field in vertical ( $y$ )-direction [ $\mu\text{m}$ ] determined by means of nanoDAC; in the background of the contour lines an AFM topography scan is illustrated.

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  $\mu\text{m}$ , 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.

## 4 FIBDAC PRINCIPLE

Measurement of residual stresses is an important demand for MEMS and sensor development. Loading of devices can produce stresses, which superpose with inherent residual stresses. Because internal stresses cannot be measured directly as forces, indirect approaches have to be looked for. One classical method is the release of residual stresses by material removal and subsequent measurement of induced respective strains at the object surface. The method became established as hole-drilling strain gage method [10], where through or blind holes are processed mechanically into the material. Released strains are commonly measured by strain gages attached to the object surface. Unfortunately, mechanical or laser based material removal is restricted in size. Also strain gages can not be placed easily on the object surface of sensors or MEMS. For these reasons, FIB milling seems to be an effective tool to extend the hole-drilling approach to submicron or even nano scale. Accompanying FIB material removal with spatially high resolution deformation measurement methods like DIC or Moiré is another prerequisite to downscale the classical method to the micro and nano region.

Therefore the fibDAC (focused ion beam based Deformation Analysis by Correlation) was developed. In the presented example the ion beam of the FIB station is used as a milling tool which causes the stress release at silicon microstructures of a MEMS device. Figure 5 shows an overview of the device (gas sensor) and the FIB-milled hole for stress release measurement.

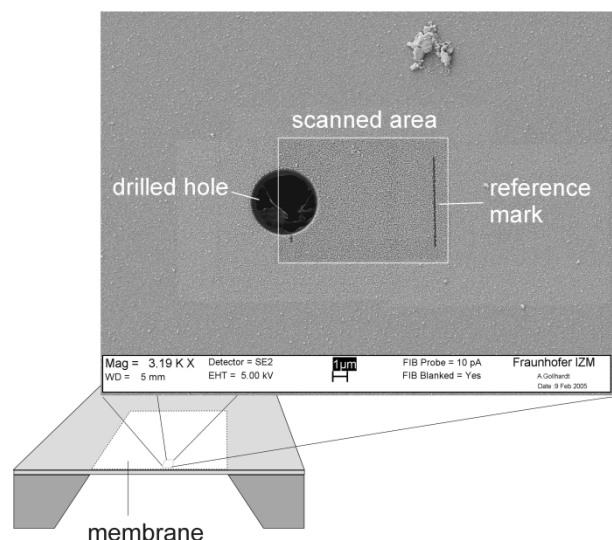


Fig 5: Scheme of micromachined membrane and overview of the scanned area after hole-milling process

## ACKNOWLEDGEMENTS

The authors wish to acknowledge the collaboration with Neus Sabate and Carles Cane from Departament d'Electronica, Universitat de Barcelona (EME-UB), Spain.

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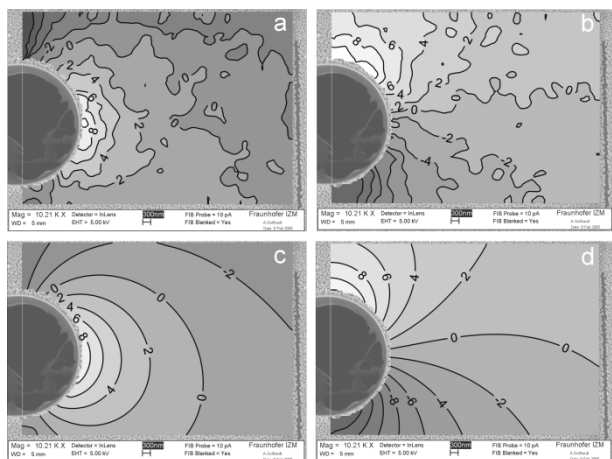


Figure 6: a) Displacement in horizontal direction,  $u_x$   
b) Displacement field in vertical direction  $u_y$   
c)  $u_x$  analytical fit d)  $u_y$  analytical fit; all data in [nm]

The analysis of the stress release is achieved by DIC applied to load state SEM images captured in cross beam equipment (combination of SEM and FIB). The results of the DIC analysis are displacement fields  $u_x(x,y)$  and  $u_y(x,y)$  with particular displacement of the analyzed image. Figure 6(a) and (b) shows the experimental contour lines for  $u_x$  and  $u_y$  respectively. The experimental results can now be fitted to displacement fields calculated by finite element analysis or analytical solutions, Fig. 6 (c) and (d). Fitting has to be performed for the whole 2-D displacement field with respect to the rigid body translation and rotation. With the knowledge of other material parameters (Young's modulus, Poisson's ratio for isotropic materials) residual stresses can be evaluated.

In another step which is not discussed in this paper the resolution of the method has been improved by the application of trench milling instead of milling of holes [11]. Thereby the accuracy of the method has been improved.

## 5 CONCLUSIONS

A new approach for nanodeformation measurement based on SPM images has been established. With the nanoDAC method research of nanomechanical effects can be addressed directly with the combination of nanoanalytical SPM techniques and digital image correlation (DIC) software codes. In addition the fibDAC method gives access to high resolution residual stress measurements. Stresses are determined by trench opening/closing displacement measurement based on DIC results in combination with analytical solutions or Finite-Element modelling. Both methods are suited for fundamental and applied research at MEMS/NEMS and micro- or nanostructural building blocks of bulk materials.