JPK BIOAFM **Product Note**

Bruker BioAFM Contact Resonance (CR) Module

Introduction

The quantitative characterization of nanomechanical properties with high resolution has always been a major advantage of atomic force microscopy (AFM). Several technics have been developed in the last decade, such as PeakForce QNM® and QI™, that offer the nanomechanical characterization of a sample with both high resolution and speed. However, as most of these characterization techniques are based on force spectroscopy measurements, the finite stiffness of the cantilever effectively limits the addressable stiffness of the sample to about 10 GPa[1].

Until now, nanoindentation was mainly used to characterize stiffer samples, which limits the resolution that can be achieved and typically damages the sample. These limitations affect solid state physics and biology, where stiff materials like teeth, bones, implants and so forth need to be characterized in great detail. Fortunately, using the contact resonance technique, the strengths of AFM can be combined with the ability to mechanically characterize samples with a stiffness of up to 300 GPa. In this method, the cantilever is held in steady contact with the sample (contact-mode), while, in addition, being excited either through the tip[2,3] (**U**ltrasonic **AFM**) or through the sample (**A**tomic **F**orce **A**coustic **M**icroscopy)[2,4]. Figure 1(A) depicts the principal setup of an AFAM experiment.

A small excitation (typically less than a nanometer) of the cantilever via an ultrasonic transducer is used to determine the resonance frequency and quality factor of the cantilever while it is in contact with the sample, either by sweeping or by phase-locked-loop-based (PLL) resonance tracking techniques. The resonance frequency and shape of the contact resonance peak change in response to the tip-sample interaction, depending on, for example, stiffness and dissipation, as shown in Figure 1(B). Higher contact resonance frequencies correspond to stiffer materials, while contact resonance peaks with a higher quality factor correspond to less dissipation or damping and vice versa. Although contact resonance was originally developed to measure the elastic properties of stiff materials with improved sensitivity[3,4], it has evolved to also measure viscoelastic properties like storage and loss modulus (E', E", tan δ)[4-8].

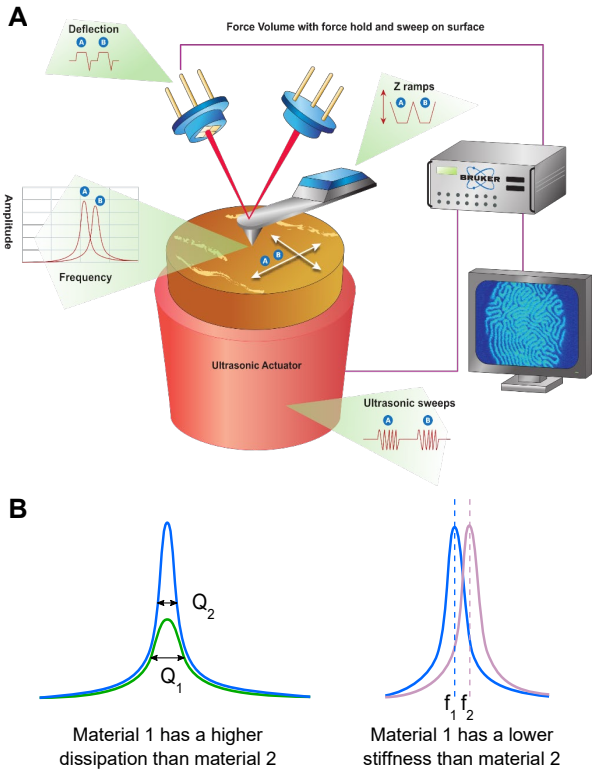

Figure 1: *(A) Setup of an AFAM experiment. The ultrasonic actuator located underneath the sample is used to excite the cantilever. (B) Response of the cantilever resonance on two different materials with different properties. (images taken from Bruker Application Note #148)*

Content of the Bruker BioAFM CR module

Until now, the implementation of contact resonance was hindered by its slow imaging speed, complex analysis, and the specialized hardware required for imaging and full-spectrum acquisition. Bruker has removed these hurdles by providing customized hardware, test/reference samples and flexible,

Figure 3: *Contact Resonance module sample support with integrated ultrasonic transducer (bottom left). NanoWizard® BioAFM with Contact Resonance module in place (top right).*

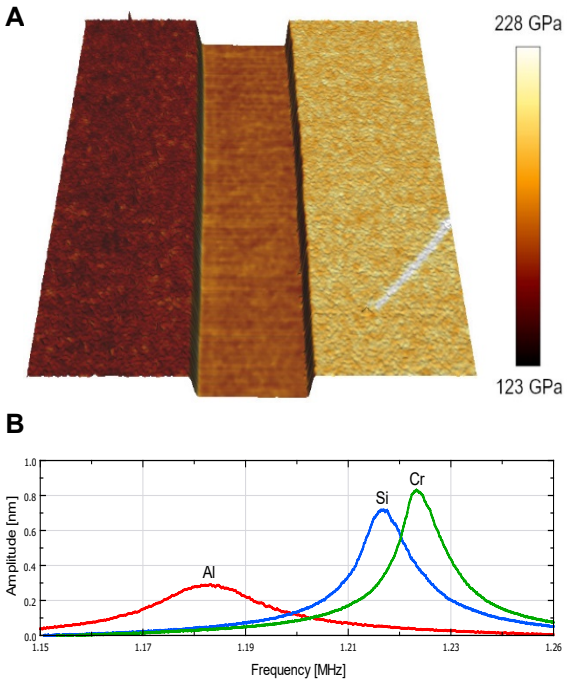

Figure 2: *(A) Contact resonance scan of the reference sample, as contained in the package. It shows a 3D-representation of the topography overlaid with the color scale of the indentation modulus. (B) Representative contact resonance sweeps on the 3 different materials aluminum, silicon and chromium of the reference sample.*

easy-to-use software. The contact resonance module contains the following major components:

1. **Sample holder with ultrasonic transducer** for excitation of the cantilever (see Figure 2).
2. **Extensive software package** which enables flexible, easy-to-use data acquisition and analysis.
3. **A set of reference and calibration samples** containing a layered reference sample made of aluminum, silicon and chromium (see Figure 3), and several calibration samples of known moduli.
4. **A set of probes** with a diamond-like carbon coating providing high wear resistance.

Operation Modes

The CR module offers two operation modes for collecting the contact resonance data, a classical PLL-based imaging mode and a fast, force mapping-based mode.

PLL-based Imaging Mode

The PLL-based imaging mode scans the sample in the classical contact mode, while a PLL is used to track the actual value of the contact resonance frequency. In this way, the storage modulus of the sample can be determined with typical imaging speeds and resolution. Drawbacks of this operation mode are tip wear and sample damage due to the comparatively high lateral forces.

Fast Force Mapping Mode

The fast force mapping mode eliminates lateral forces by halting the motion of the XY scanner during the cantilever approach and measurement of the sample surface at each individual pixel. While the tip is in contact with the sample, a contact resonance tune is collected before the tip retracts and moves on to the next pixel.

The collected tunes can be analyzed (fitted) with respect to the frequency and quality factor of the contact resonance, thus, enabling the determination of the viscoelastic properties of the sample in addition to the storage modulus.

While tip wear and sample damage are significantly reduced by minimizing lateral forces in this mode, it comes at the expense of a slight increase in the acquisition time.

Data Analysis

Different models have been implemented in the software to derive the corresponding mechanical material properties from the acquired data. While the calculation of the storage modulus is based on one model only, namely a Bernoulli beam model of the cantilever coupled to the sample via a Kelvin-Voigt model[10,2], the calculation of the loss modulus is implemented by different, optional models[10,11,2] as the scientific community has yet to come to an agreement on the most suitable model. In general, the loss moduli derived, which depend on the accurate determination of the contact resonance peak shape, are less accurate than the derived storage moduli which rely primarily on the frequency peak position only.

In addition to the contact resonance data, the analysis requires the resonant frequency and quality factor of the oscillation modes of the cantilever in free air. Furthermore, in order to achieve more reliable data, it is recommended to perform a calibration measurement on a reference sample with known mechanical properties to eliminate typically unknown or error-prone parameters, such as tip radius and loading force. Once the system is calibrated, the software automatically performs the calculations and provides images/maps of the calculated material properties, including storage modulus and loss modulus (mapping mode only).
All of the required steps for calibration and analysis are carried out through dedicated step-by-step software modules.

A Broad Range of Applications

Contact resonance can measure a wide variety of materials, from biological samples to polymers and metals. For biological and medical applications, this enables the detailed mechanical investigation of samples, such as teeth, bones, seeds, wood, medical implants etc., which until now was not possible to investigate with this level of accuracy.

Figure 4, for example, shows the results of contact resonance imaging on a cross section of a human milk tooth. The data was acquired at the enamel-dentine junction (indicated by the blue arrows). The calculated modulus of the enamel and dentine are approximately 50 GPa and 18 GPa, respectively, which concurs well with the values found in literature[1].

Conclusion

Contact resonance can quantitatively measure the elastic modulus of specimen in a broad range of approx. 0.1GPa up to 300 GPa, while maintaining the high resolution and speed typical for AFM. By bundling specialized, easy-to-use software and hardware, Bruker's CR module has removed the main hurdles associated with contact resonance in the past. In particular, the implementation of a Fast Force Mapping-based contact resonance imaging mode significantly reduces tip wear and sample damage, while, in addition, providing quantitative measurements of viscoelastic properties. The variety of applications possible with this broad range of moduli and the ability to probe both elastic and viscoelastic properties make contact resonance a powerful tool for performing nanomechanical measurements.

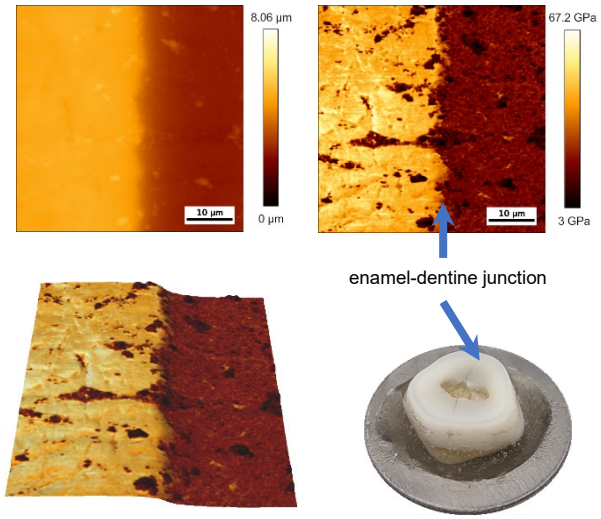

Figure 4: *Contact resonance measurement on a polished cross section of a human milk tooth showing the enamel-dentin junction. The upper row shows the topography (left) and storage modulus (right). The bottom row shows a 3D-representation of the topography overlaid with the color scale of the modulus (left) and a photograph of the investigated sample (right).*

References

1. E. Rettler, S. Hoeppener, B.W. Sigusch and U.S. Schuber, *Mapping the mechanical properties of biomaterials on different length scales: depth-sensing indentation and AFM based nanoindentation,* J. Mater. Chem. B 2013, 1, p. 2789-2806
2. U. Rabe, *Atomic force acoustic microscopy,* Applied Scanning Probe Methods, B.B.a.H. Fuchs, Editor. 2006, Springer: Berlin.
3. K. Yamanaka, H. Ogiso and O.V. Kolosv, *Ultrasonic force microscopy for nanometer resolution subsurface imaging*. Applied Physics Letters 1994, 64(2): p. 178.
4. U. Rabe and W. Arnold, *Acoustic microscopy by atomic force microscopy*. Applied Physics Letters 1994, 64(12): p. 1493–95.
5. J.P. Killgore et al., *Viscoelastic property mapping with contact resonance force microscopy*. Langmuir 2011, 27: p. 13983–87.

6. P.A. Yuya, D.C. Hurley and J.A. Turner, *Relationship between Q-factor and sample damping for contact resonance atomic force microscope measurement of viscoelastic properties*. Journal of Applied Physics 2011, 109(11): p. 113528.

7. D.G. Yablon et al., *Quantitative mapping of viscoelastic properties of polyolefin blends with contact resonance atomic force microscopy*. Macromolecules 2012, 45(10): p. 4363–4370.

8. D.G. Yablon, J. Grabowski and I. Chakraborty, *Measuring the loss tangent of polymer materials with atomic force microscopy-based methods*. submitted, 2013.

9. S.E. Campbell, V.L. Ferguson and D.C. Hurley, *Nanomechanical mapping of the osteochondral interface with contact resonance force microscopy and nanoindentation*. Acta Biomater 2012, 8(12): p. 4389–4396.

10. M.K. Phani, et al., *Elastic stiffness and damping measurements in titanium alloys using atomic force acoustic microscopy*. Journal of Alloys and Compounds, 2016.

11. P.A. Yuya, D.C. Hurley and J.A. Turner, *Contact resonance atomic force microscopy for viscoelasticity*. Journal of Applied Physics 2008, 104(7).

JPK BioAFM Business
Bruker Nano GmbH
Am Studio 2D, 12489 Berlin, Germany
productinfo@bruker.com
www.bruker.com/bioafm · www.jpk.com