High sensitivity acoustic emission module

Introduction

High frequency Acoustic Emission (AE) monitoring during nanomechanical tests such as nanoindentation, nano-scratch, nano-impact, micro-pillar compression and micro-cantilever bending provides valuable additional information to improve our understanding of material behaviour at the nano-scale [1-3]. In many cases the conventional load-depth-time record does not provide sufficient information and microscopic imaging does not directly provide information on the dynamics of the process being studied. The acoustic waves emitted during mechanical tests at the nano-micro scale are rich source of information about the deformation behaviour (e.g. initiation and propagation of cracks, plastic instabilities, etc.) that may be otherwise inaccessible.

AE monitoring is popular in studying interfacial and cohesive failures at the macro-scale. The energy released during these tests are much higher in comparison to the highly-localized volumes in nanomechanical and nanotribological tests so at the nanoscale AE sensors with optimized design have to be used to achieve sufficient sensitivity and reliability. Dedicated sample holders with inbuilt AE sensors with optimized design featuring high mechanical stiffness have been developed. Due to their advanced design with improved sensitivity and extremely low noise they are well suited for routine use in the NanoTest.

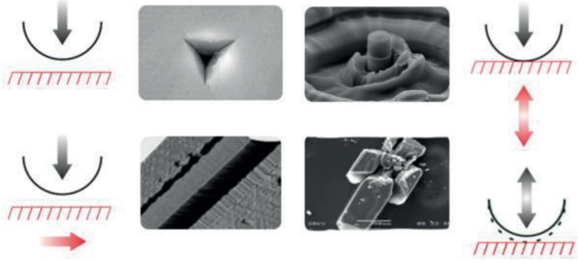

Figure 1 AE monitoring can be combined with wide range of nanomechanical tests in the NanoTest

Experimental

Acoustic emission activity was continuously recorded and analyzed throughout the nano- or micro-scale tests with a high-sensitivity ZEDO system (Dakel, Prague, Czech Republic) with a high dynamic range and sampling frequency of 10 MHz. This specification allows the study of very weak as well strong AE events at nanosecond scale in a broad frequency range up to 2 MHz. With direct synchronization with the NanoTest the depth-load-time records can be related to AE signal. During the test the samples were mounted on the low noise/high-sensitivity AE sample holders that contain dedicated structured piezo-element and inbuilt pre-amplifiers. The AE hit waveform sampled at MHz frequency is described by several characteristics that provide different insights into the origin of the detected signal. These are summarised in the Specifications on page 4. Due to very high sampling frequencies the signal envelope for specific time periods are averaged and presented as compressed overall signal envelope to provide a visual overview of the AE signal as a function of time.

In this Technical Note we illustrate several applications of the advanced AE monitoring capability and software analysis developed in collaboration with Palacky University and Dakel. Examples include pop-ins on yield of metallic materials, cracking of brittle glasses, ceramics and hard coatings in indentation, scratch and impact.

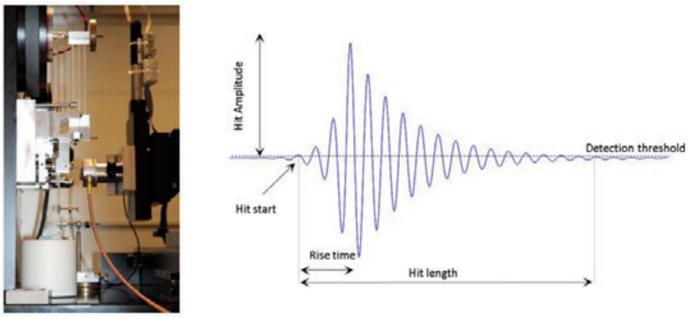

Figure 2 Implementation of AE monitoring in the NanoTest and schematic of AE waveform

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Case studies

1. Onset of plastic flow in metallic materials

The model bcc material Fe-Si(3 wt.%) can undergo an abrupt displacement burst (“pop-in”) during indentation loading. To study this a Fe3Si single crystal was prepared by floating-zone melting and electrochemically polished so that the <100> direction was perpendicular to the surface to be indented. A spheroconical indenter with end radius of 8.5 µm was used to indent the sample to 20 mN at 1 mN/s, with 10 s at this load before unloading at 2 mN/s.

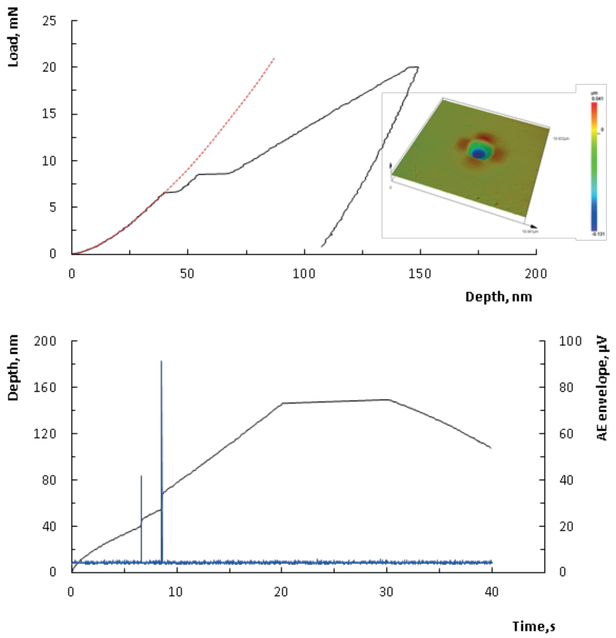

Figure 3 Indentation curve and AE envelope for Fe3Si (pop-ins coincide with AE events)

Initial stages of indentation are purely elastic and follow the Hertz theory (red curve in Fig. 3). At the critical point for the onset of plasticity sudden displacement bursts on the indentation curve (pop-ins) may be observed. Activation of slip and/or nucleation of dislocations occur accompanied by strong AE signal (blue curve). Before pop-in shear stresses are of the order of theoretical strength.

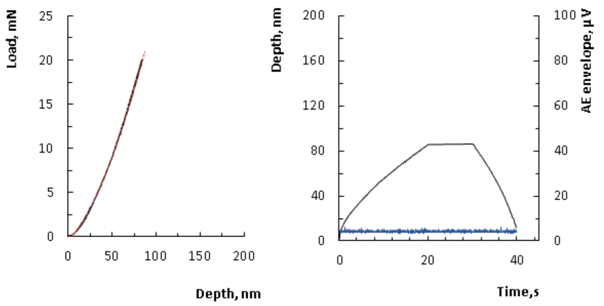

Figure 4 No dislocation activity and acoustic emission signal were detected for purely elastic indentation.

2. Cracking in nanoindentation of brittle solids

During the nanoindentation of brittle solids cracking can occur during loading, during the hold at peak load or during the unloading process. In 100-500 mN nanoindentation testing of fused silica with a Berkovich indenter there are no features on the loading curve to indicate cracking but the international standard for instrumented indentation, which the NanoTest is fully compliant with (ISO 14577), notes that cracking occurs at over 75-100 mN. AE monitoring shows discrete bursts of AE activity during loading and additionally during unloading from higher loads (Fig. 5).

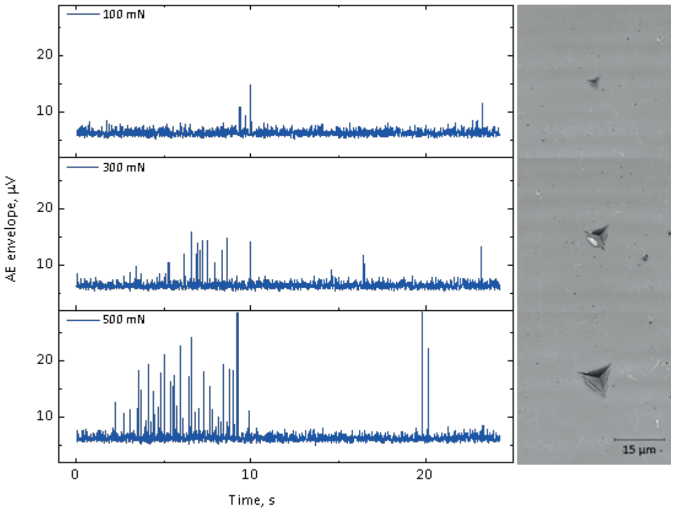

Figure 5 AE envelopes for fused silica indentation at 100, 300 and 500 mN (loading time = 10 s, hold at peak load before unloading = 5 s)

Cracking of chemically toughened glasses (such as Gorilla Glass) can require significantly more load and the NanoTest high load (30 N) head has been used to perform microindentation tests. For Gorilla Glass AE monitoring revealed cracking beginning around 700 mN with discrete bands of cracking activity as the load increases.

Advanced spectral analysis reveals differences in the frequency signatures for the events at different load levels.

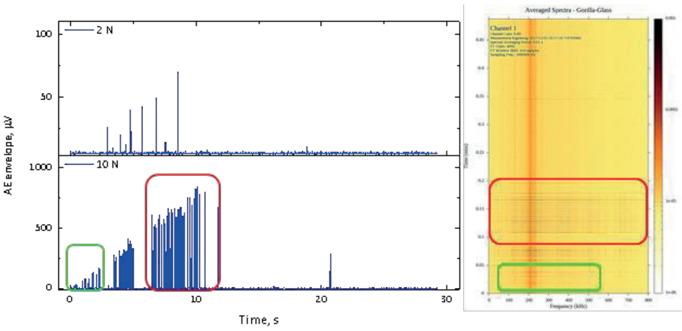

Figure 6 AE envelopes for Gorilla Glass indented to 2 and 10 N. The spectrogram for the 10 N indentation on the right shows a visual representation of the spectrum of frequencies of the AE signal as a function of time.

3. Nano-scratch of hard coatings

The assessment of critical loads in the nano-scratch test from microscopic observations of the residual scratch track can in some cases give misleading results. Initial cracks may appear much later on the visible surface than they actually initiate at the buried coating-substrate interface, resulting in an incorrect critical load identification. This behaviour is illustrated below for a 2.7 µm thick a-SiCN coating deposited using magnetron sputtering on silicon substrate. A nano-scratch test with linearly increasing load until 500 mN was performed with a 5 µm spheroconical diamond probe. Only a plastic residual groove can be seen on the top micrograph with angular cracks from the groove edges starting around the area marked “B”. However, a strong AE signal is detected from the interface cracking at a lower critical load (point "A").

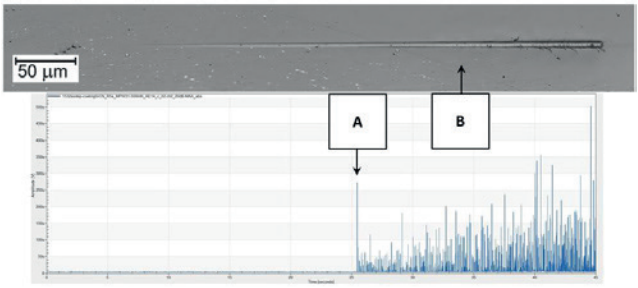

Figure 7 Micrograph of residual scratch of SiCN-Si coating-substrate system with simultaneous AE signal record

4. Nano-impact of partially-stabilised zirconia

The behaviour of partially-stabilised zirconia (PSZ) has been evaluated under repetitive impact under very low load with a sharp cube corner probe. The indenter was accelerated from 20 µm above the surface with 5 mN impact load to produce an impact every 4 s for 300 seconds (75 impacts). The resistance to impact fatigue was assessed by following the progression of the impact depth with continued impacts.

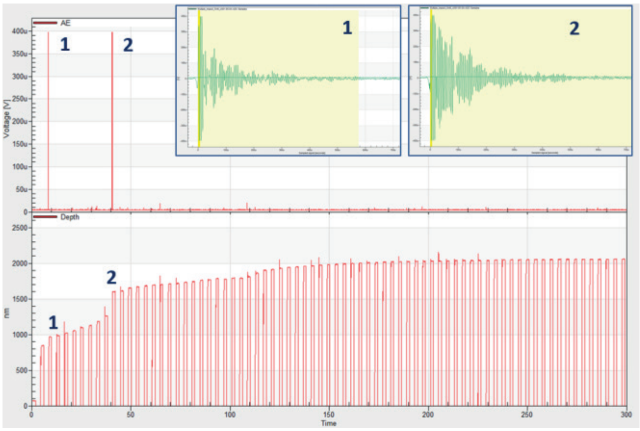

Figure 8 Repetitive impact on PSZ at 5 mN. AE bursts accompany the abrupt displacement increases

PSZ showed a brittle response with progressive deformation and in some cases abrupt displacement bursts. AE was able to show that they were due to cracking rather than slip.

An example is shown in Figure 8. In this test there were two large AE signals (labelled 1 and 2) on impacts #2 and #10. In these tests the AE bursts were correlated to abrupt displacement increases. The hit detector records for these are shown in the two inserts at the top of the figure (y-axis covers 700 µs). These reveal that the AE burst for the larger displacement step (impact #10) was longer (inset 2).

5. Fracture of silicon carbide micro-cantilevers

AE monitoring can provide additional information on the nature of fracture during bending of micro-cantilevers. Micro-cantilevers were FIB prepared from matrix and interphase regions of SiC/SiC fibre-matrix composites at the University of Oxford.

These were loaded with a 2 µm diamond probe until failure. An abrupt increase in displacement occurred when the cantilevers fractured. The results of five of these tests are shown in Figure 9.

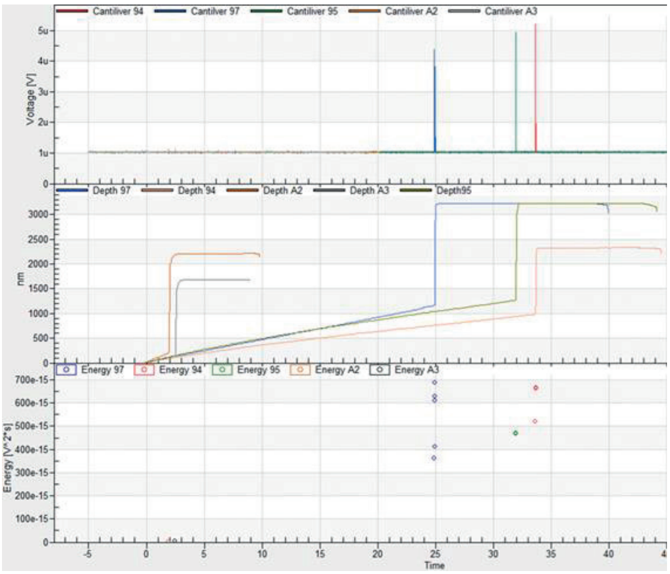

Figure 9 Integrated AE and NanoTest results for cantilever bending tests. Top = AE envelope; Middle = depth vs. time; Bottom = Hit detector records

The interphase cantilevers required ≈0.1 mN to drive failure whereas the matrix cantilevers required ≈1.5 mN. This larger load was associated with a much greater burst of AE after 25-33 s.

The detailed Hit detector records showed that for some cantilevers multiple discrete fracture events were involved in the failure process.

NanoTest AE module Specifications

- Integrated ZEDO AE sensor with inbuilt pre-amplifier (42 dB)
- Continual and triggered sampling and signal storage
    - Frequency bandwidth 30 kHz – 2 MHz
    - 16-bit@10MSPS A/D converter
    - time resolution 1 ns
    - total gain up to (42 + 70) dB
- Synchronised NanoTest and ZDAEMON software
    - Intuitive and robust user-friendly control and analysis software
    - Automatic data export
    - Comprehensive data post-processing
    - 3 independent real-time + unlimited postprocess hit detectors
- Analysed AE parameters include signal amplitude, RMS, ASL, signal energy, HIT rise time, AE counts, cumulative counts, spectral analysis….
- AE monitoring fully integrated with NanoTest indentation, scratch and impact modules in low load (0-500 mN) and 0-30 N high load range.

AE Parameter	Information on
Signal Amplitude	Intensity of the AE generating event (e.g. cracking, plastic deformation, phase transition, delamination, debonding, friction etc.)
Signal Energy	Extent of the AE event
Hit rise time	Lifetime of the physical event generating elastic strain waves
Number of counts	Statistics of AE events, crack propagation
Advanced frequency analysis	Detailed “signature” of the AE event – spectrogram provides a visual representation of the spectrum of frequencies in the AE signal as a function of time

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Conclusions

1. The development of a fully integrated AE monitoring capability with advanced analysis overcomes limitations from relying only on the displacement-load history, in some cases revealing cracking not visible at all in the displacement data in nanoindentation, nano-scratch, nano-impact and micro-cantilever bending tests.

2. AE analysis provides a better understanding and more complex interpretation of nanomechanical and nanotribological results at nano/micro scale. Due to its high sensitivity many different fracture and yield-related phenomena can be explored.

3. AE coupled with nanomechanical tests enables detection of the very first sub-surface cohesive and/or adhesive failures as well as the fundamental processes governing plastic deformation that are inaccessible by other methods.

Benefits of AE measurements

- Improved accuracy in scratch test critical load
- Uncover cracking events in nanoindentation where load-depth profiles are smooth
- Differentiate between slip and fracture in impact
- Study yield behaviour
- Differentiate between different cracking processes
- Improved understanding of nanotribological tests

References and acknowledgements

[1] N.H. Faisal et al, Int Mater Rev (2011) 98-142.
[2] J. Tomastik et al, Coatings 8 (2018) 196.
[3] J. Tomastik et al, Sci Rep 8 (2018) 10428.

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