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Automated Control of Stable Crack Growth for R-curve and Rapid v-K-Curve Measurements

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(This presentation is extended and slightly revised. Some animations are removed.)

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Outline

1. **Introduction**
2. **Crack growth (theoretical basis)**
   a) R-curves
   b) v-K-curves
3. **Experimental set-up**
   a) Principle of measurement
   b) Operation of machine
   c) Data reduction
4. **Measurements (glass, Si_3N_4)**
   a) Tests (Young's modulus)
   b) R-curves (load-displacement diagrams)
   c) v-K-curves
5. **Summary and future prospects**

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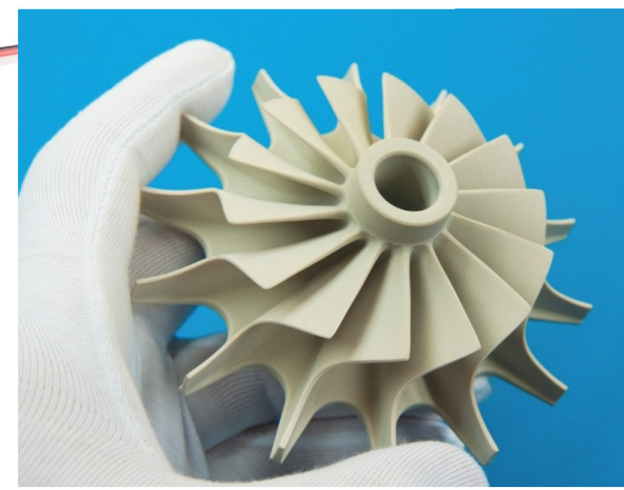
Technical challenge



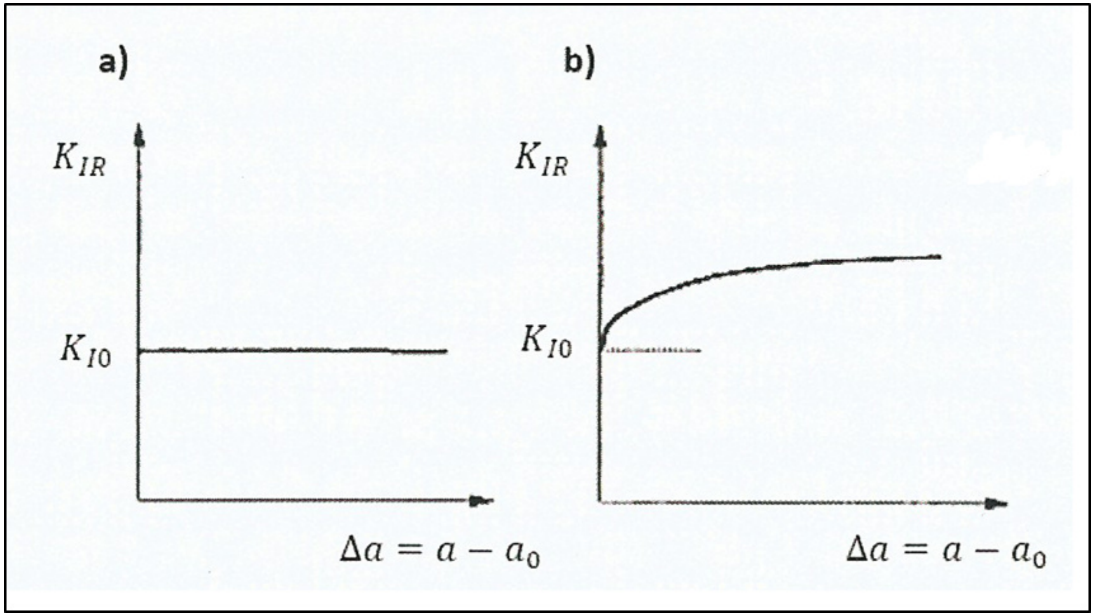
Measurement of
- R-curves
- v-K-curves
- Young‘s modulus
- (– compression strength, $F < 800$ N)
- (– bending strength, small samples)
- (– K_{IC}, unstable fracture)

Pictures (from left to right): Direct Industry, PI Ceramic GmbH, Redhill Precision Balls Inc., Fraunhofer IKTS

K_{IC} dependent on crack length → R-curve

a) R-curve of ideal brittle materials, such as glass

b) Typical R-curve of ceramic materials

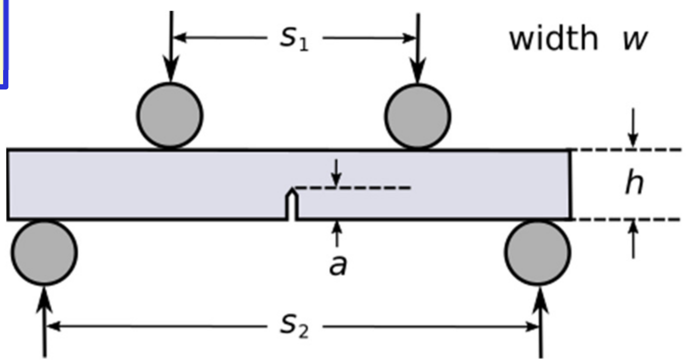


Toughening mechanism:

- crack bridging
- micro cracks near crack tip
- phase transformation
- domain switching
- etc. …

$$K_I = \sigma \cdot \sqrt{a} \cdot \Gamma(\alpha) \quad \text{with } \alpha = {}^{a}/_{h}\,, \quad \text{fracture: } K_I \geq K_{IC}$$

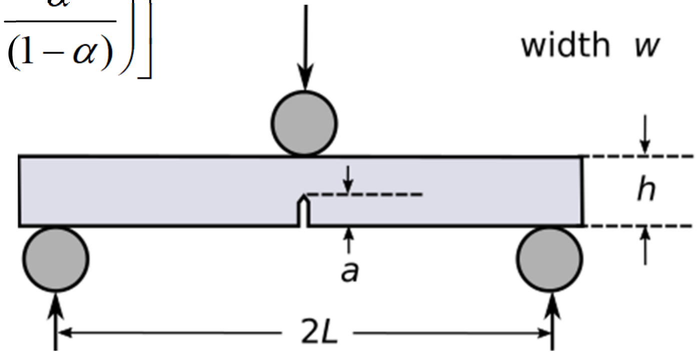
σ : characteristic stress
K_I : stress intensity factor
K_{IC} : fracture toughness



4-point-bending [1]

$$K_I = \frac{3F\,(s_2 - s_1)}{2\,w\,h^2}\sqrt{a}\;\Gamma_M(\alpha)$$

$$\Gamma_M(\alpha) = \frac{1{,}1215\sqrt{\pi}}{(1-\alpha)^{3/2}}\left[\frac{5}{8} - \frac{5}{12}\alpha + \frac{1}{8}\alpha^2 + 5\alpha^2(1-\alpha)^6 + \frac{3}{8}\exp\left(-6{,}1342\frac{\alpha}{(1-\alpha)}\right)\right]$$



3-point-bending [2]

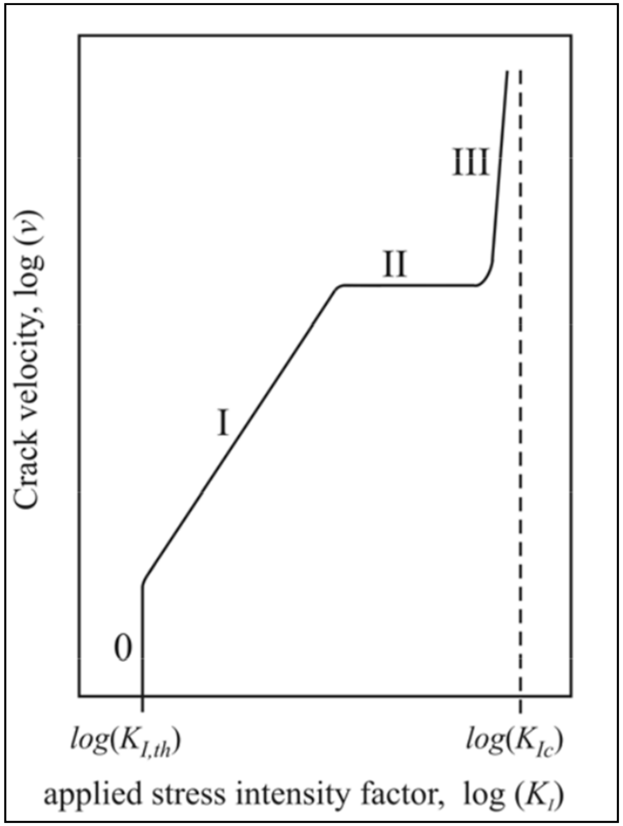
$$K_I = \frac{3\,F\,L}{w\,h^2}\sqrt{a}\;\Gamma_M(\alpha)$$

$$\Gamma_M(\alpha) = \frac{\sqrt{\pi}}{(1-\alpha)^{3/2}}\left[0{,}3738\,\alpha + (1-\alpha)\sum_{\mu,\nu=0}^{4} A_{\mu\nu}\alpha^{\mu}(h/2L)^{\nu}\right]$$

Advantage: Equations are valid for $0 \leq \alpha \leq 1$, i.e., for any crack length.

[1] D. Munz, T. Fett: Ceramics – Mechanical Properties …, Springer (2001)
[2] T. Fett, D. Munz: Stress Intensity Factors… **Comp. Mech. Publ.** (1997)

Subcritical crack growth with: $K_I < K_{Ic}$



[3, 1]

- **Section I:**

  $$v = A(K_I)^n = A^* \left(\frac{K_I}{K_{Ic}}\right)^n$$

  n = Crack growth exponent

- **Section II:** A plateau (possibly)

Dependent on temperatur and environment!

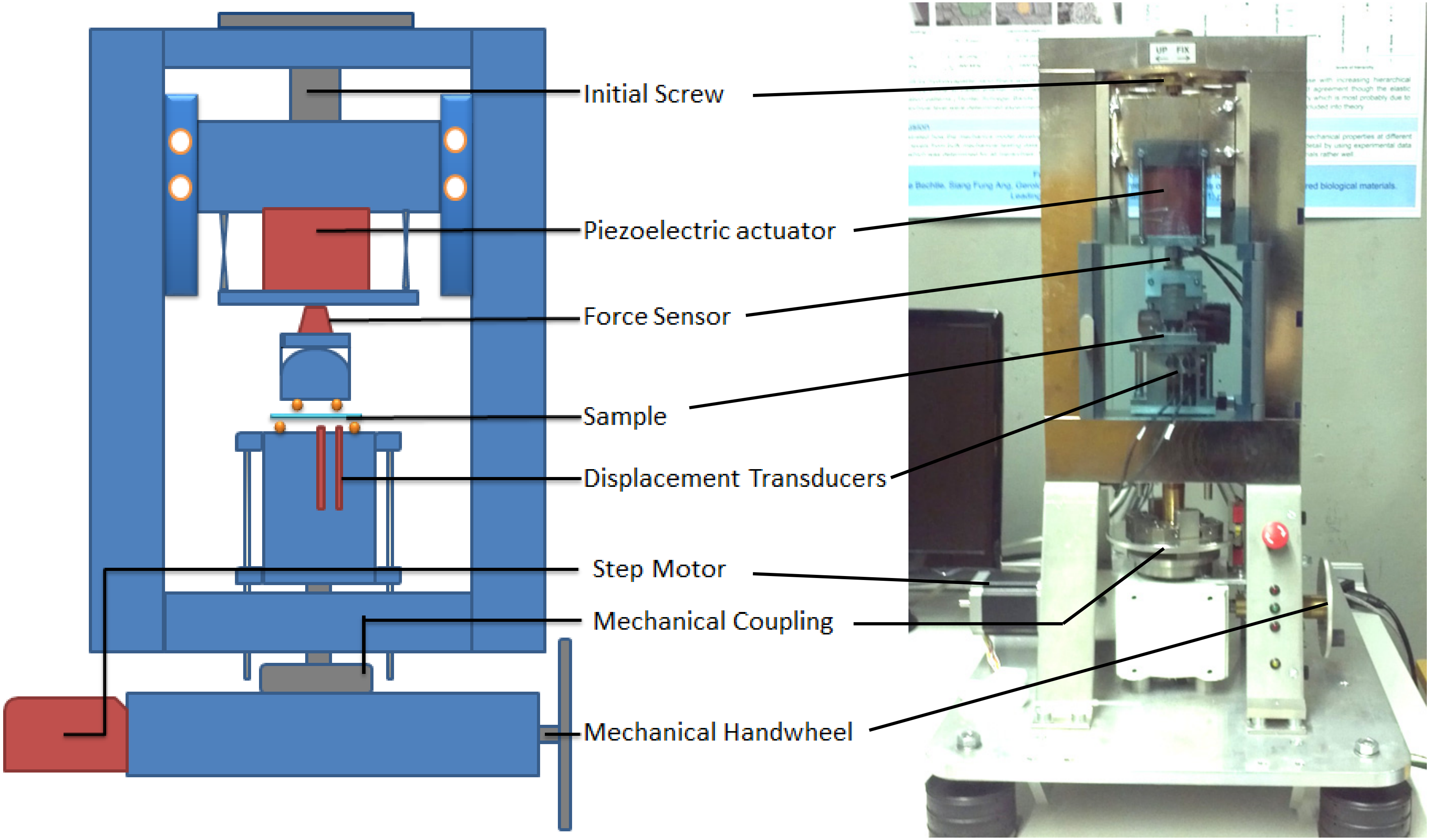
- **Section III**: Reaching K_{Ic}

[3] B. R. Lawn: Fracture of Brittle Solids. 2. Edition, Cambridge (1993)

What is new?

1. Automatic detection of crack growth
2. Crack length determination from compliance of sample
3. Optimization of hardware and software
4. Application: Rapid measurement of v-K-curves

3a. Experimental set-up, principle of measurement



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Machine compliance at 50 N: 0.065 μm/N with 4 rollers
0.055 μm/N without rollers

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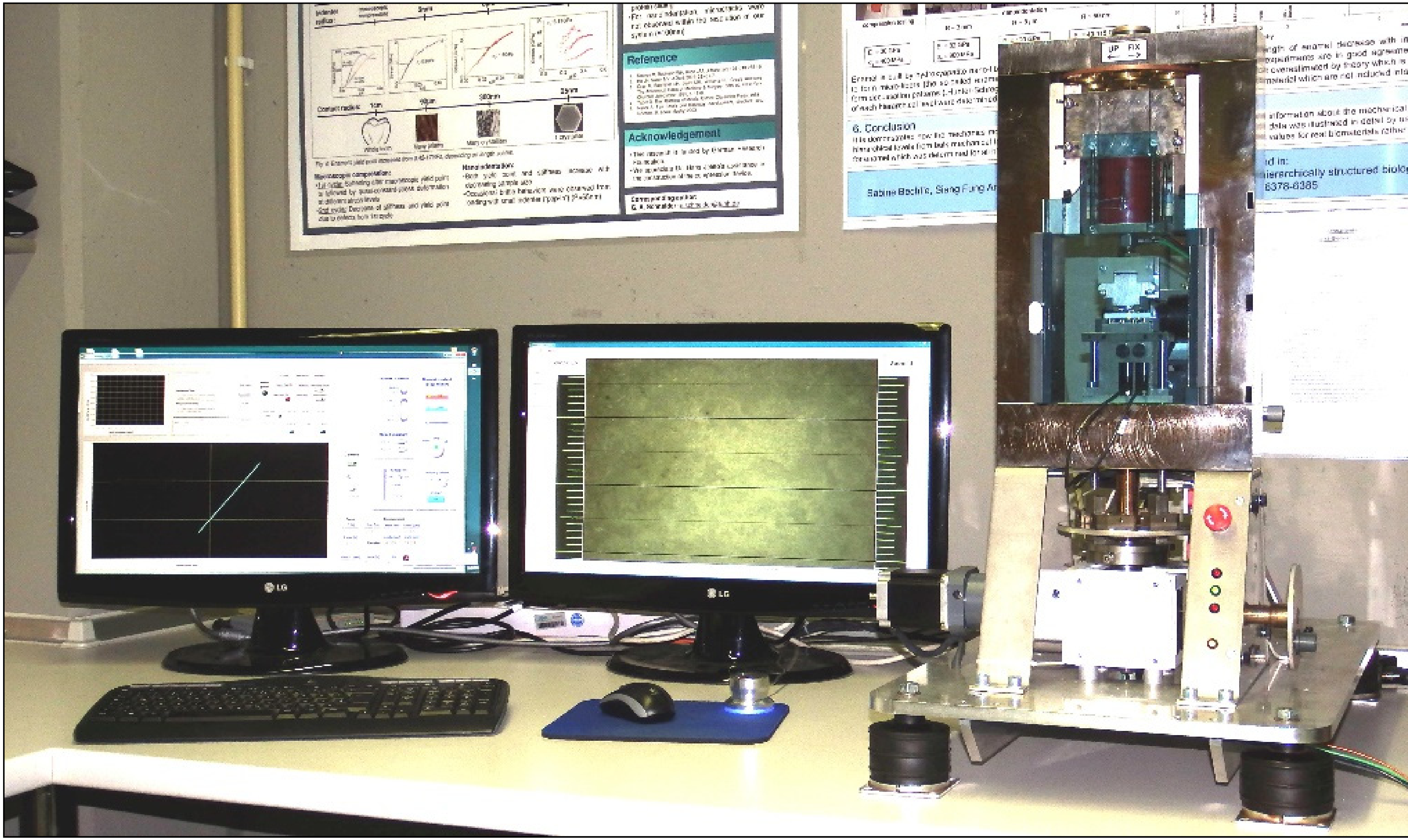
3a. Experimental set-up, principle of measurement



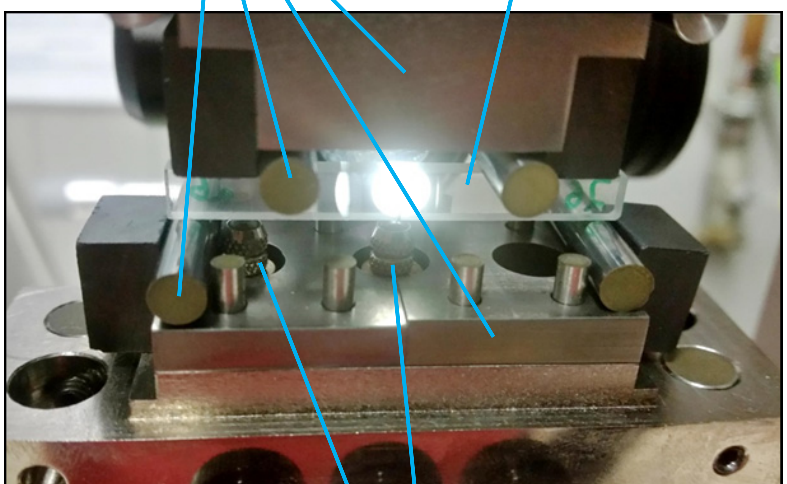
work station

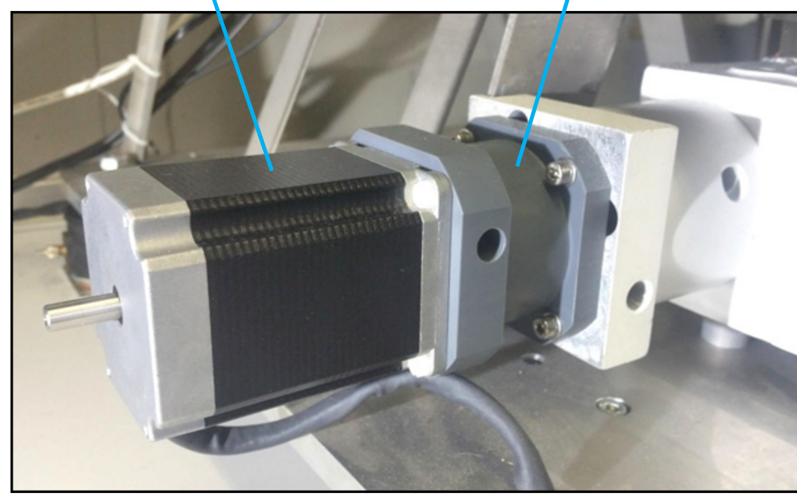
3a. Experimental set-up, principle of measurement

Optimization of hardware (two examples)

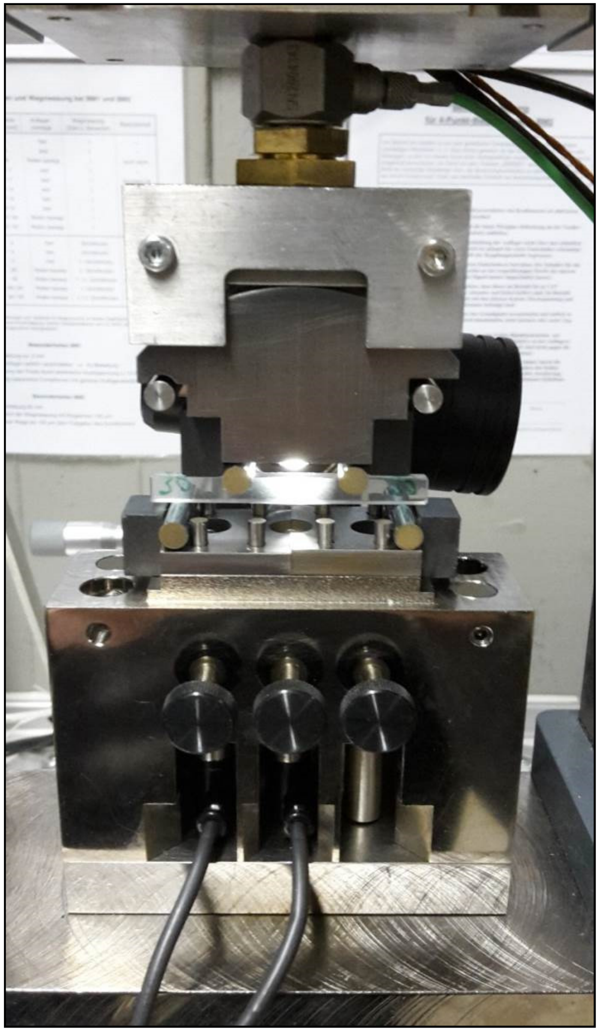
Load cell made of hard metal,
support distances 40 and 20 mm
(increases stiffness of the machine)

Electric ground of step motor and of
device are insulated against each other.
Also the axis has an insulating adapter.
(reduces noise of the displacement signal)

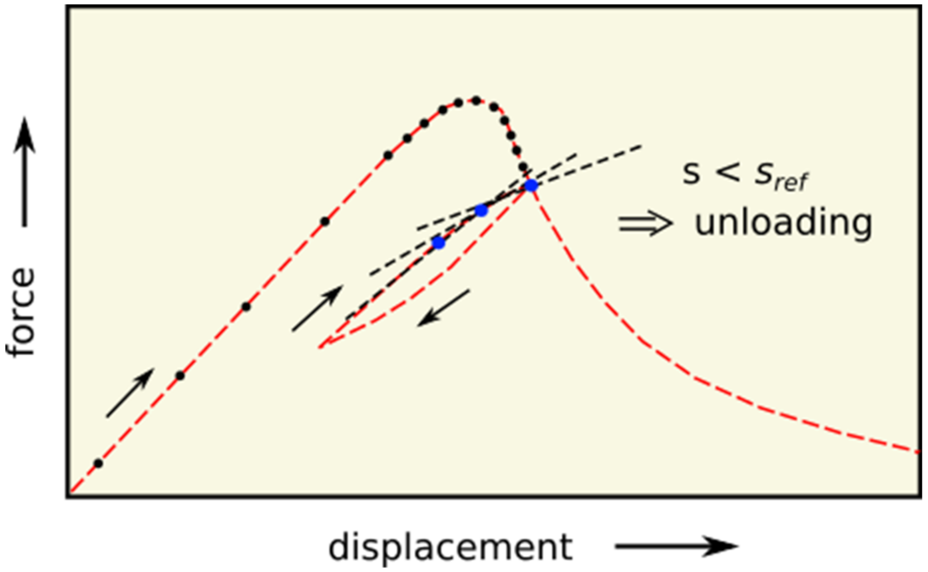




Differential measurement of the displacement with two position encoders provides the pure bending compliance of the specimen without the machine compliance. Together with the change of the force, this allows for the crack length calulation.

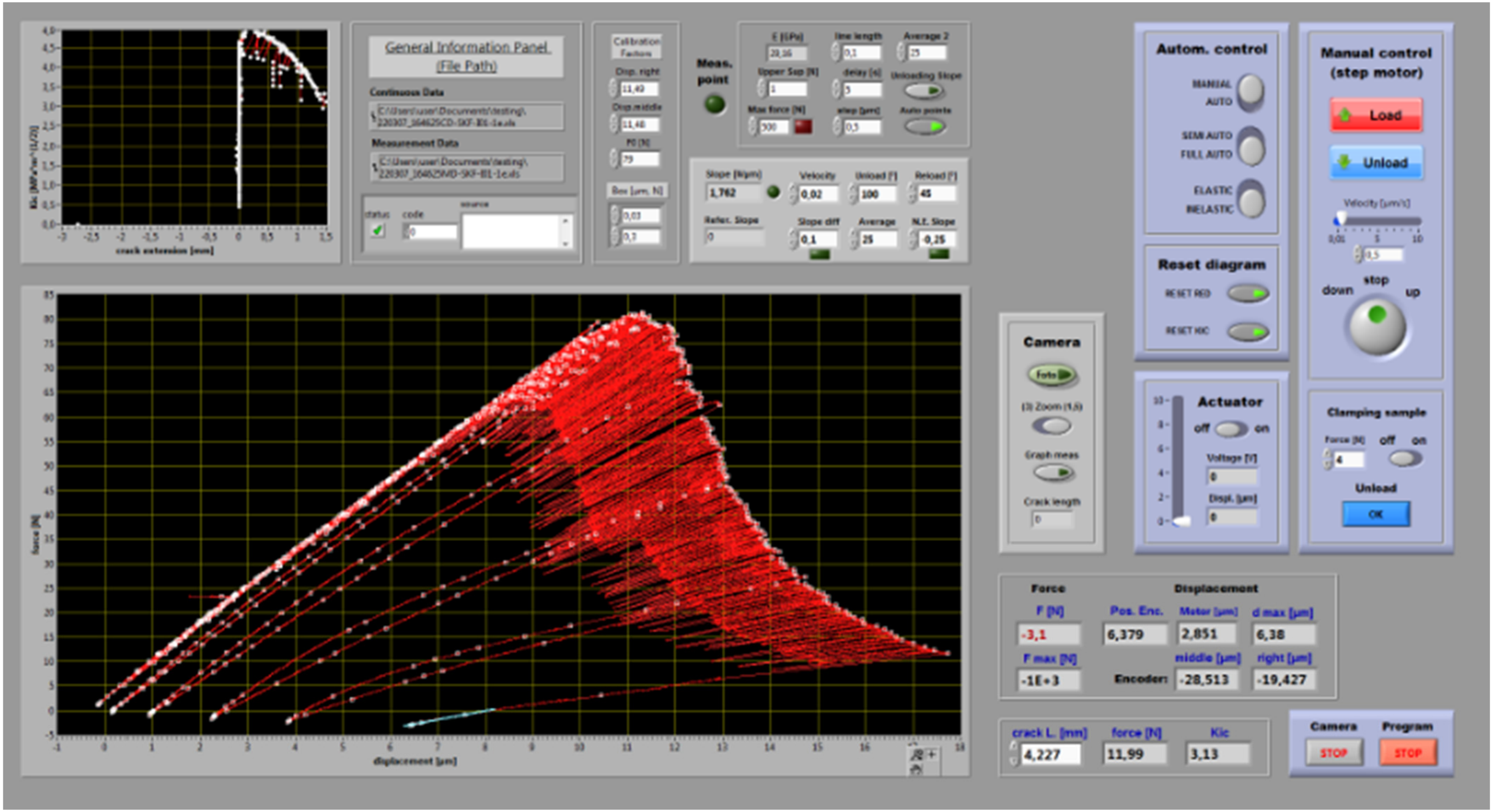


Automatic (program written in LabVIEW) [4]



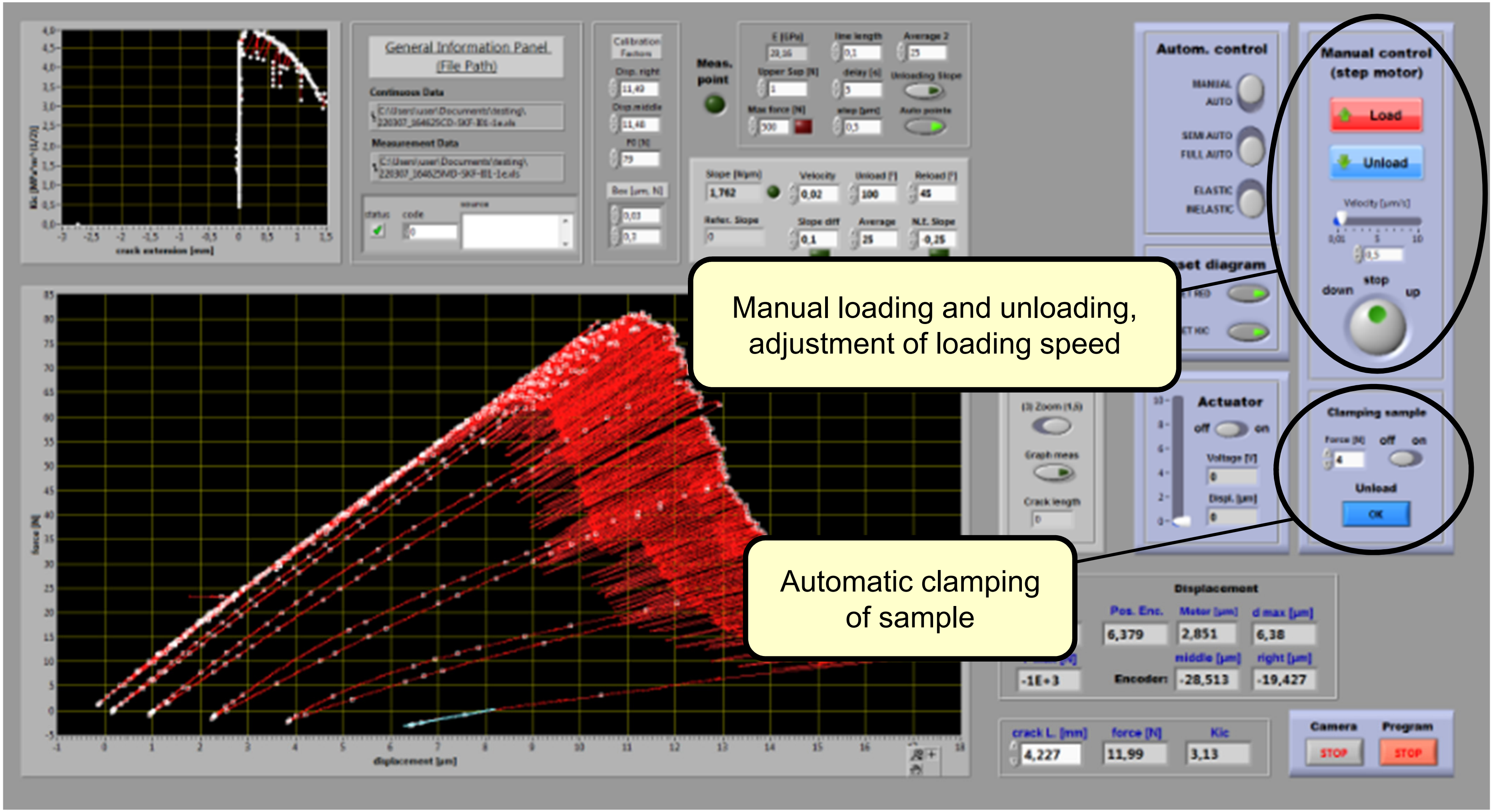
- Permanent measurement of the slope *s* in the force-displacement-diagram
- Crack growth is automatically detected according to the change of the slope.
- The unloading point can be adjusted.

[4] H. Jelitto et al.: Automated Control of Stable Crack Growth for R-Curve Measurements in Brittle Materials. **Exp. Mech. 53** (2013) 163



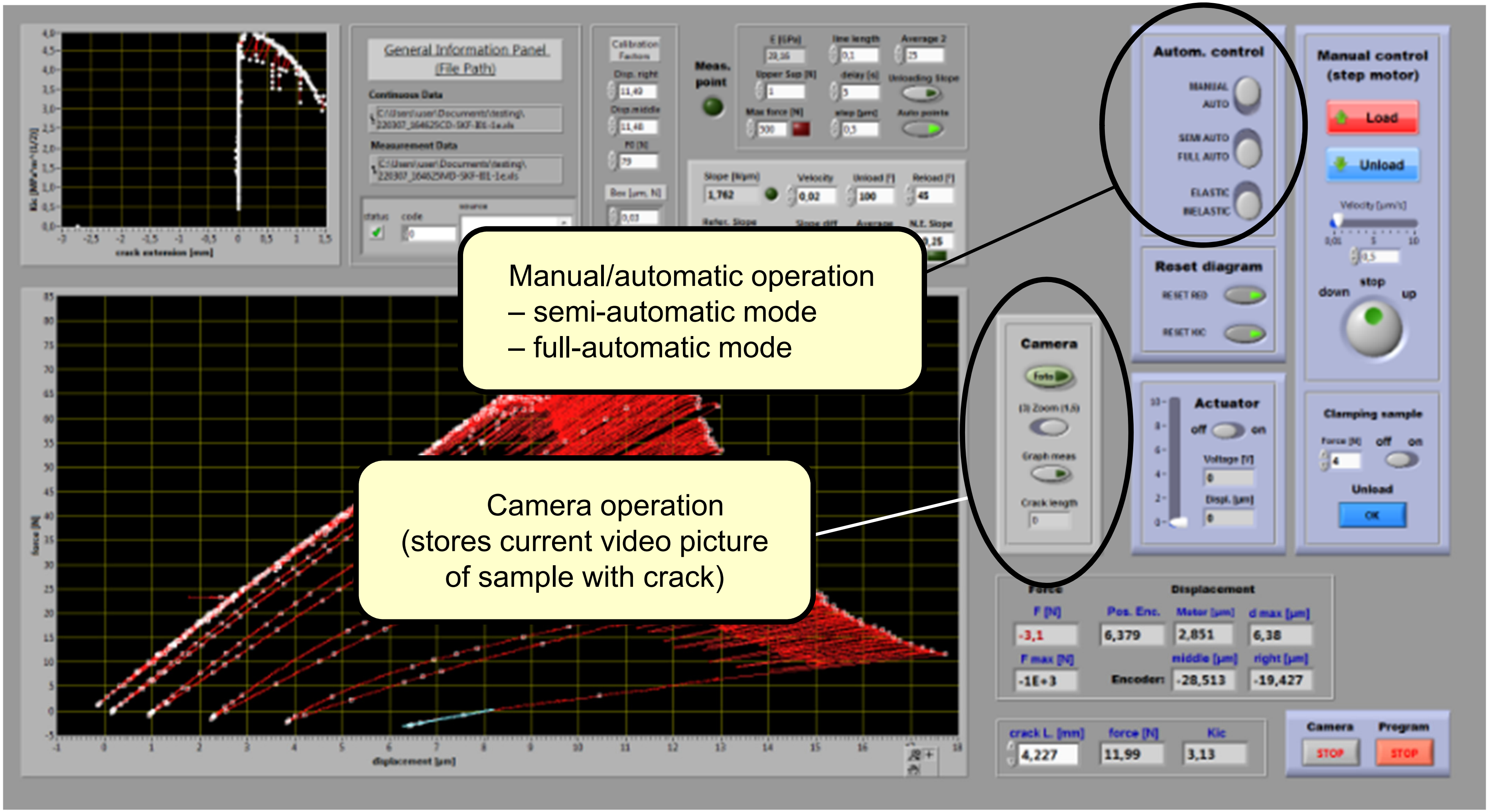
control panel (after a measurement)

3b. Experimental set-up, operation of machine



control panel (after a measurement)

3b. Experimental set-up, operation of machine



control panel (after a measurement)

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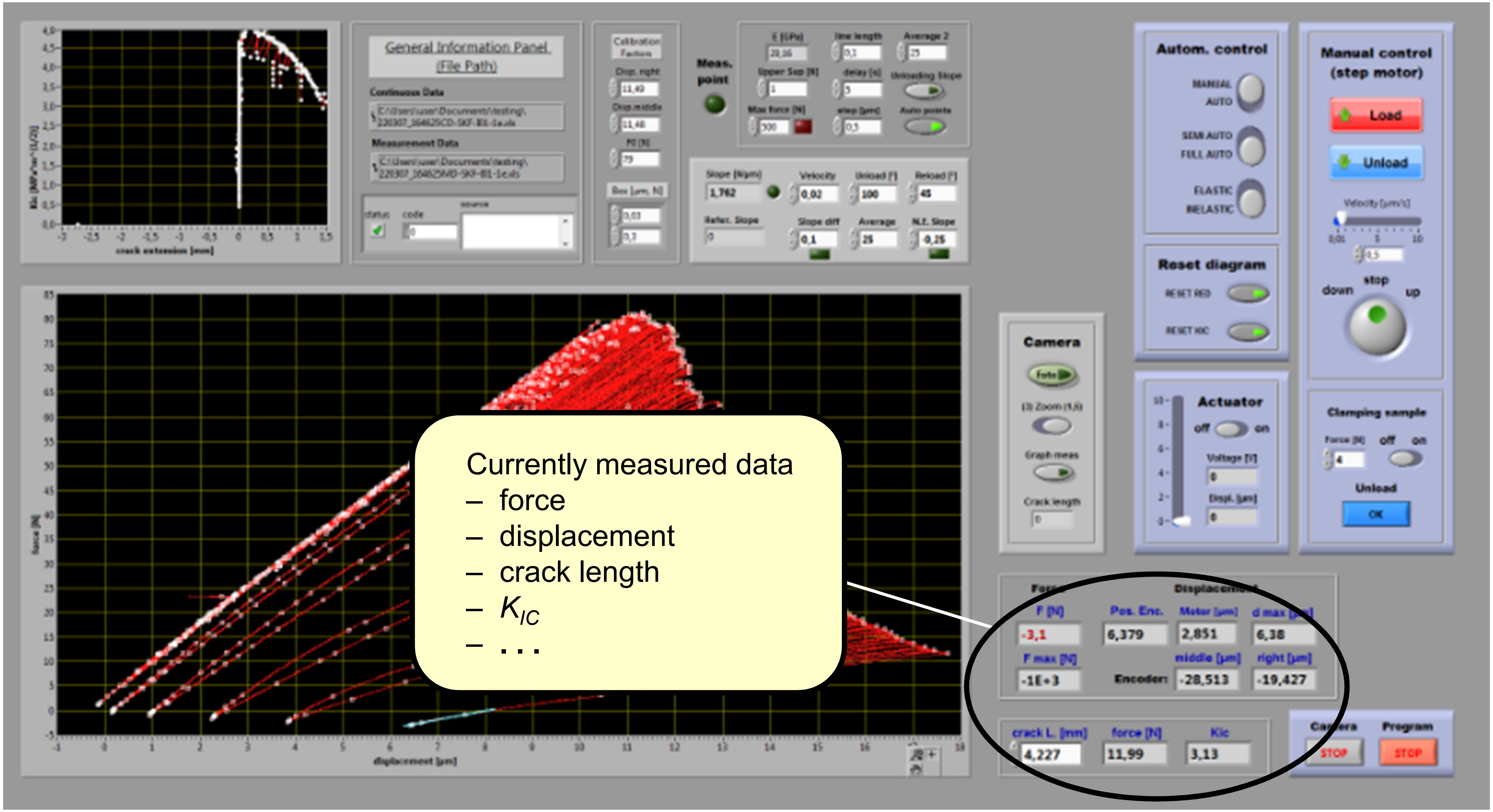
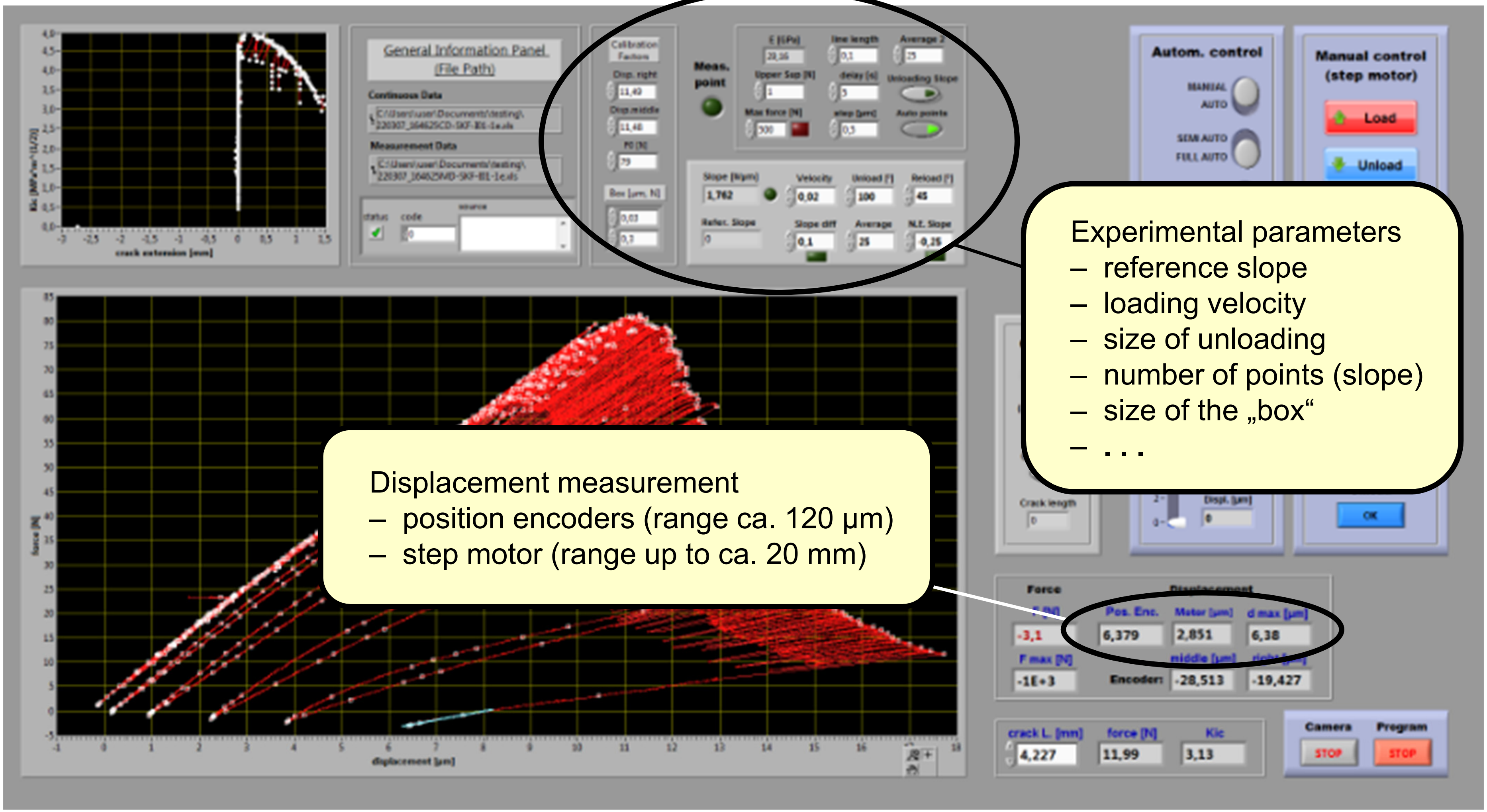
3b. Experimental set-up, operation of machine



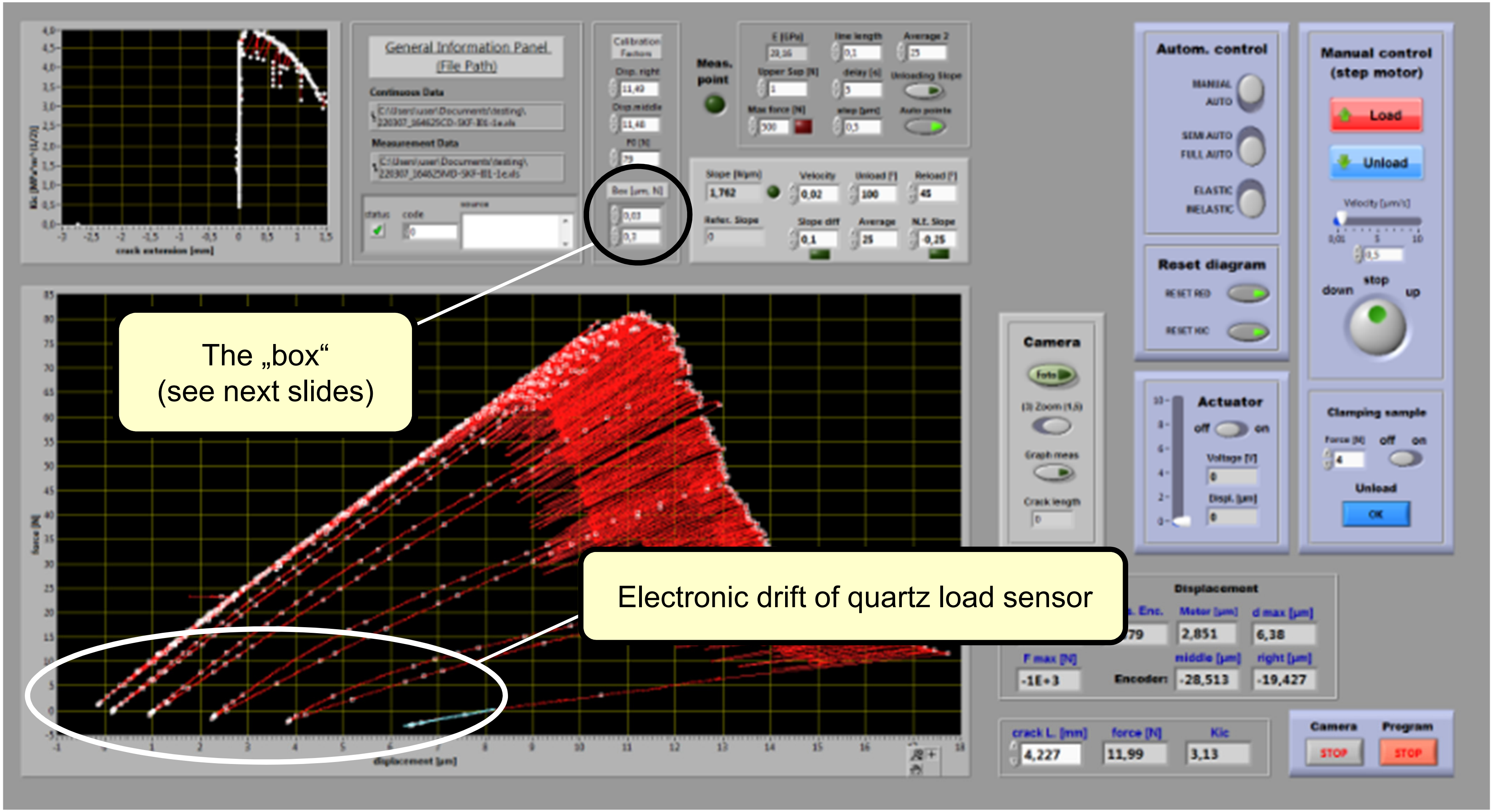
Currently measured data
- force
- displacement
- crack length
- K_{IC}
- . . .

control panel (after a measurement)

3b. Experimental set-up, operation of machine



control panel (after a measurement)



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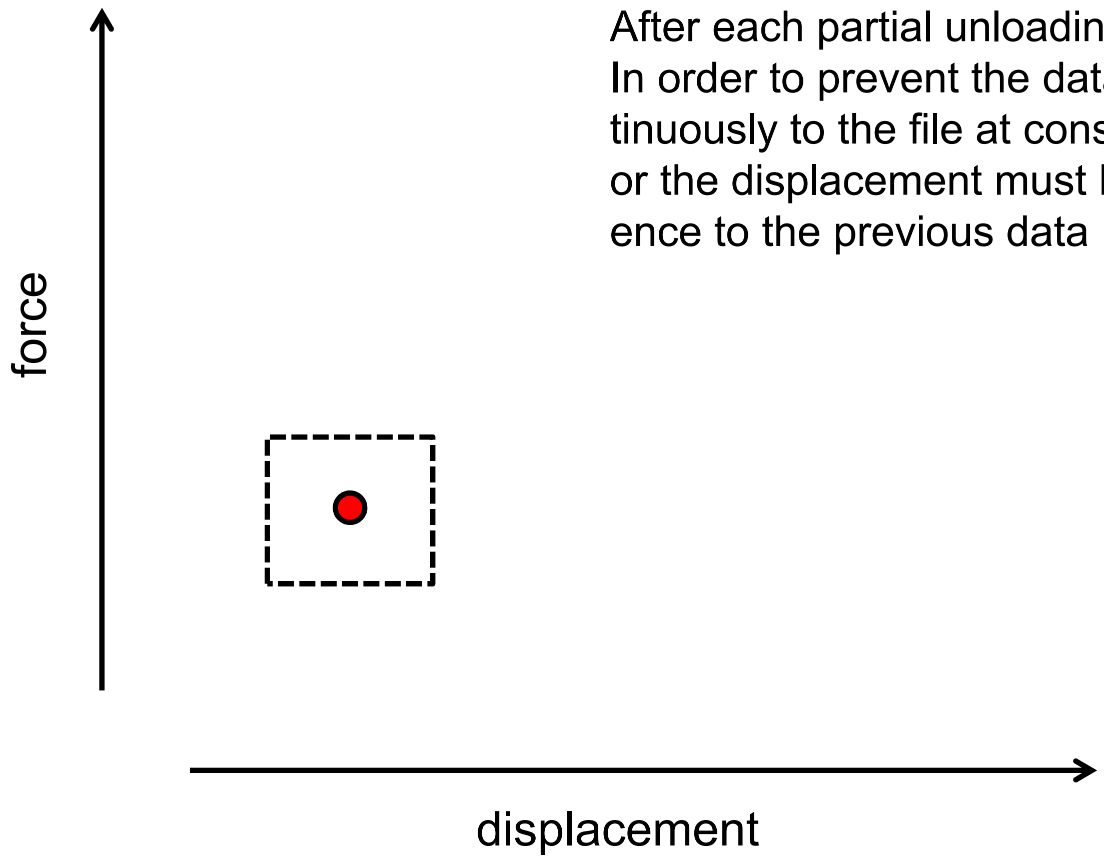
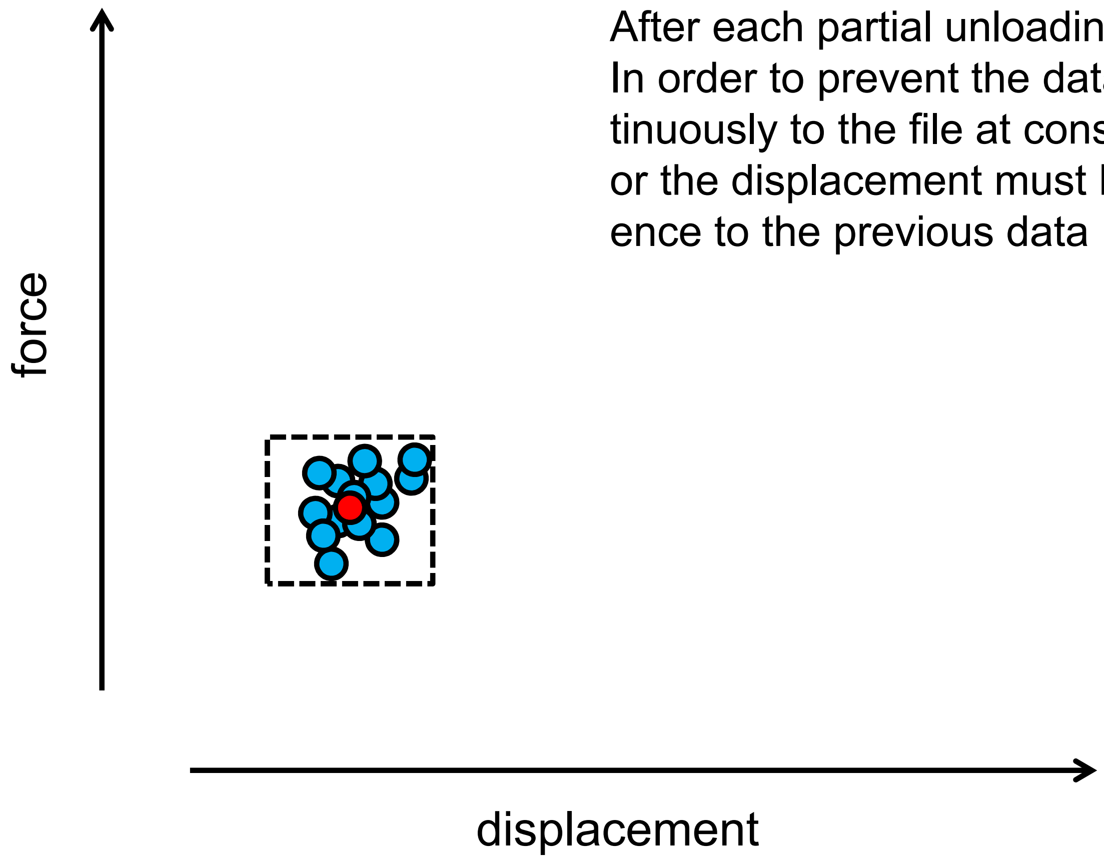


Operation of the box

After each partial unloading, the force is constant. In order to prevent the data from being written continuously to the file at constant force, either the force or the displacement must have a predefined difference to the previous data point.



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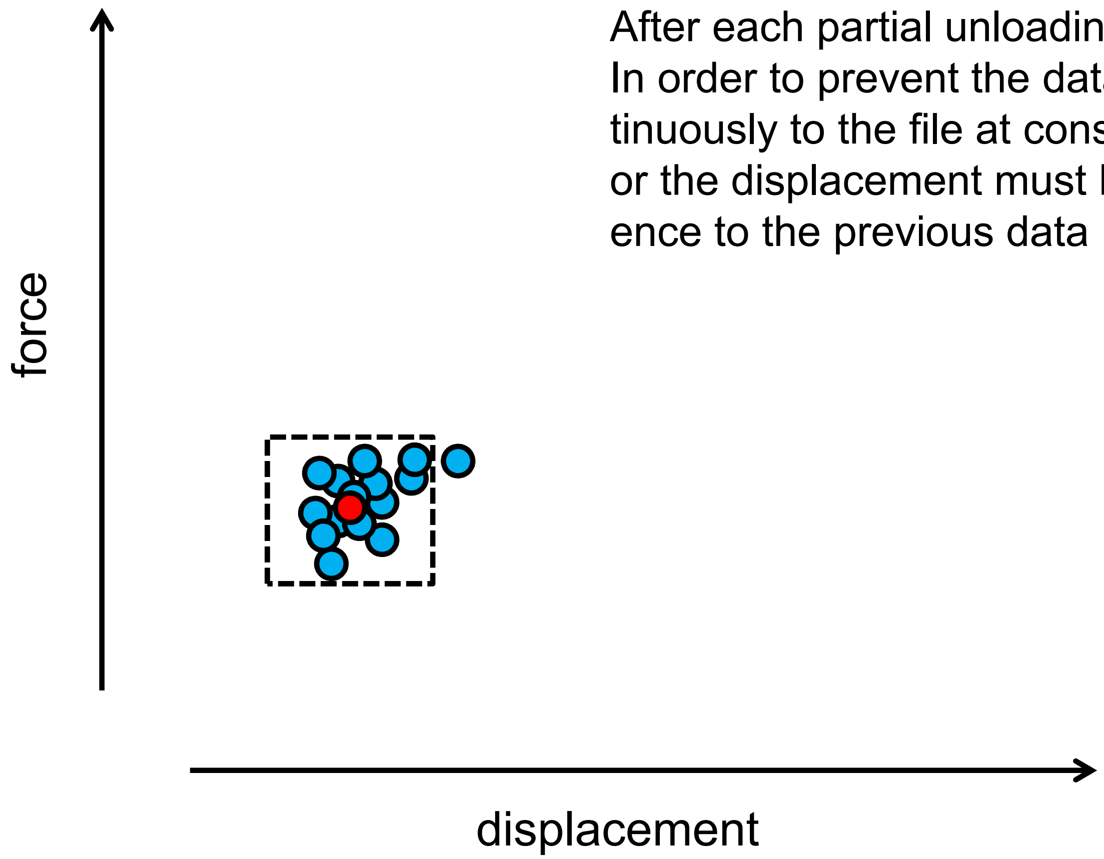

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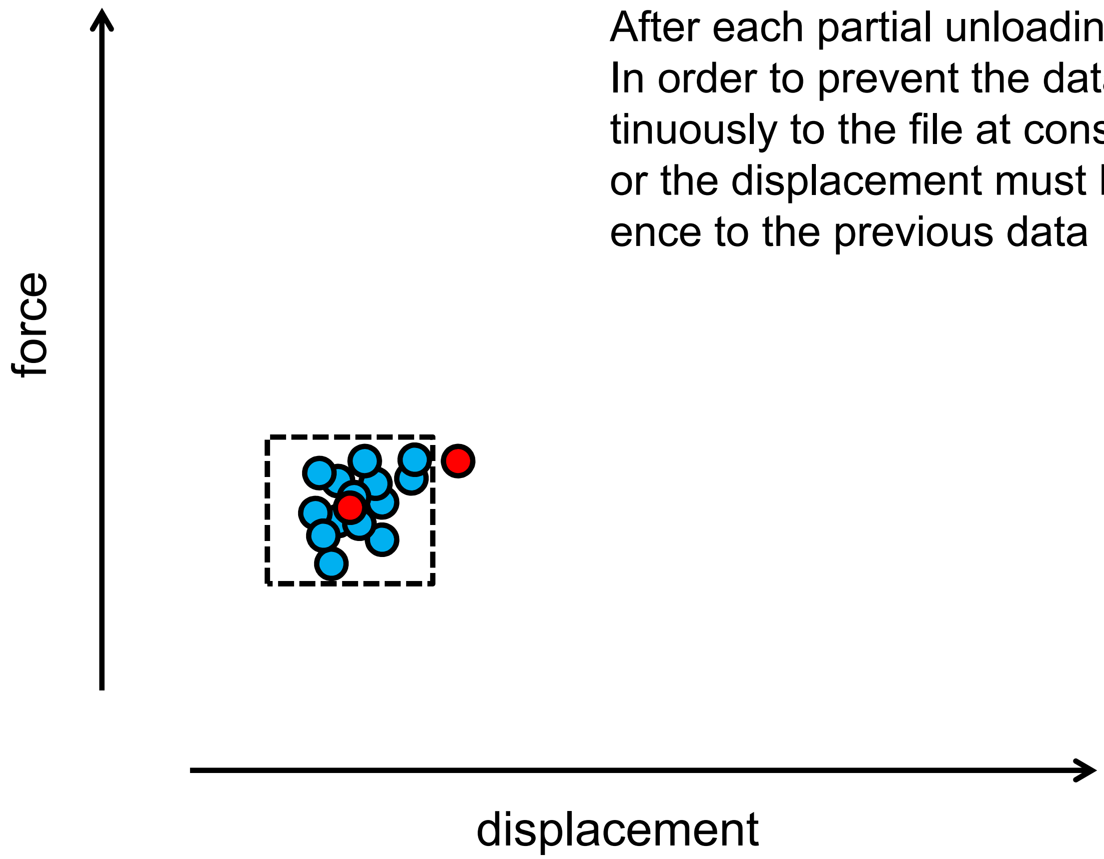

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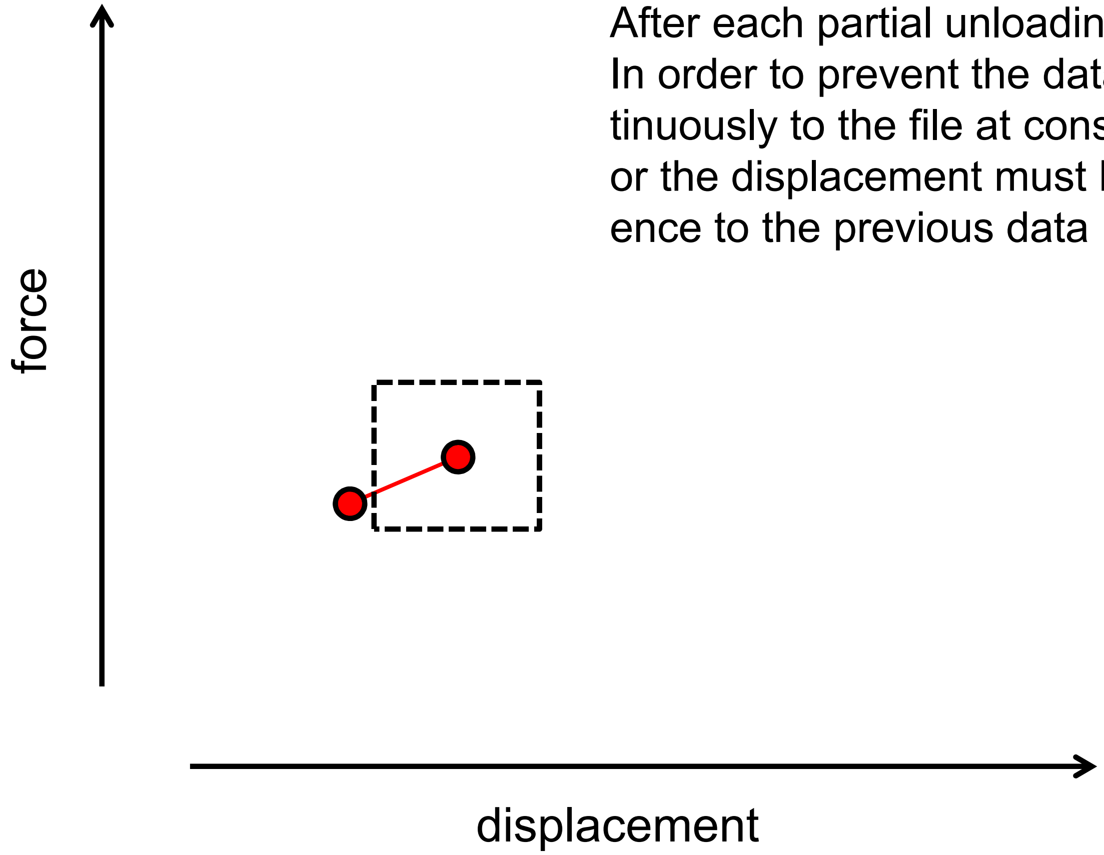

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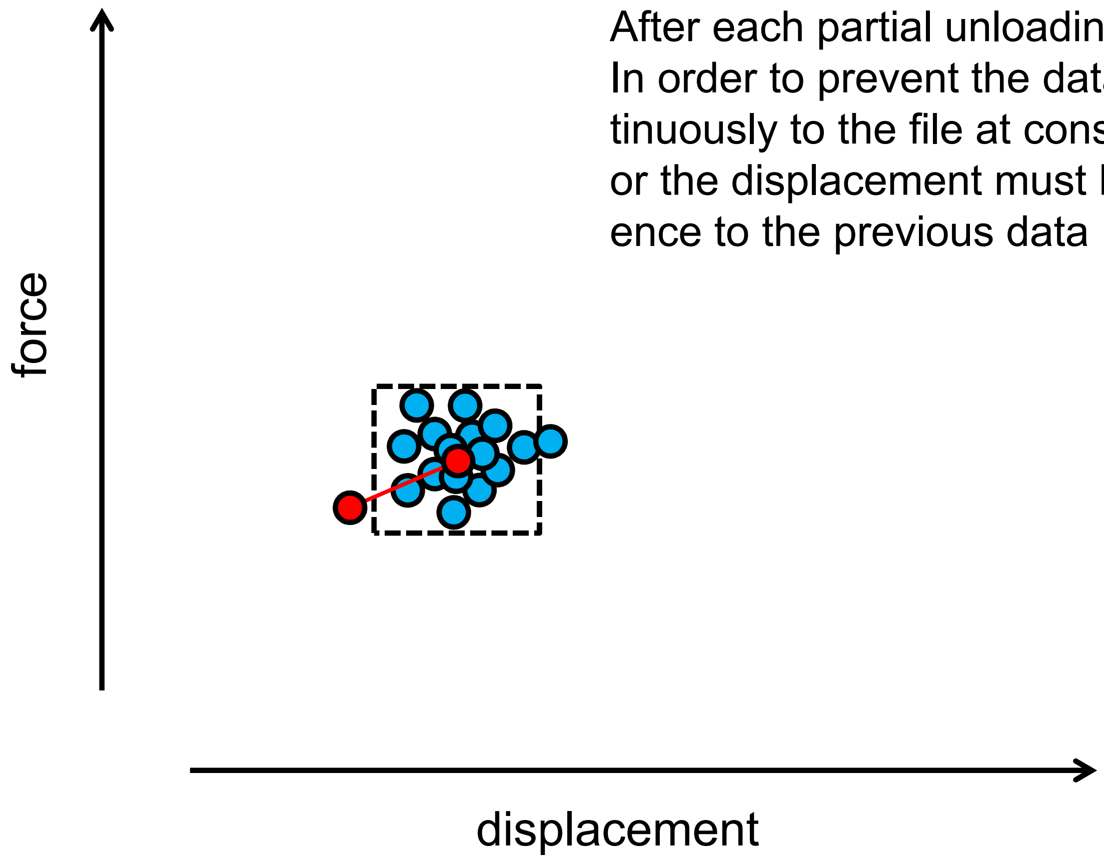

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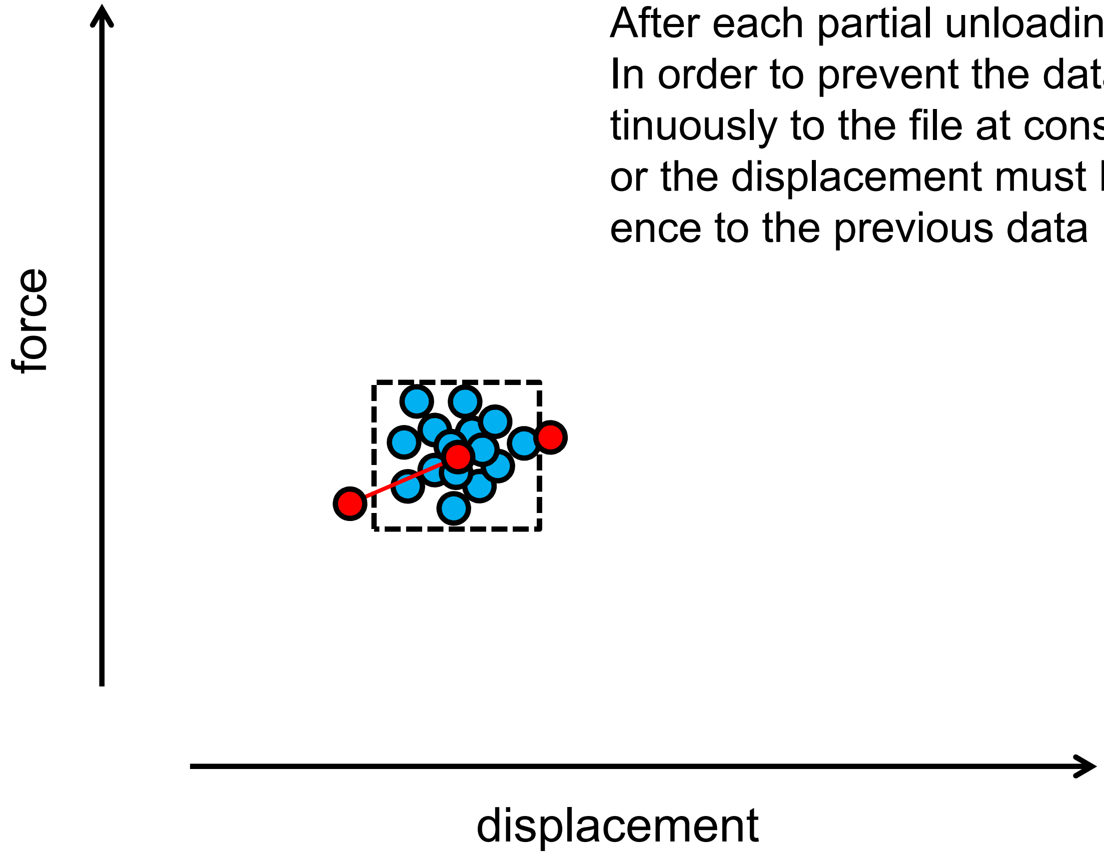

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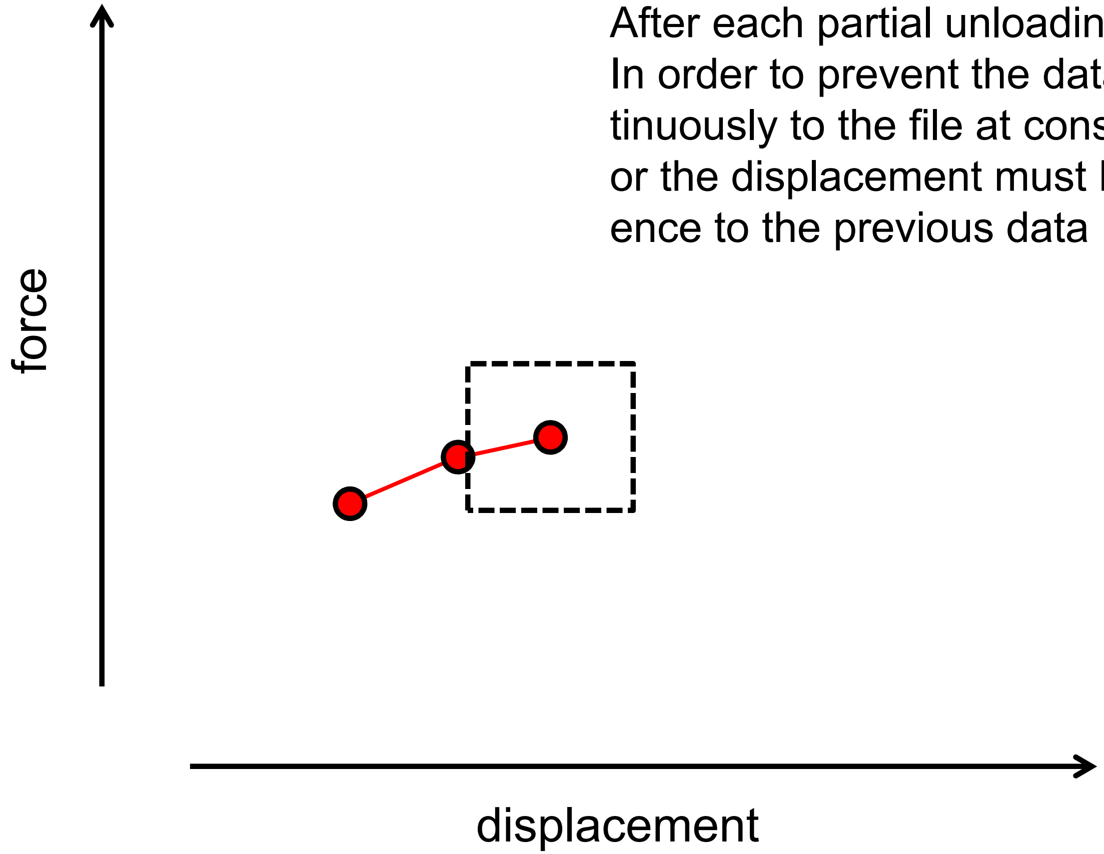

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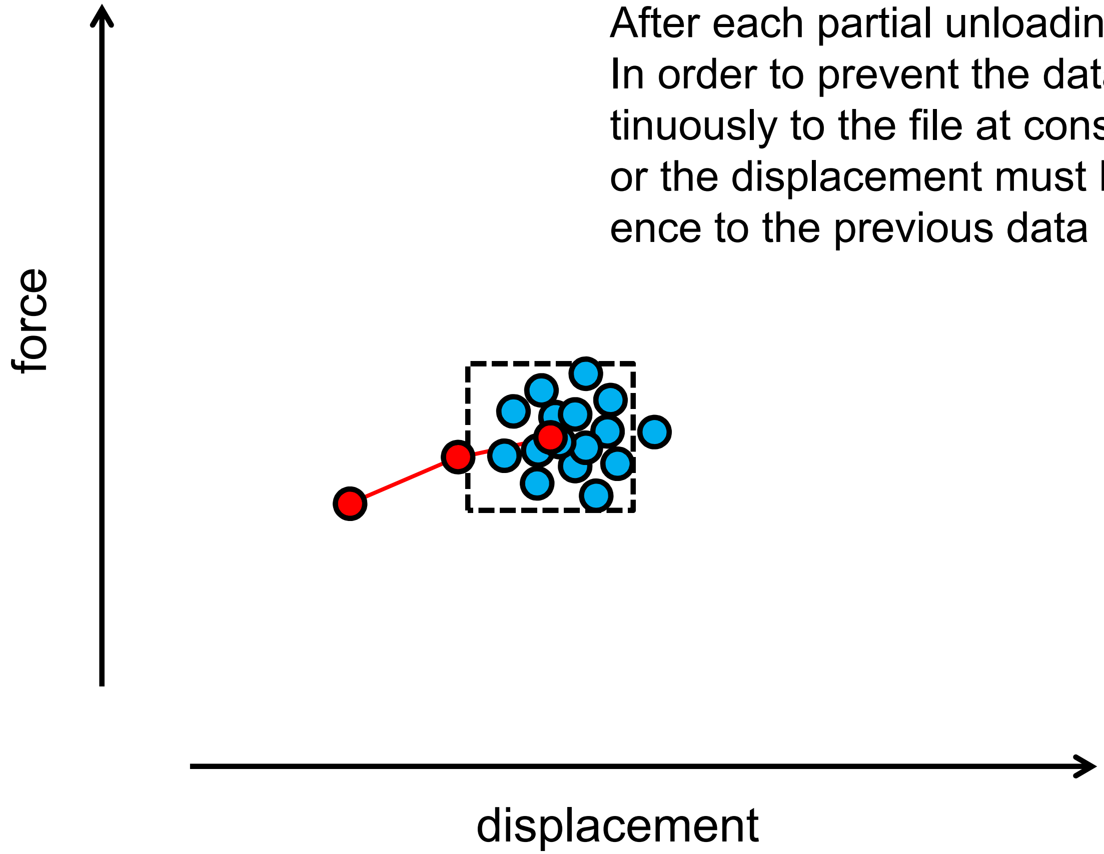

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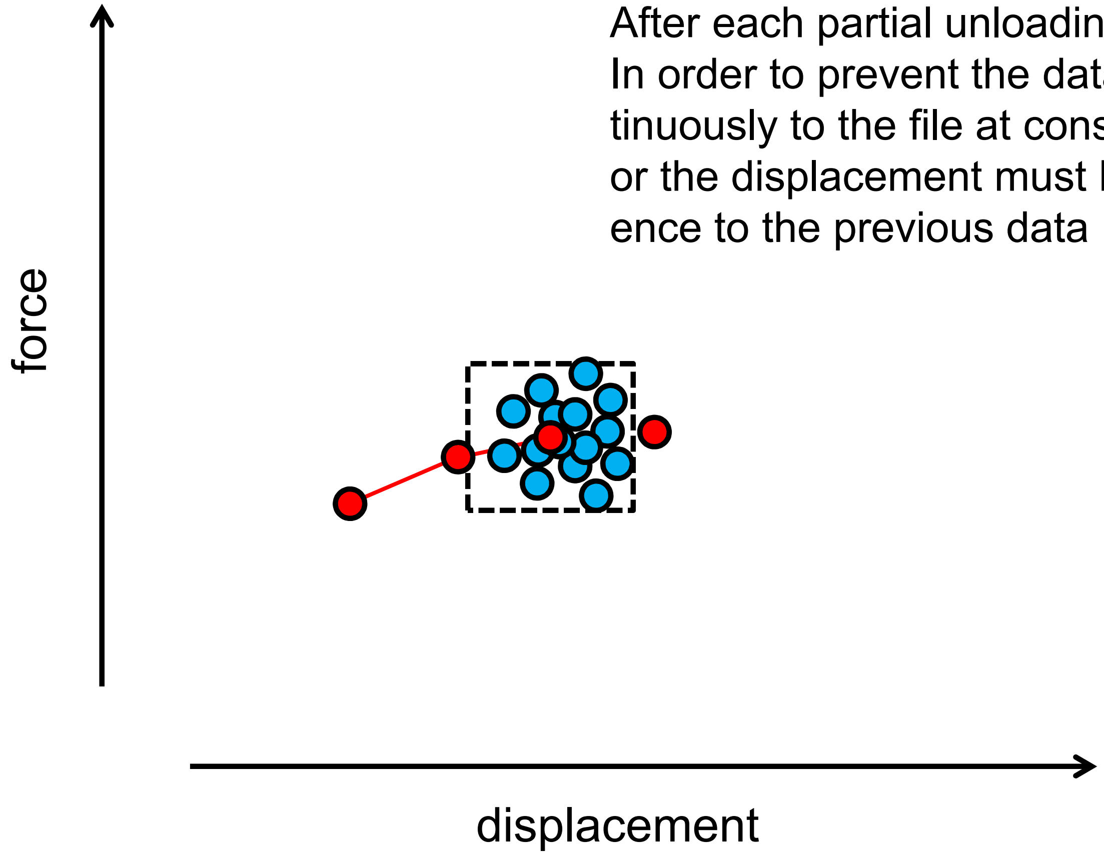

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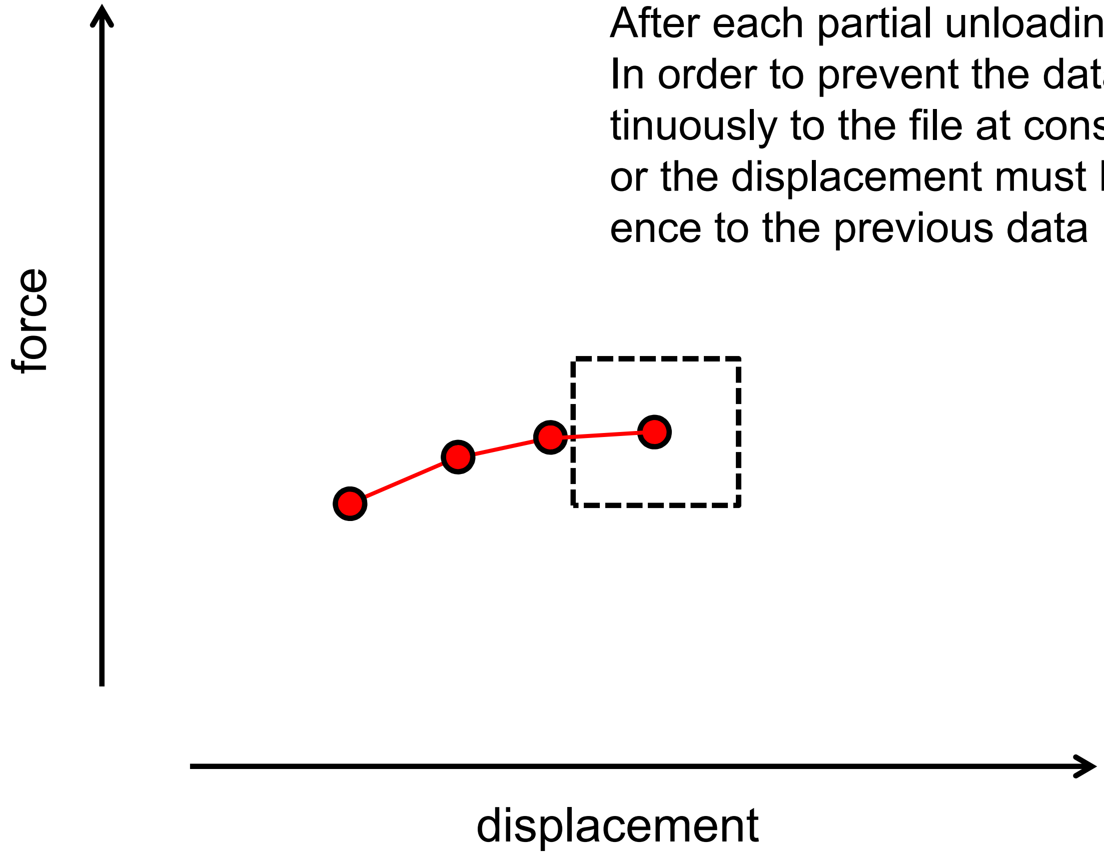

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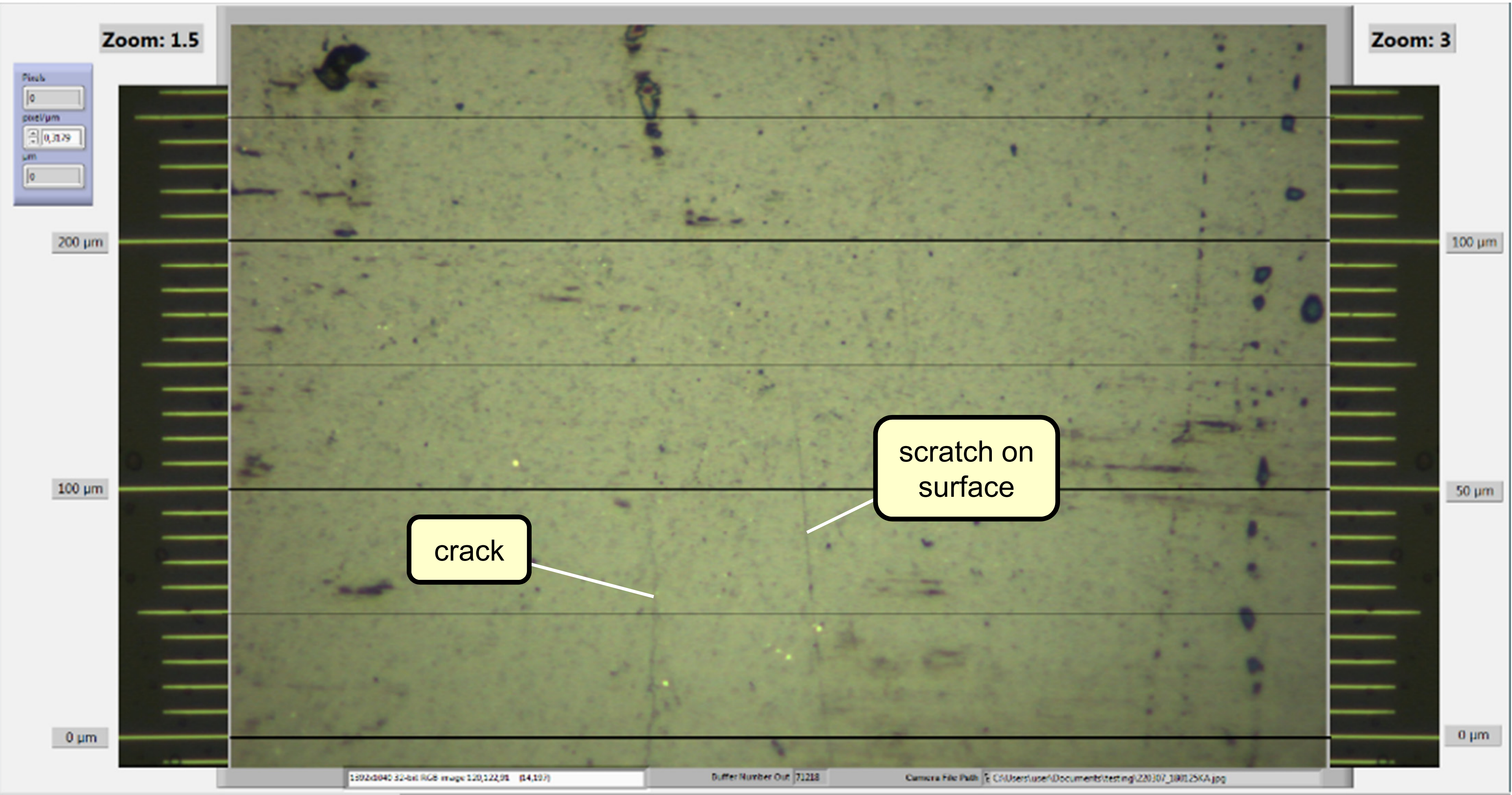
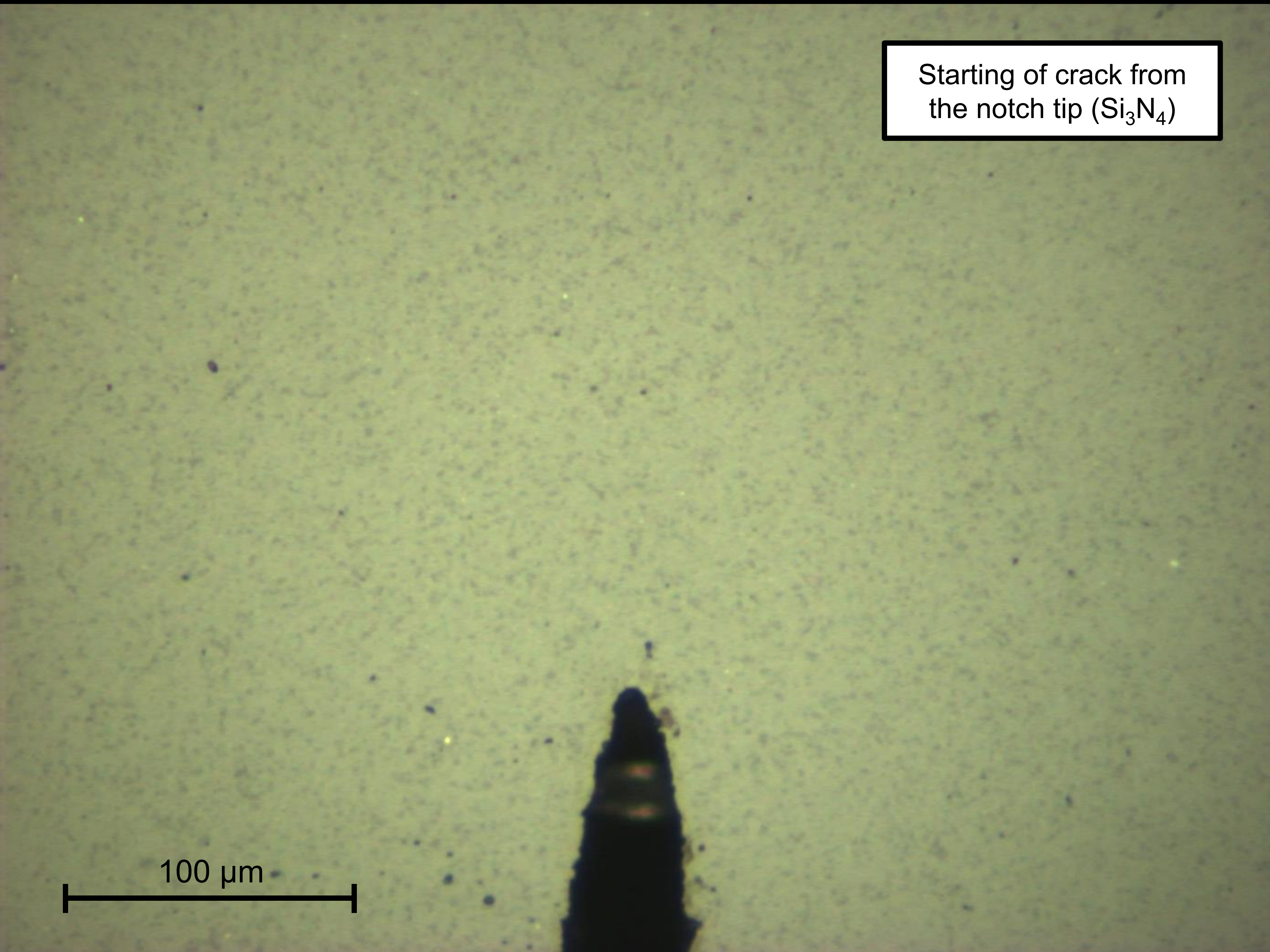
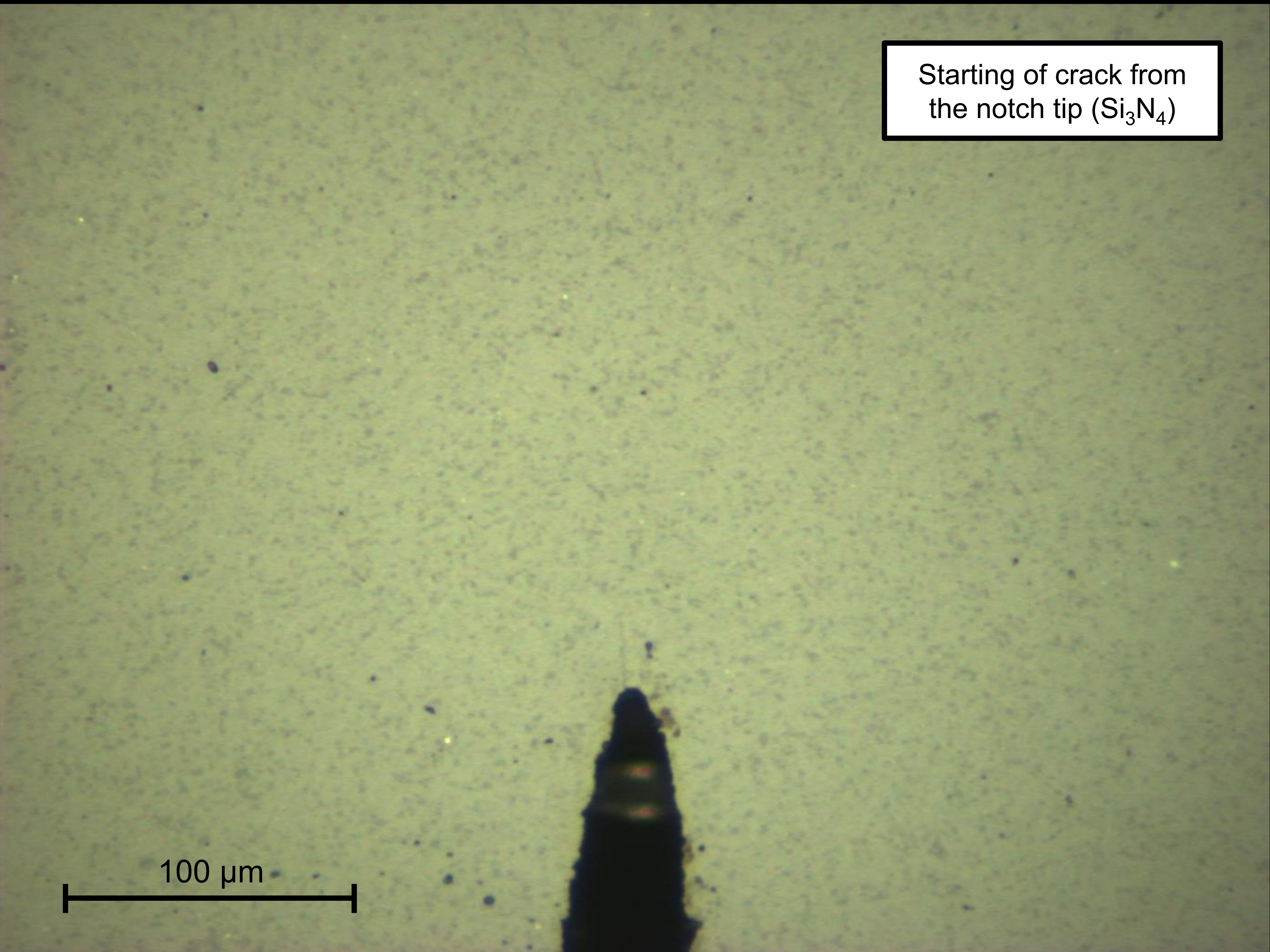


live picture of crack


Starting of crack from the notch tip (Si_3N_4)
100 µm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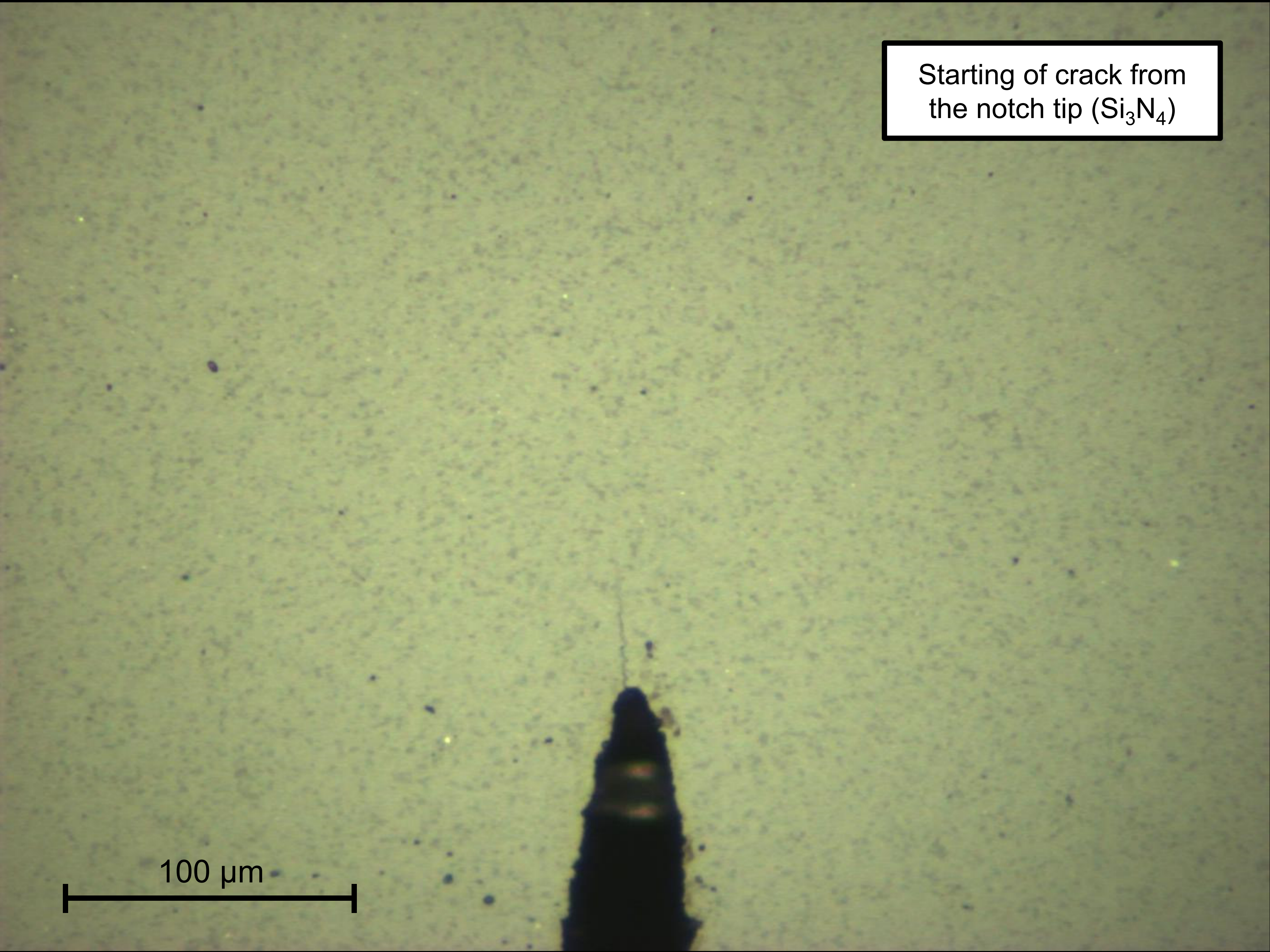
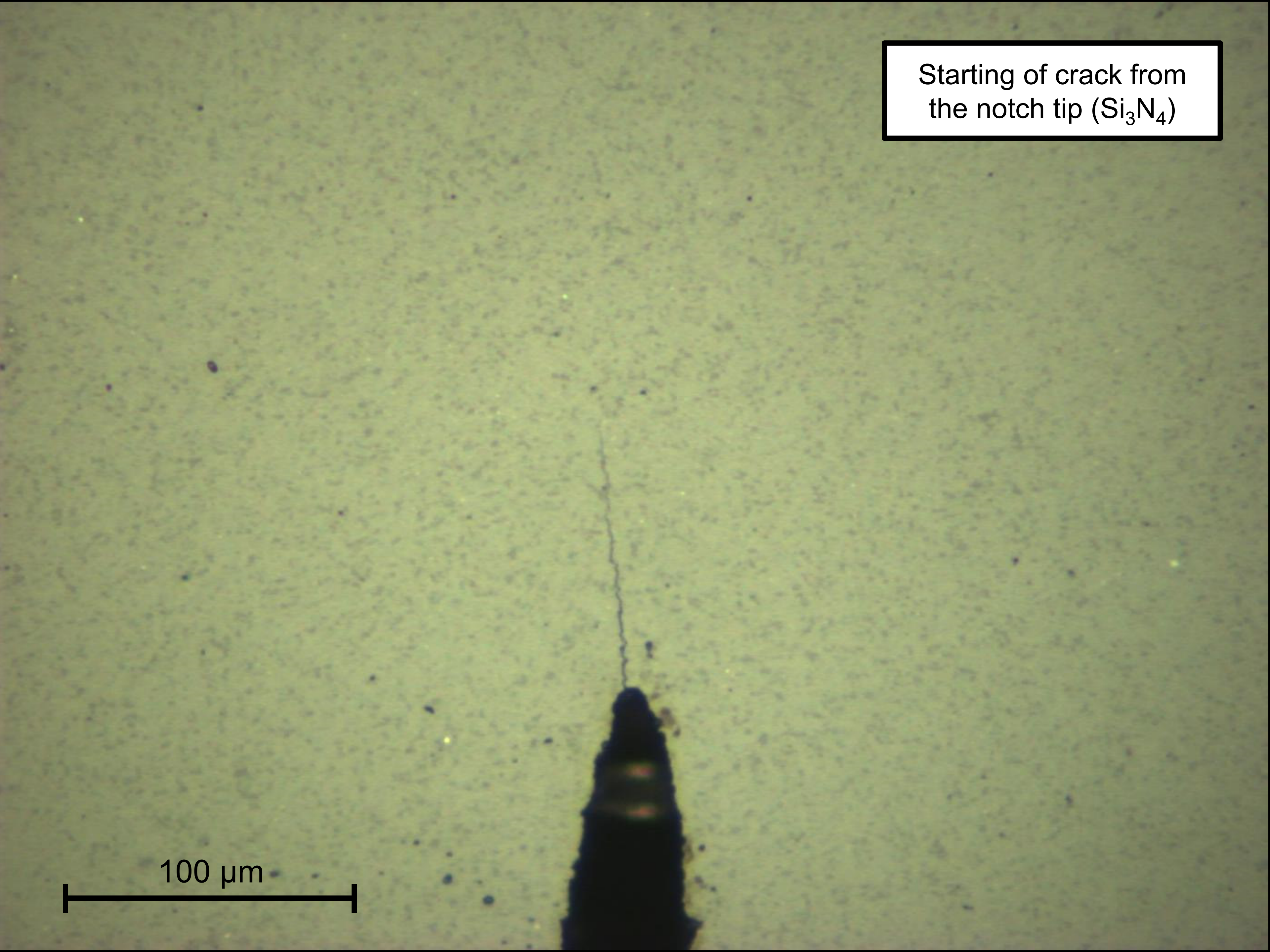
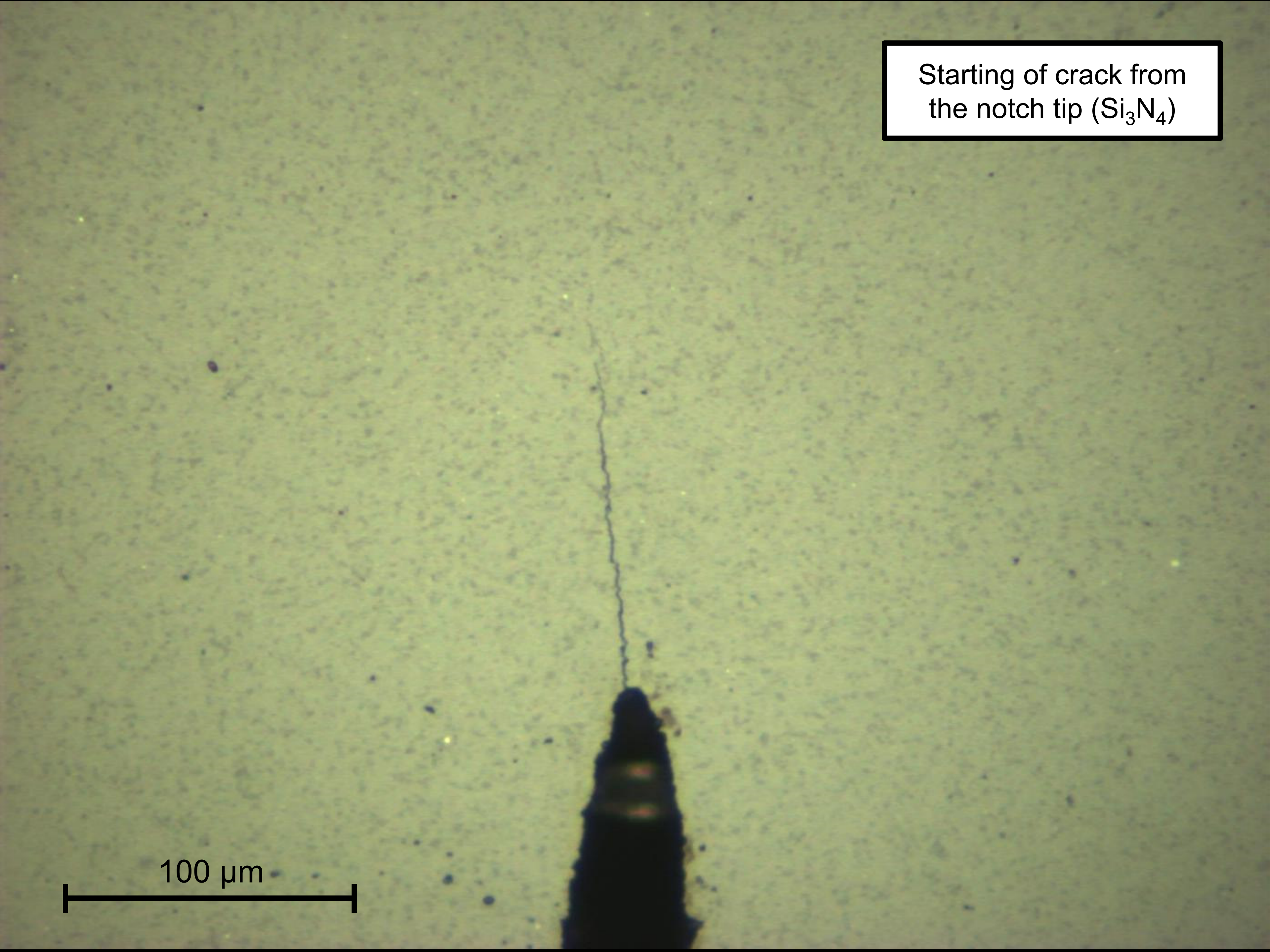
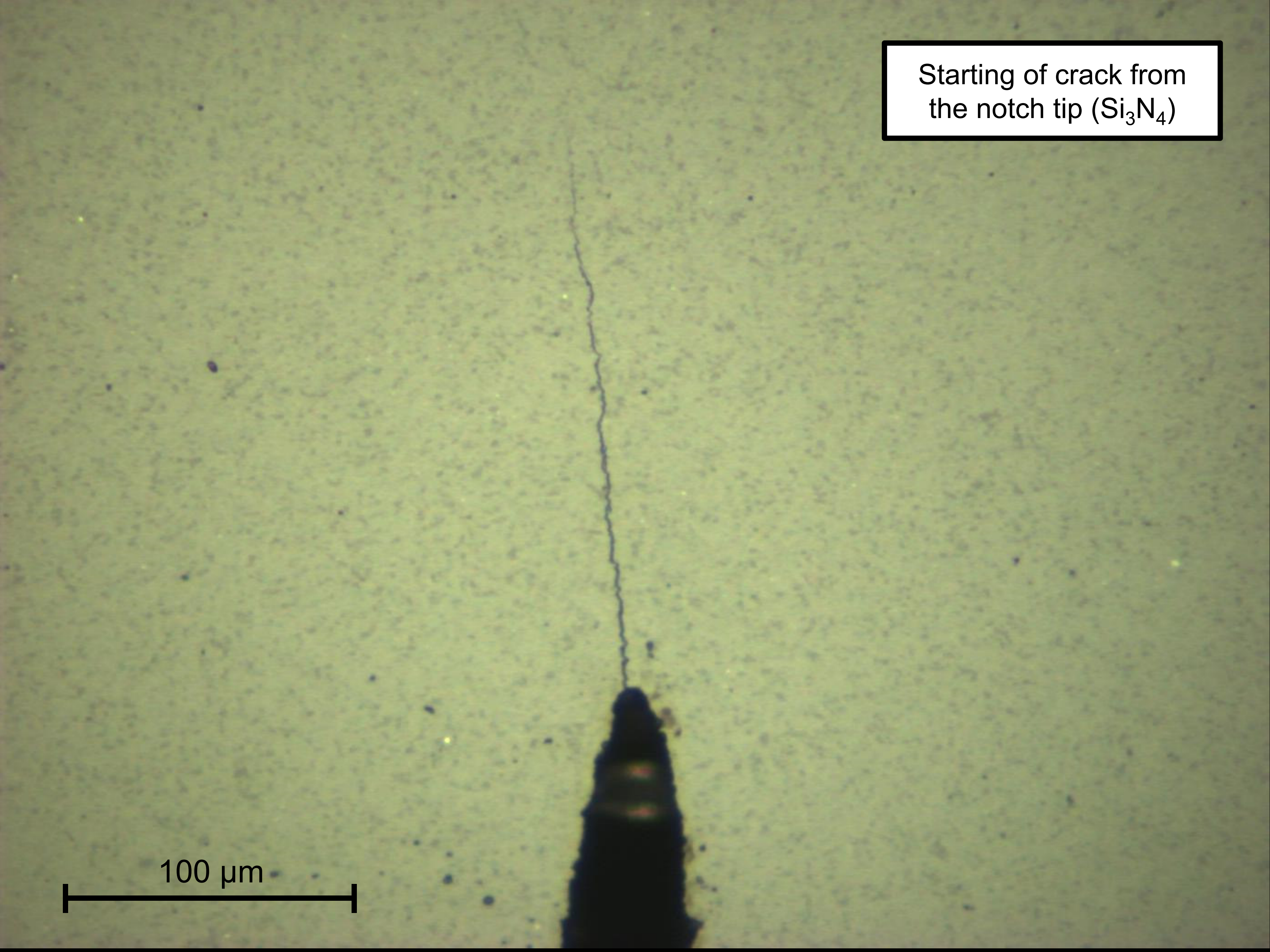
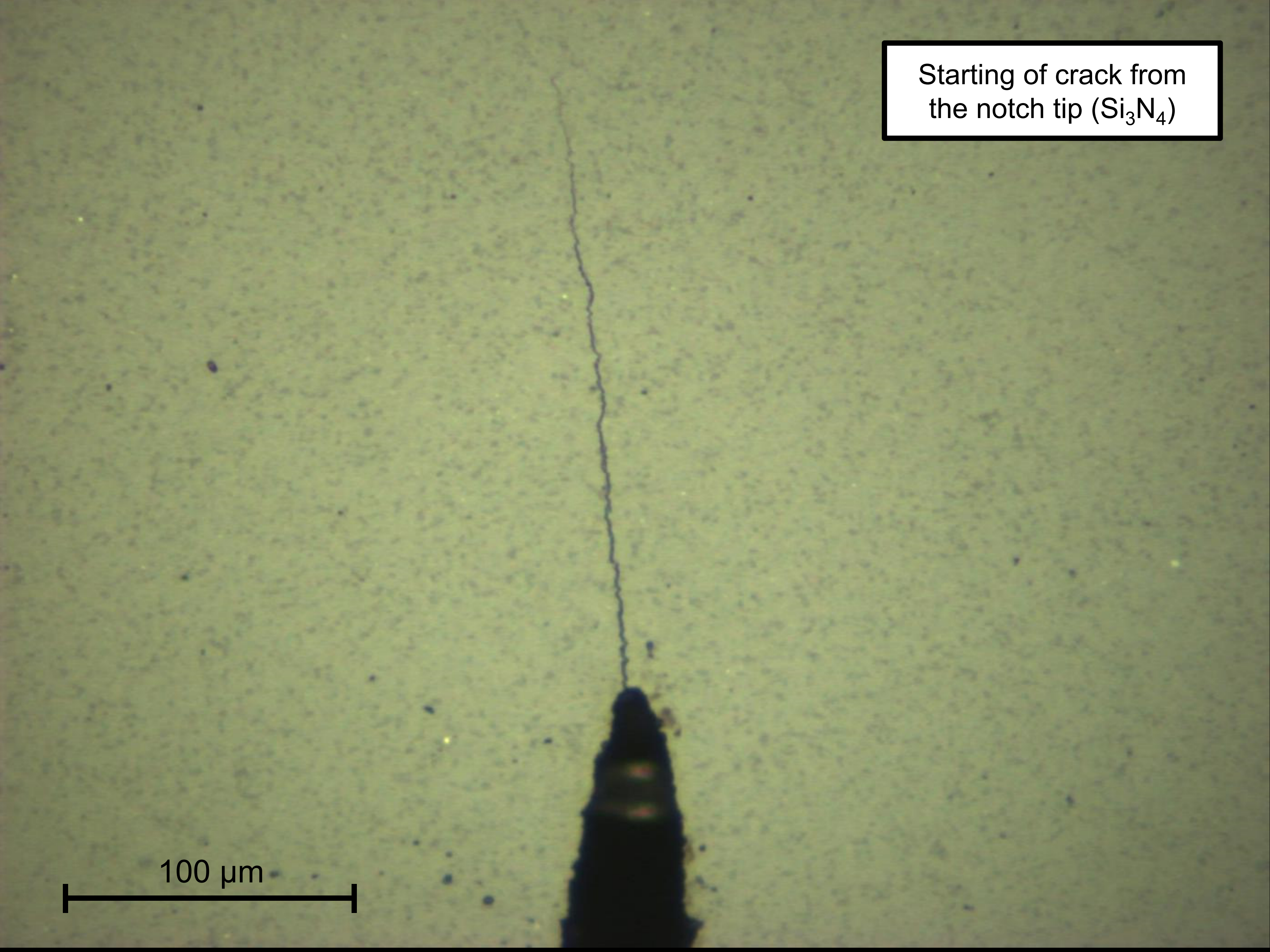
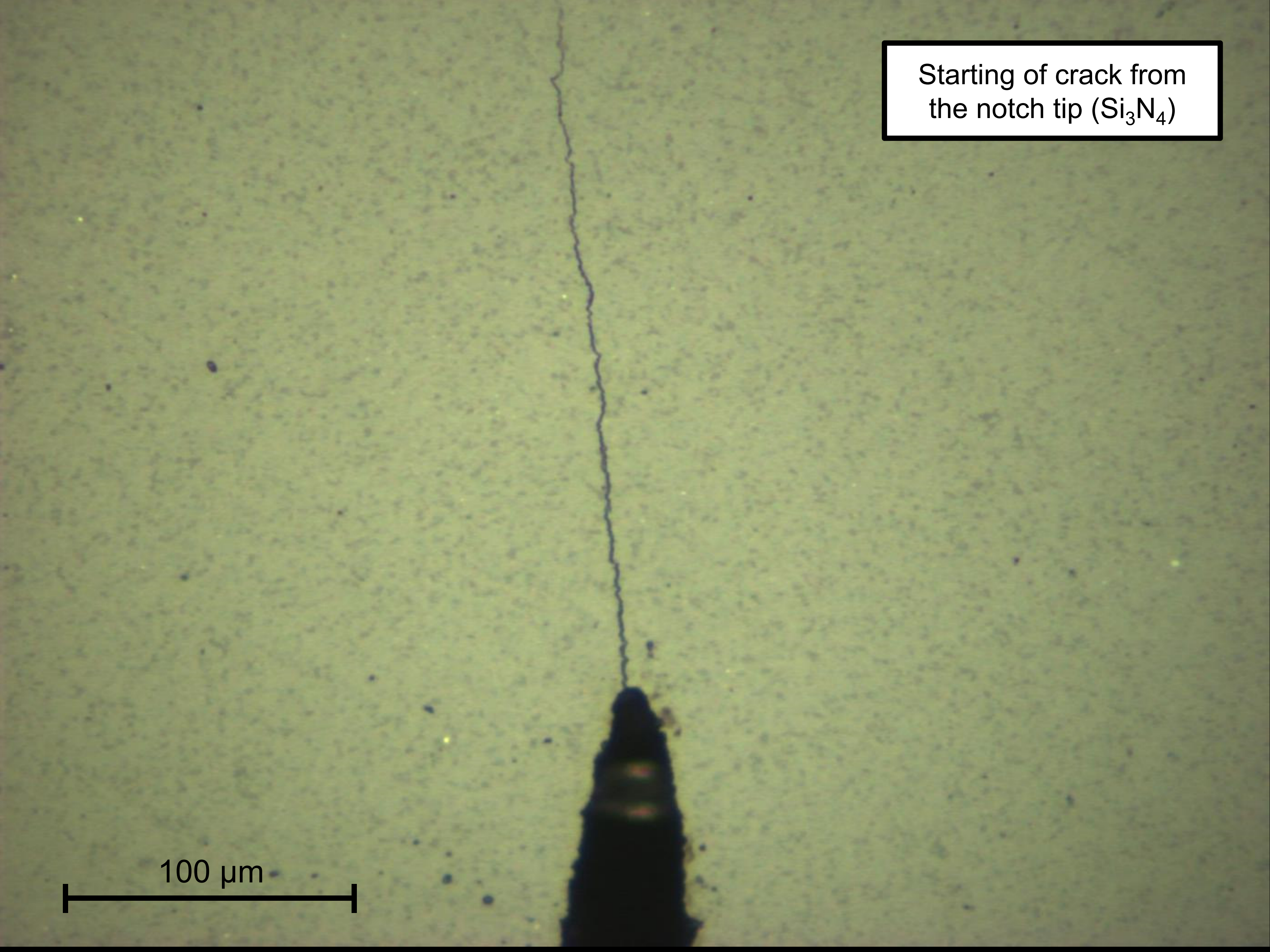

Starting of crack from the notch tip (Si_3N_4)
100 µm


Starting of crack from the notch tip (Si_3N_4)
100 μm


Starting of crack from the notch tip (Si_3N_4)
100 µm


Starting of crack from the notch tip (Si_3N_4)
100 μm


Starting of crack from the notch tip (Si_3N_4)
100 µm


Starting of crack from the notch tip (Si_3N_4)
100 µm

Determination of crack length (linear elastic fracture mechanics)

Energy release rate and toughness respectively:

$$G = \frac{F^2}{2}\frac{\partial C}{\partial \alpha} = \frac{K_{Ic}^2}{E}$$

F = force
C = compliance
A = crack surface area
E = Young's modulus (plane stress)

It follows:

$$C(\alpha) = \frac{9(s_2 - s_1)^2}{2Ewh^2}\int_0^{\alpha}\frac{\alpha'\left(Y(\alpha')\right)^2}{(1-\alpha')^3}d\alpha' + C_0 \quad \text{with} \quad \frac{Y(\alpha)}{(1-\alpha)^{3/2}} = \Gamma_M(\alpha)$$

and $$C_0 = \left(\frac{s_2 - s_1}{h}\right)^2 \frac{1}{Ew}\left(\frac{s_2 + 2s_1}{4h} + \frac{(1+\nu)h}{2(s_2 + s_1)}\right)$$

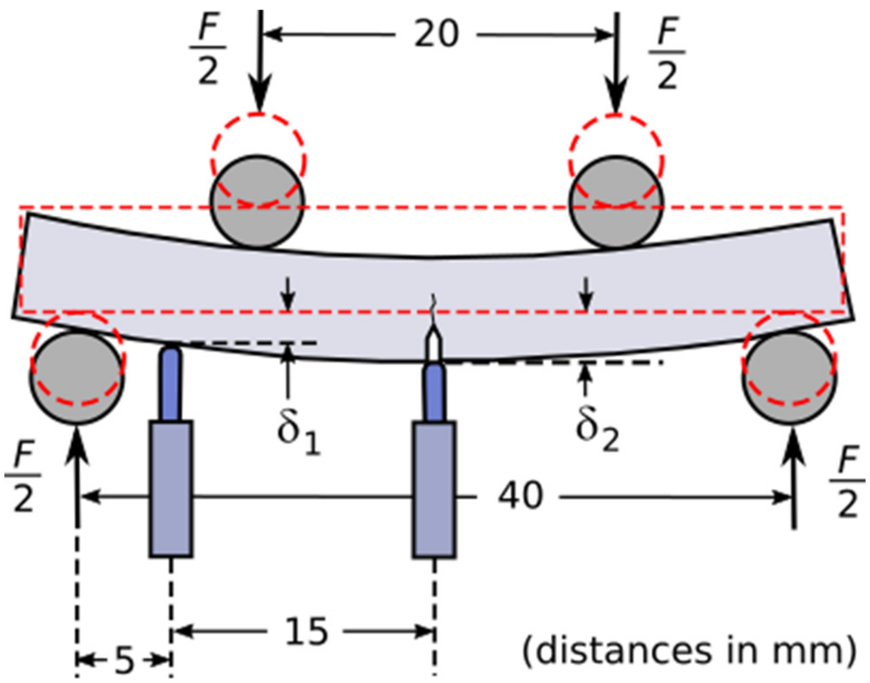
(Compliance of specimen without a crack [5], plane stress)

(ν = Poisson ratio)

The relative crack length α (upper bound of integration) has to be calculated numerically.

[5] T. Fett, E. Diegele: Indirect Measurements of Compliances in Four-Point-Bending Tests. **J. Test. Eval. 16/5** (1988) 487

Correction of measured compliance



$$C_{true} = \kappa \cdot C_{meas} + \lambda \cdot C_0$$ [5]

with $C_{meas} = \dfrac{(\Delta\delta)_2 - (\Delta\delta)_1}{\Delta F}$

and $\Delta\delta = \delta_2 - \delta_1$

For our loading geometry:

κ = 2/3

and λ = 0.4479

$$\Delta C_{true} = \frac{2}{3} \cdot \Delta C_{meas}$$

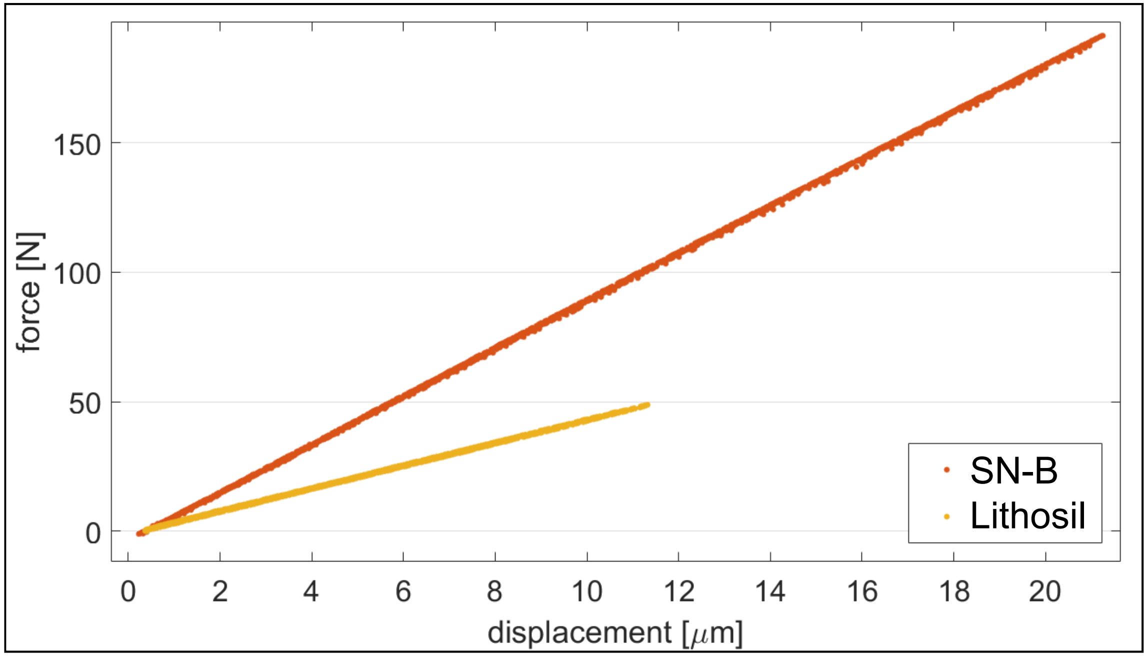
[5] T. Fett, E. Diegele: Indirect Measurements of Compliances in Four-Point-Bending Tests. **J. Test. Eval. 16/5** (1988) 487

Young‘s modulus from 4-point-bending

Young‘s modulus according to the dimensions of our load cell:

$$E = \frac{6625\ mm^3}{w\ h^3} \cdot s$$

s = slope in the force-displacement-diagram (sample without notch)



[6]

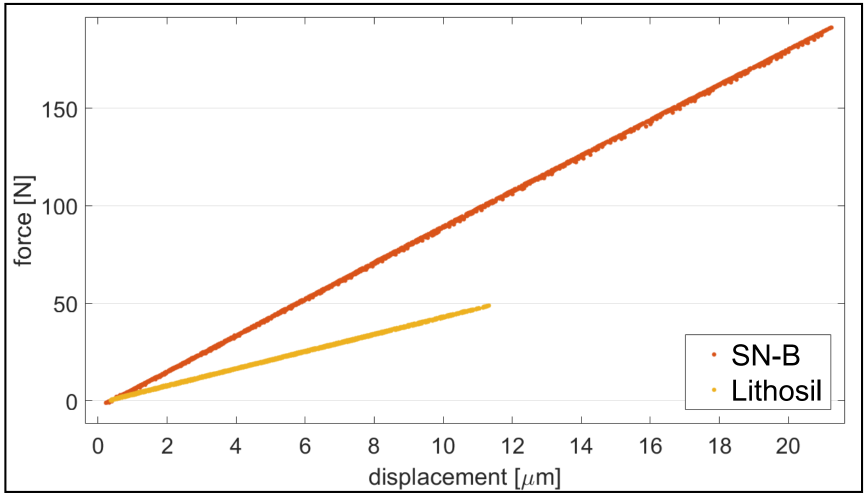
The measured materials were delivered as follows: Lithosil glass from Schott AG, Mainz and two silicon nitride materials (SN-A and SN-B) from FCT Ingenieurkeramik GmbH, Frankenblick.

[6] J. Krivohlavek: Optimierung einer neuartigen Biegemaschine für schnelle v-K-Kurven-Messungen …, bachelor thesis, TUHH (2016)

Lithosil glass	E-modulus [GPa] loading	E-modulus [GPa] unloading
1. mounting	72.70	71.96
2. mounting	72.82	72.19
3. mounting	72.74	72.04
turned by 90°	72.55	73.10
Average	**72.7**	**72.3**
standard dev.	**0.1**	**0.6**

SN-B No.	E-modulus [GPa] loading	E-modulus [GPa] unloading
6	299.5	300.3
7	301.0	301.0
8	303.2	302.5
9	302.9	302.4
10	303.8	302.5
Average	**302.1**	**301.7**
standard dev.	**1.8**	**1.0**

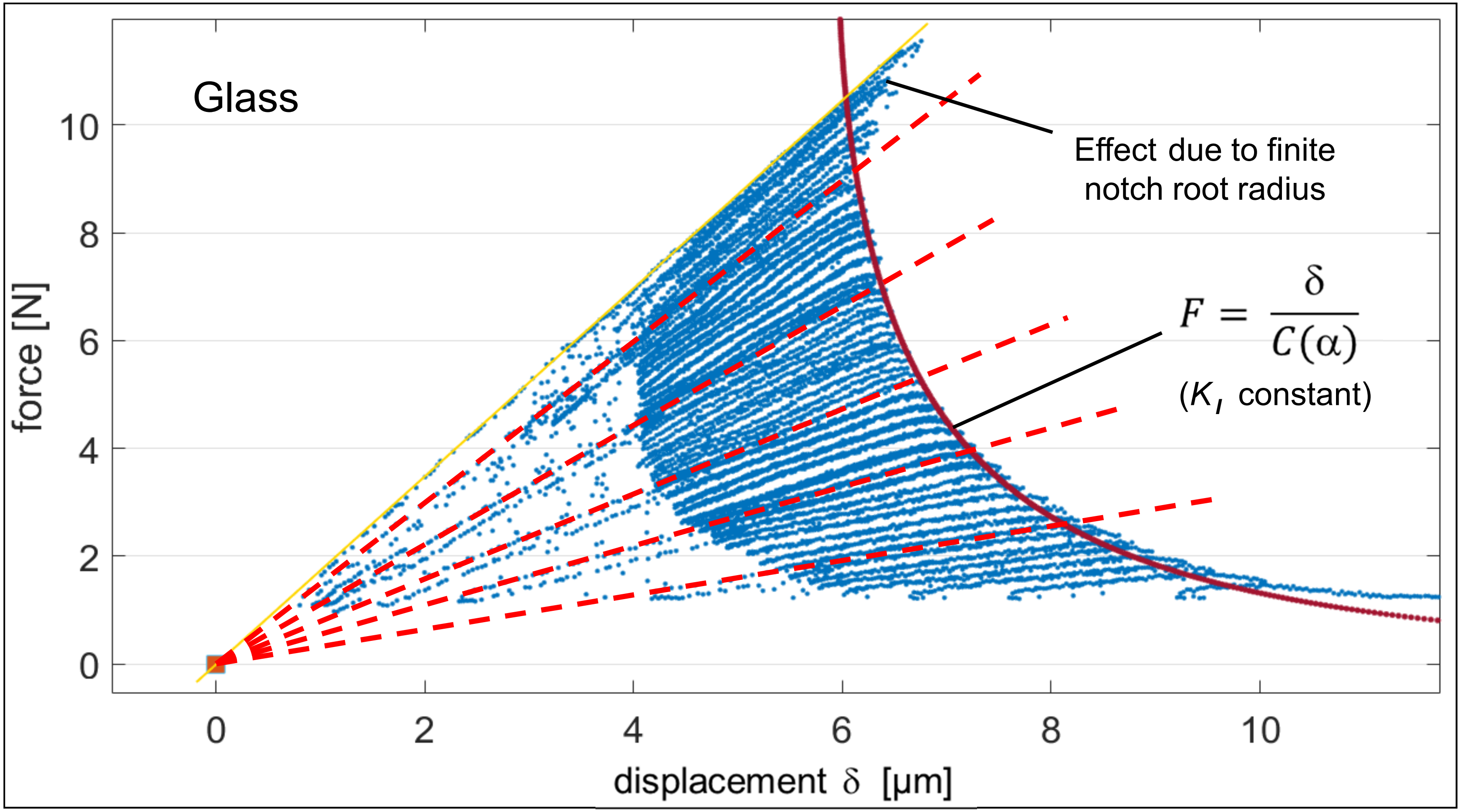
Young's modulus from 4-point-bending



Values from the manufacturers

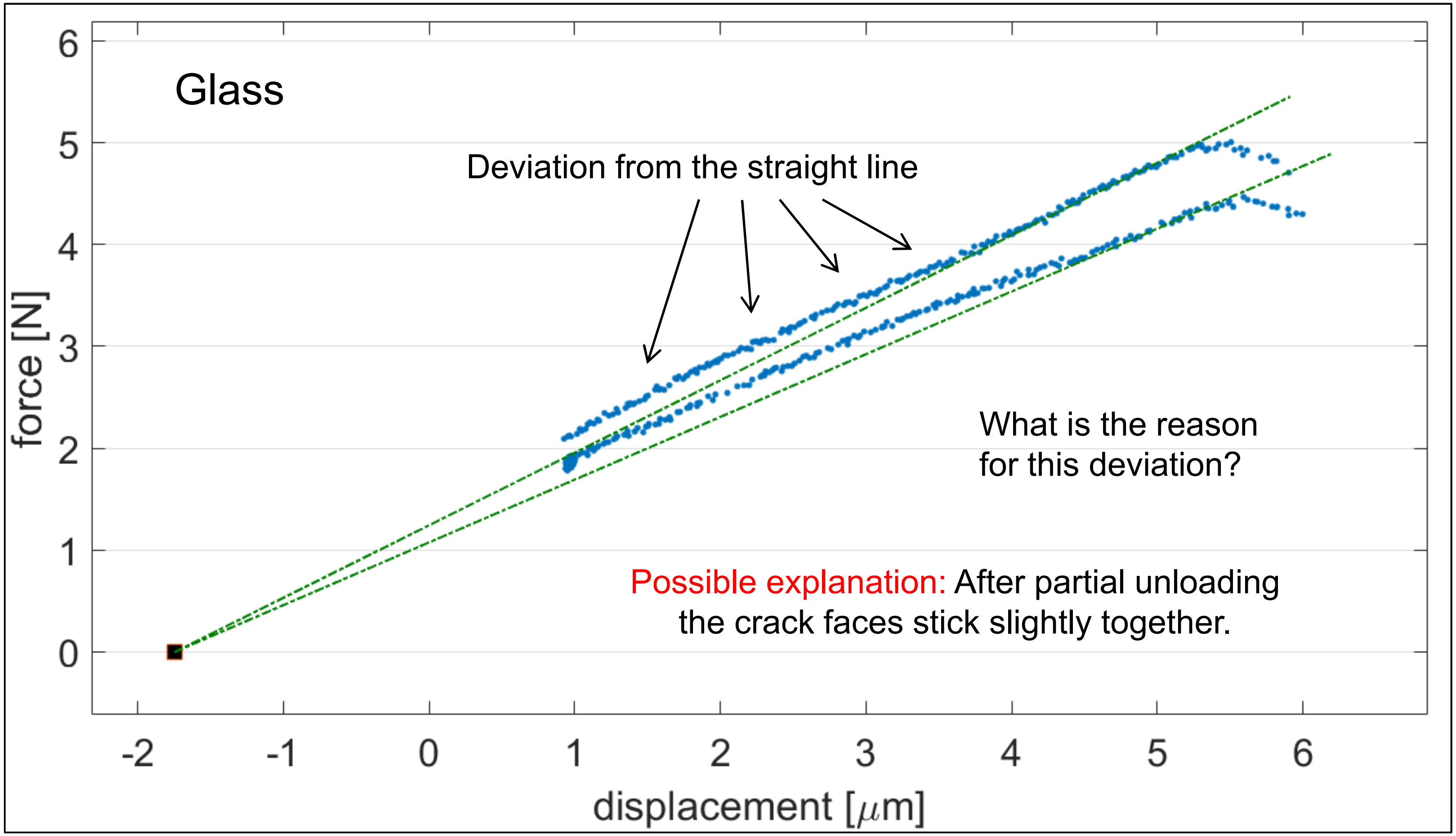
Lithosil:	72 GPa	(Schott AG)
SN-B:	299.9 ± 1.54 GPa	(FCT Ing.-keramik)

[6] J. Krivohlavek: Optimierung …, bachelor thesis, TUHH (2016)



[6] J. Krivohlavek: Optimierung …, bachelor thesis, TUHH (2016)

4b. Measurements, R-curves (load-displacement diagrams)



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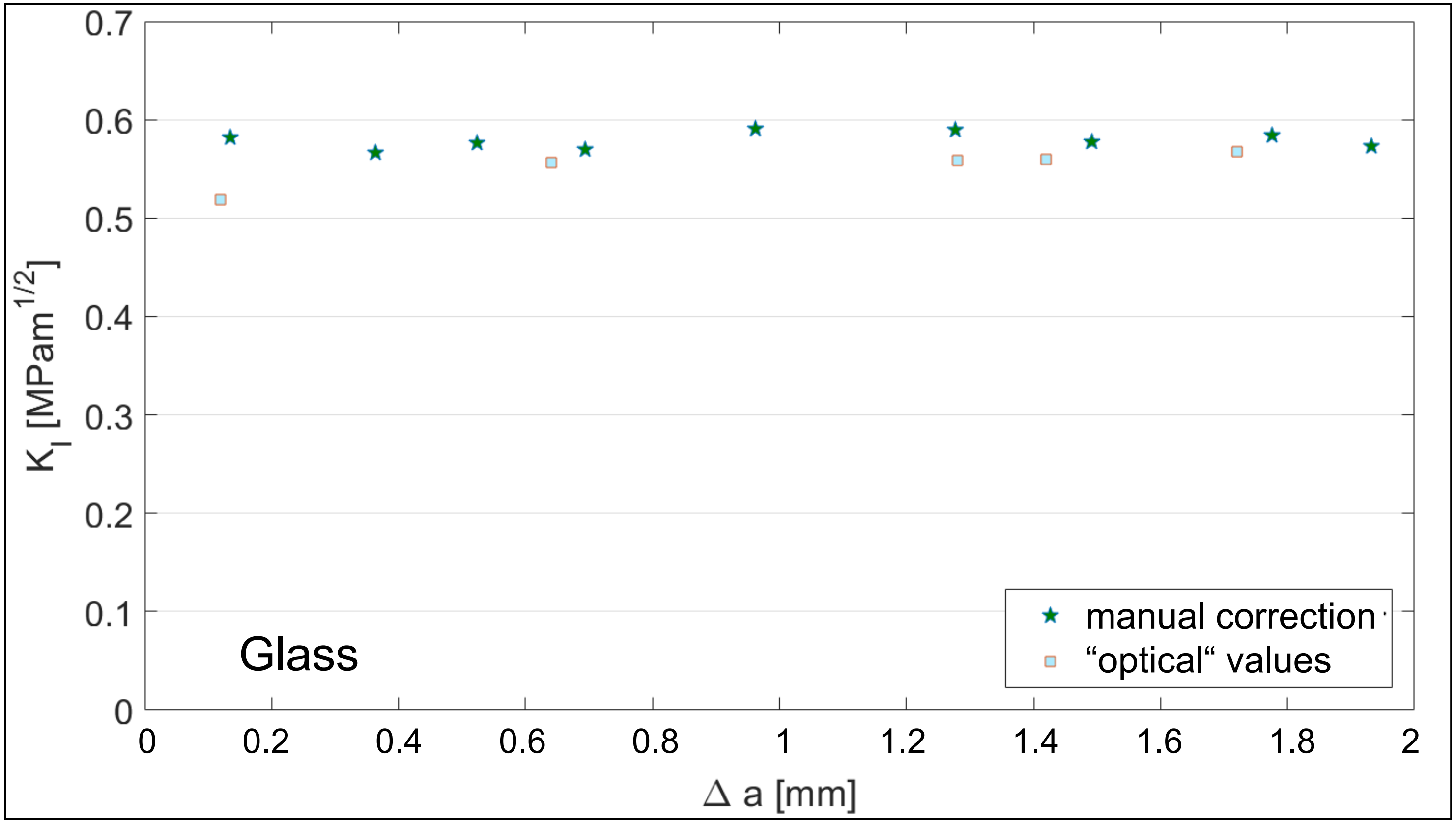
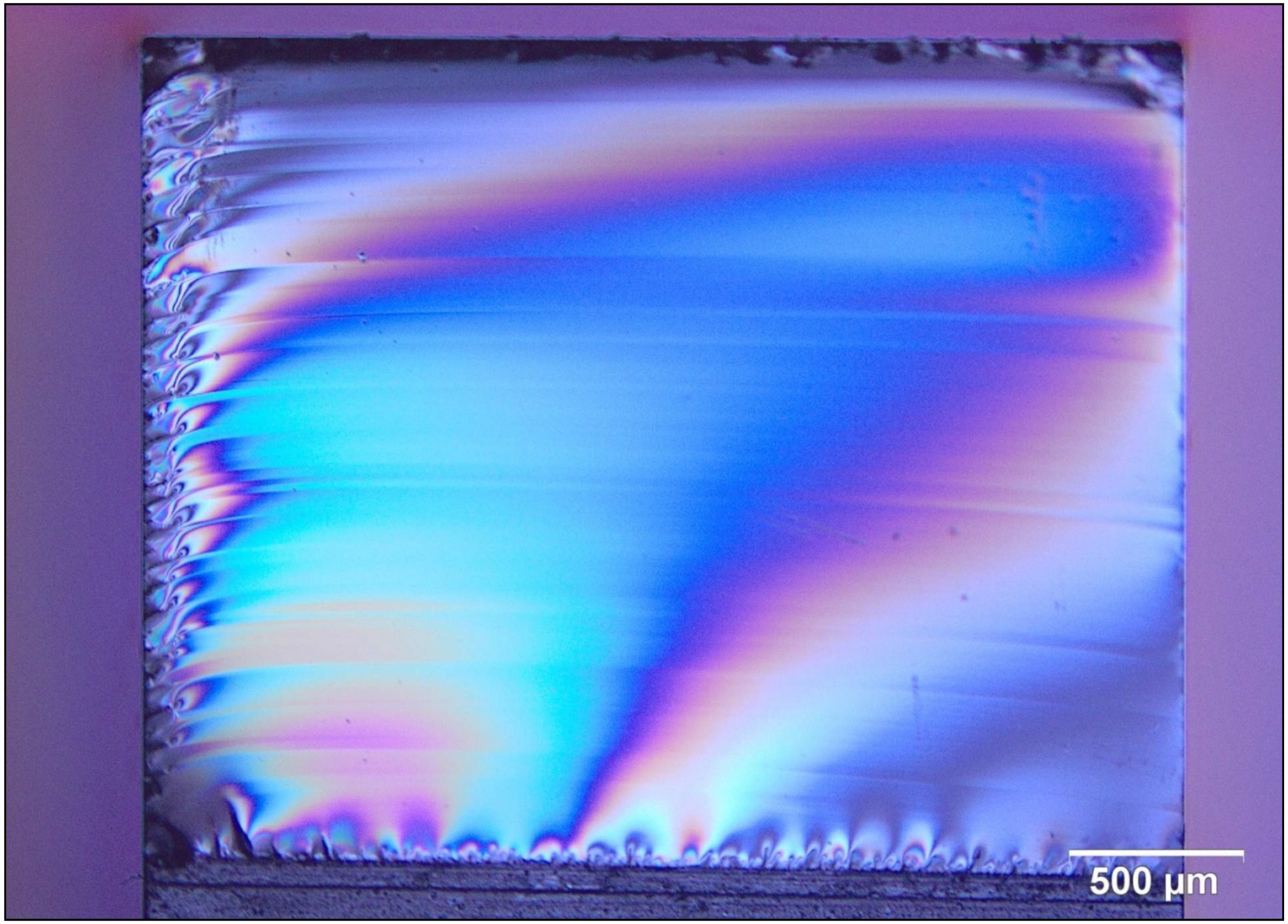


For this test, only a few data points were manually corrected (crack velocity approx. 10 µm/s).

[6] J. Krivohlavek: Optimierung …, bachelor thesis, TUHH (2016)

Crack face of glass sample with curved rest lines



The reason for the curves is that the dominating state in the middle of the crack front is „plane strain“ and near to the surface of the specimen „plane stress.“

The toughness *G* for plane strain is slightly lower than for plane stress.

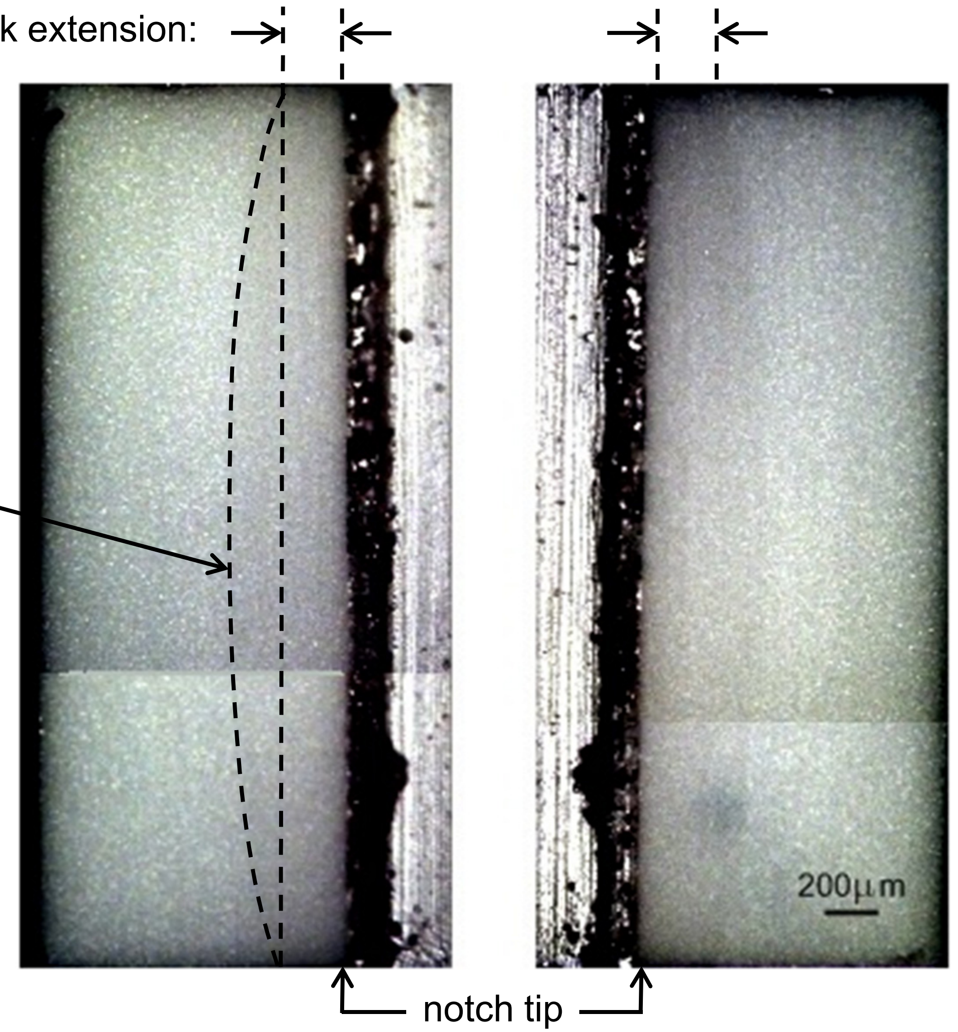
(The sample surface on the right side was polished.)

[6] J. Krivohlavek: Optimierung …, bachelor thesis, TUHH (2016)

The two crack faces of an alumina sample

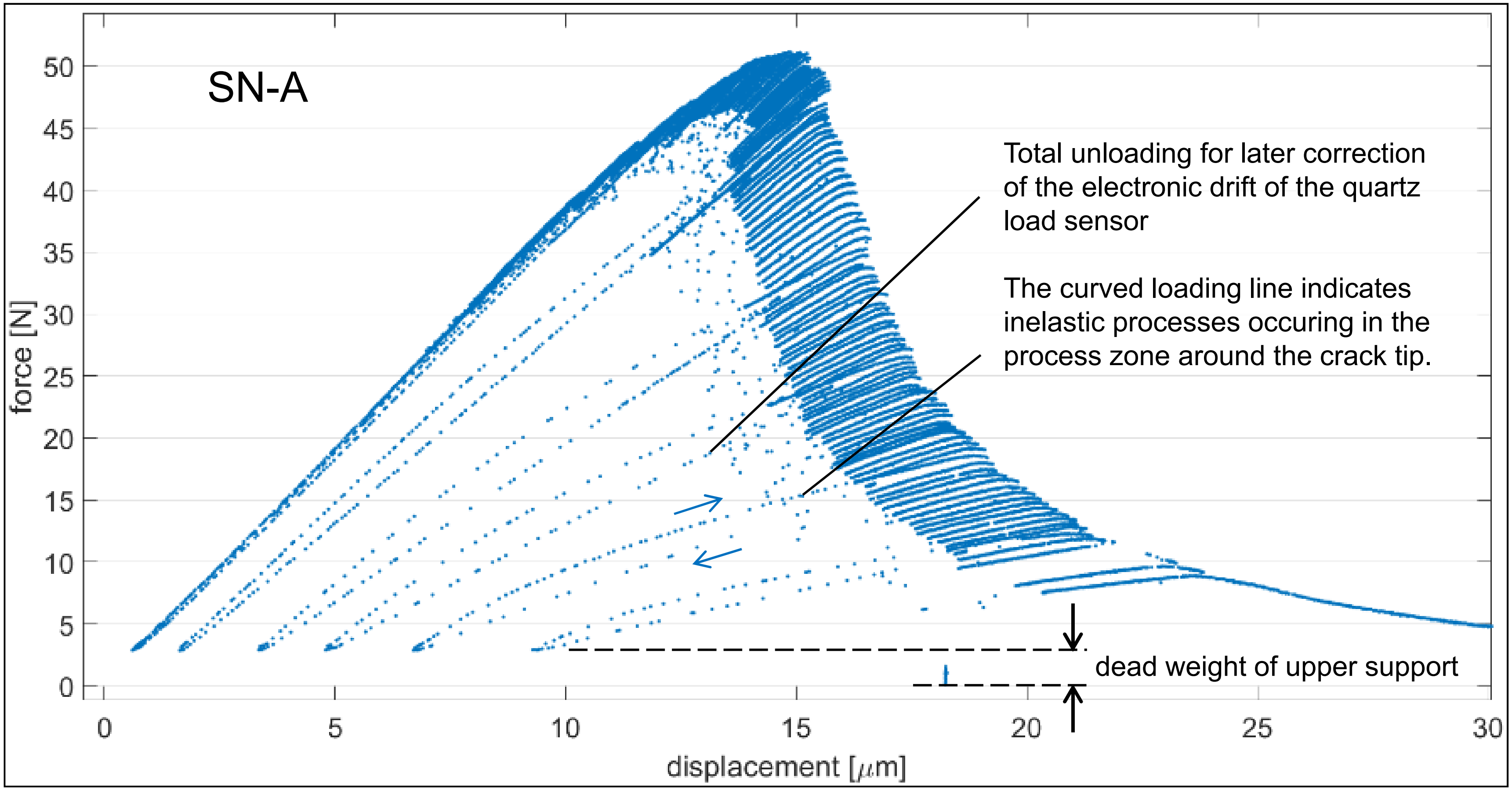
The crack front was made visible with a special liquid dye [7].

Due to the curved crack front, the K_{IC} from optical crack length measurement is a little bit lower (because of the shorter visible crack) than the K_{IC}, determined with the sample compliance.



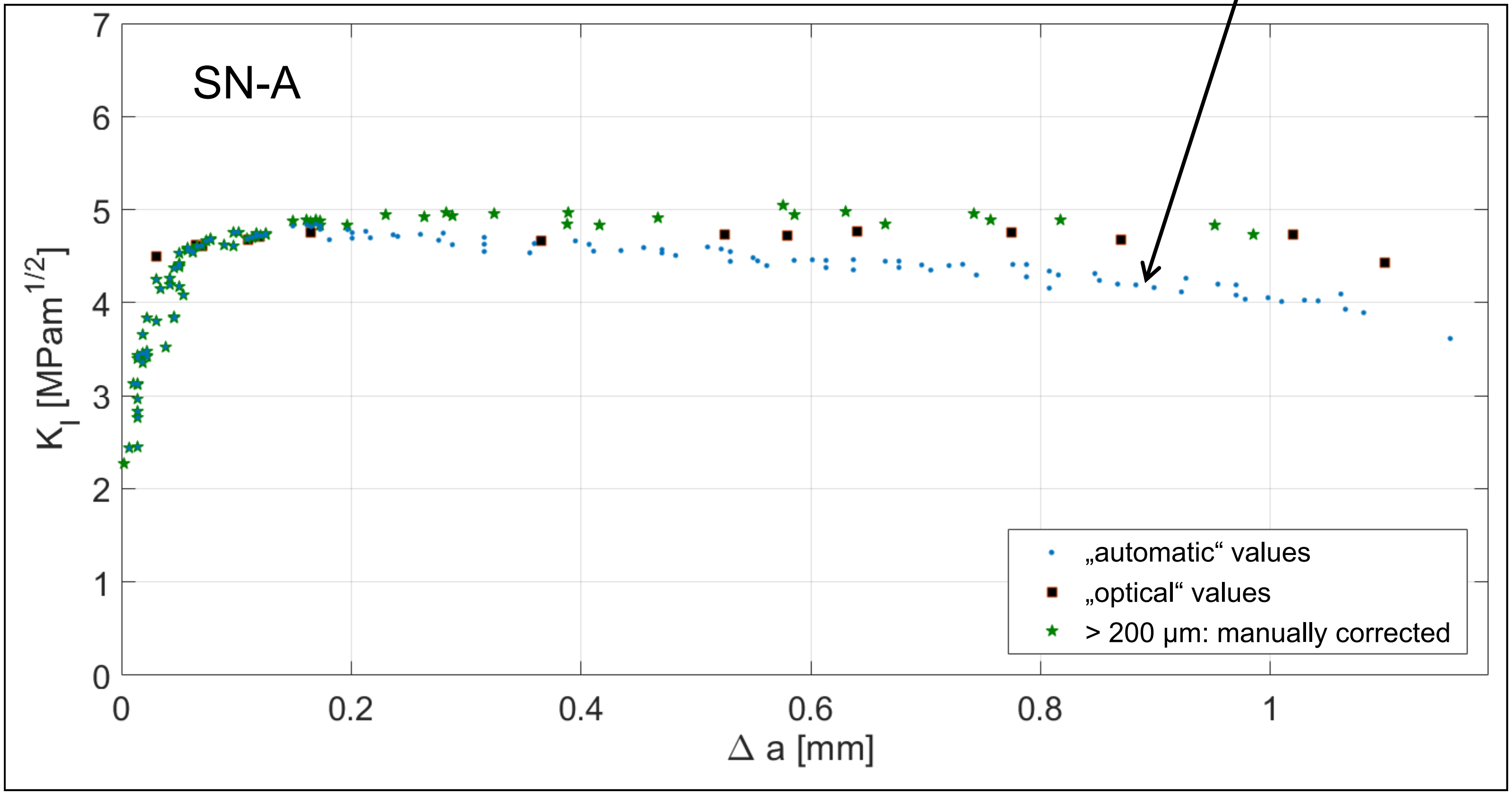
[7] V. Salikov: Untersuchungen zum Verlauf der Rissfront in Keramiken unter stabilem Risswachstum. Project work, TUHH (2009)

4b. Measurements, R-curves (load-displacement diagrams)



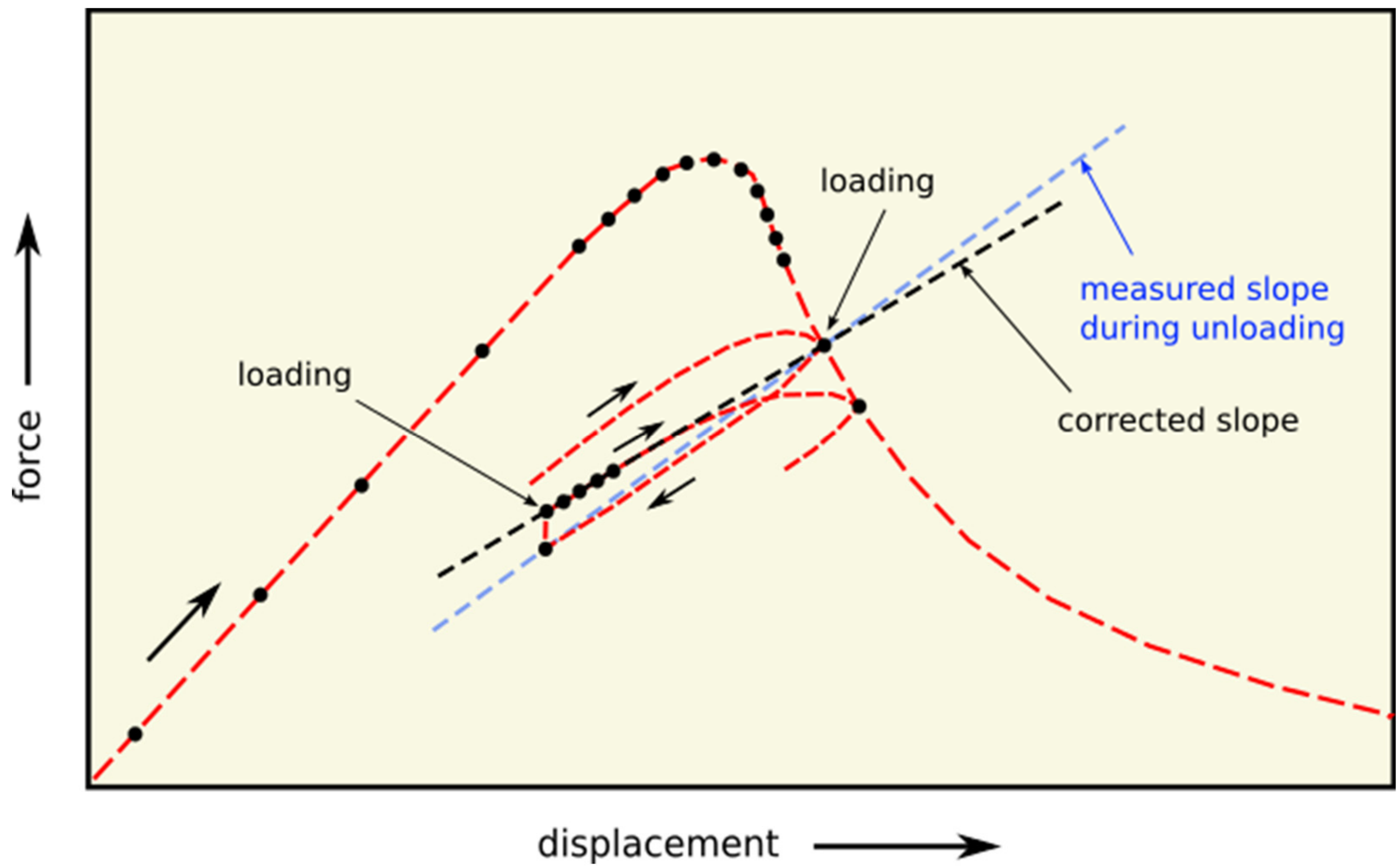
[6] J. Krivohlavek: Optimierung …, bachelor thesis, TUHH (2016)

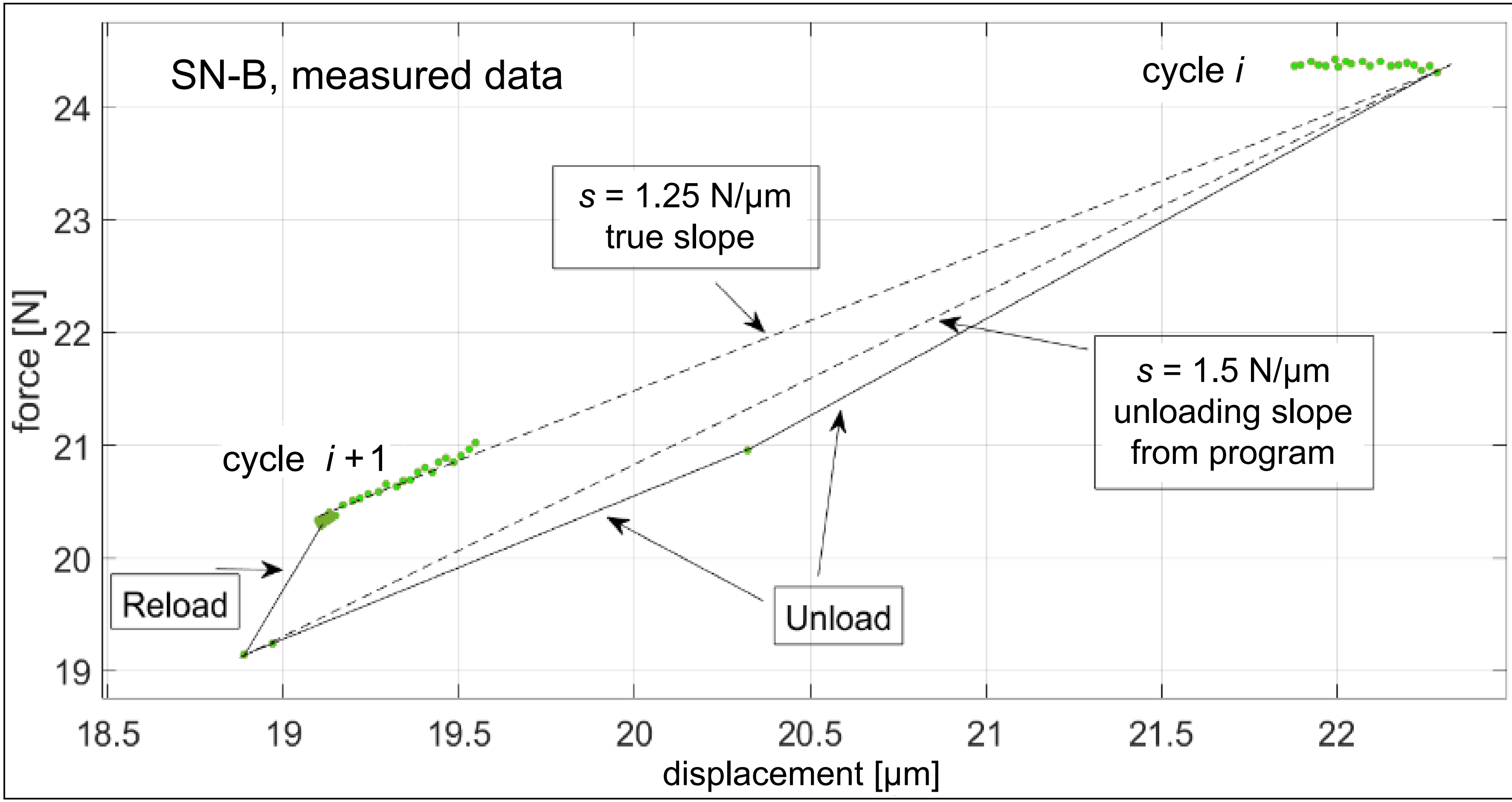
Problem: The fracture toughness, measured automatically with the compliance, decreases slightly.



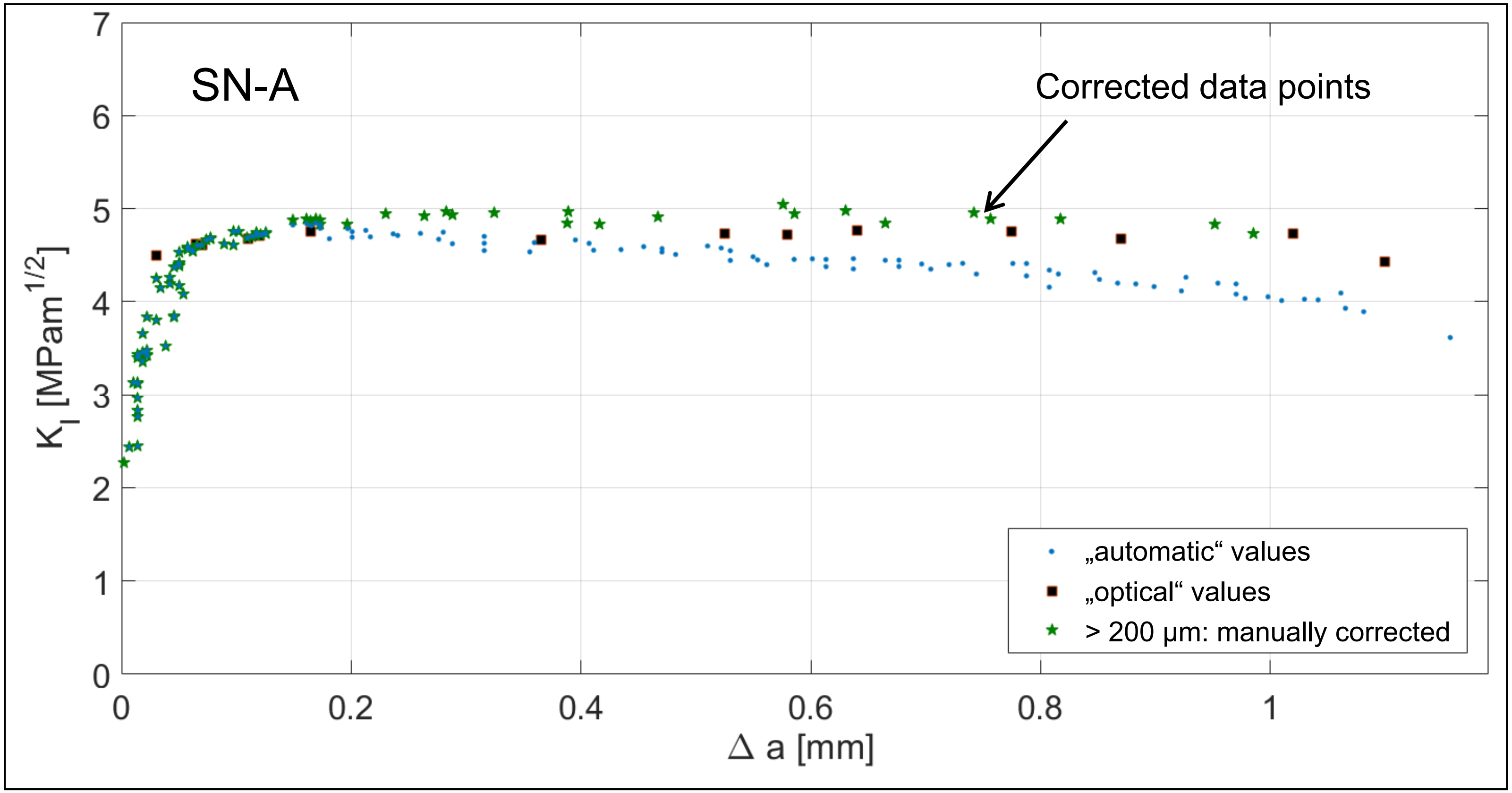
[6] J. Krivohlavek: Optimierung …, bachelor thesis, TUHH (2016)

Schema for manual correction of measured compliance





[6] J. Krivohlavek: Optimierung …, bachelor thesis, TUHH (2016)

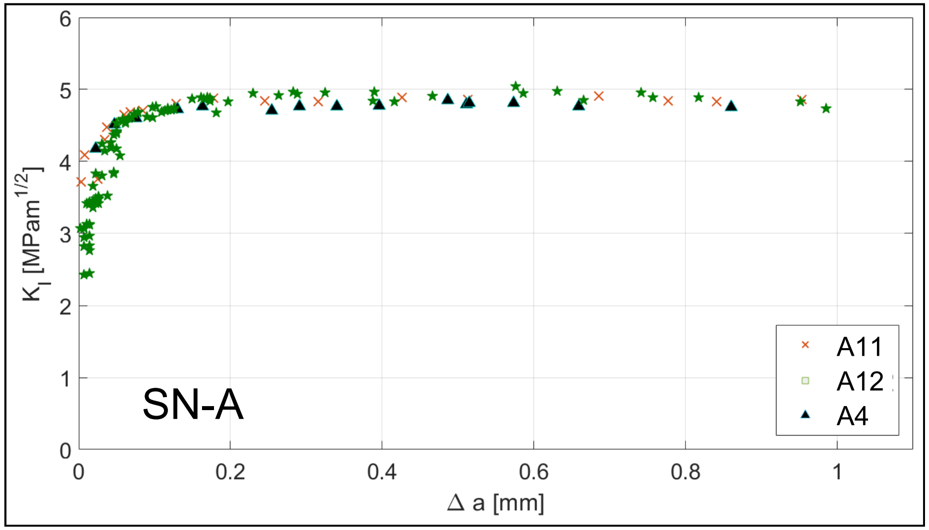


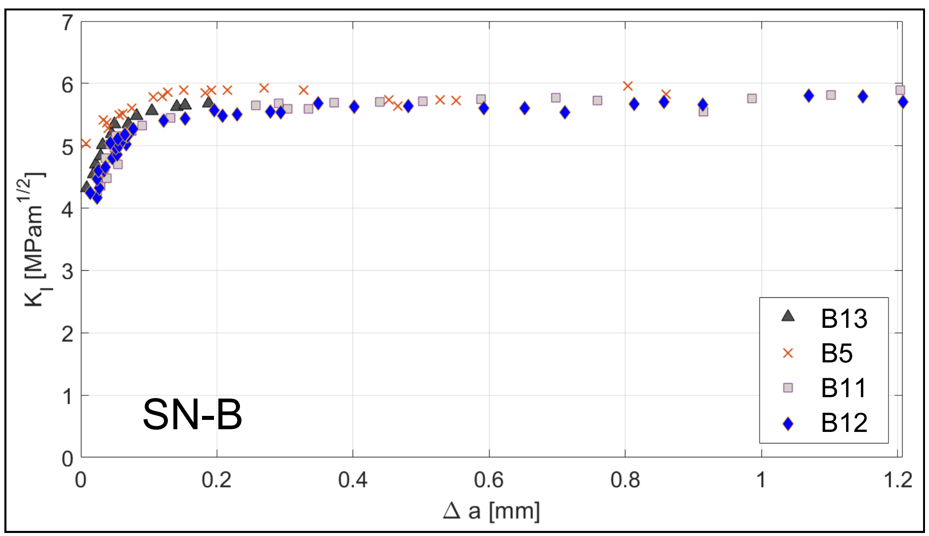
("R-curves" is written in quotation marks, because due to subcritical crack growth the plateau value of the R-curve is slightly dependent on the loading speed. Higher loading speed means a slightly higher plateau value.)

[6] J. Krivohlavek: Optimierung …, bachelor thesis, TUHH (2016)

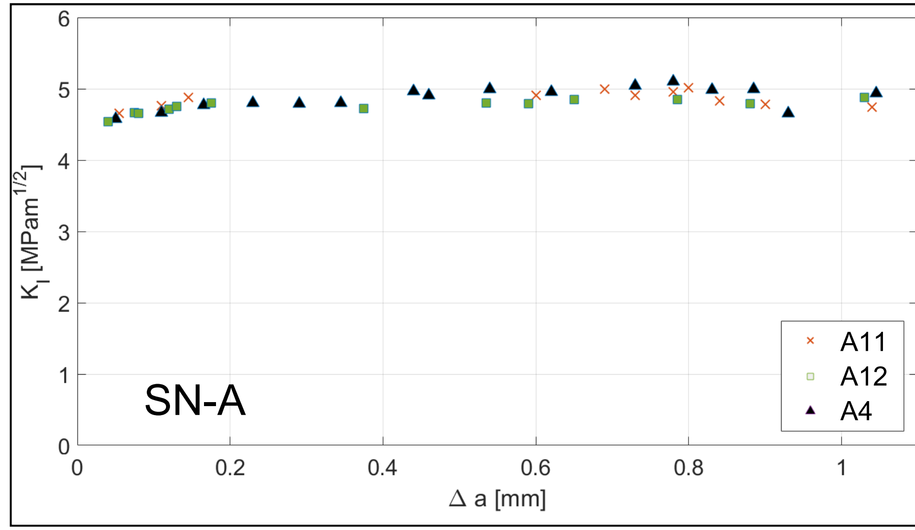
4b. Measurements, R-curves (load-displacement diagrams)

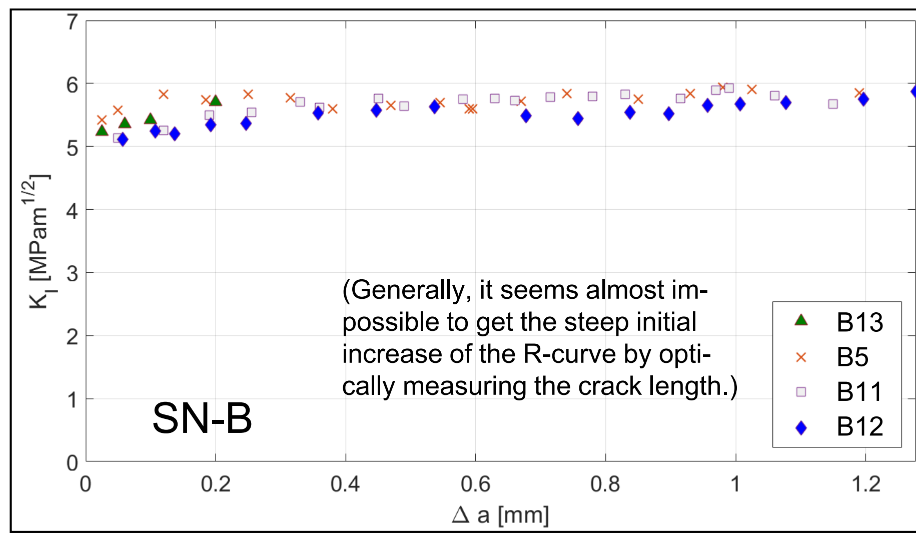
Compliance method



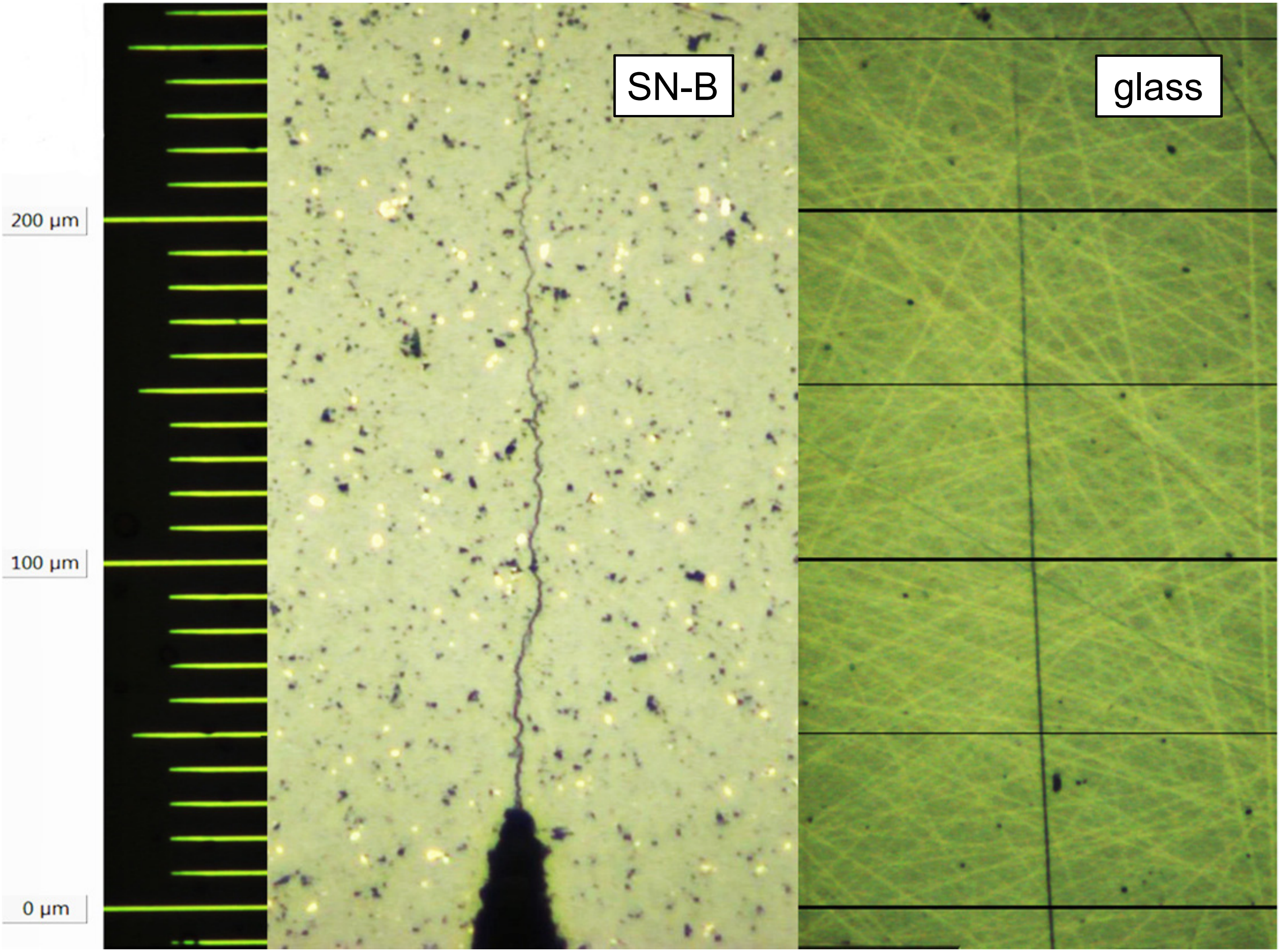


"Optical" method





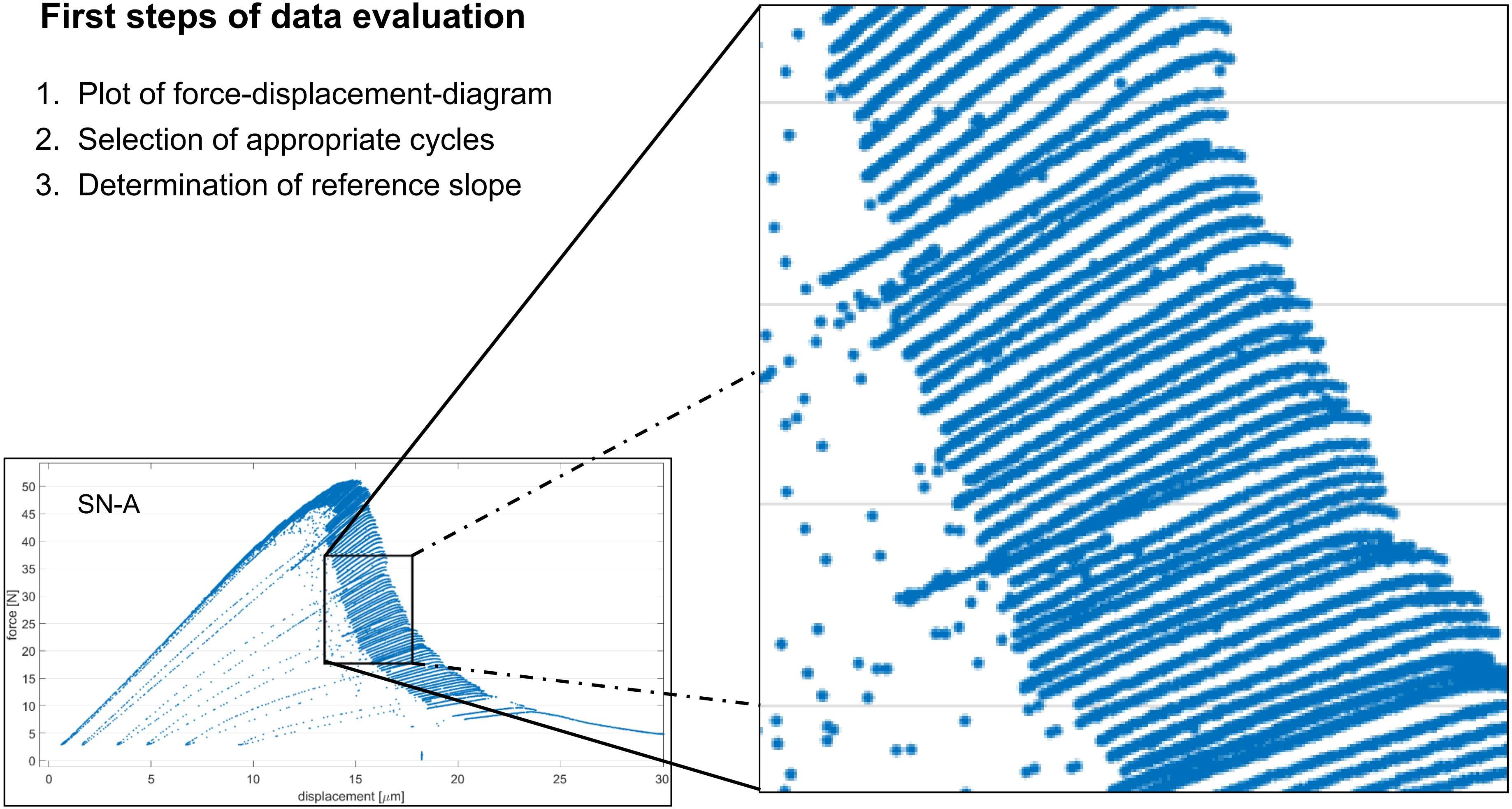
4b. Measurements (optical crack images)



[6] J. Krivohlavek: Optimierung …, bachelor thesis, TUHH (2016)

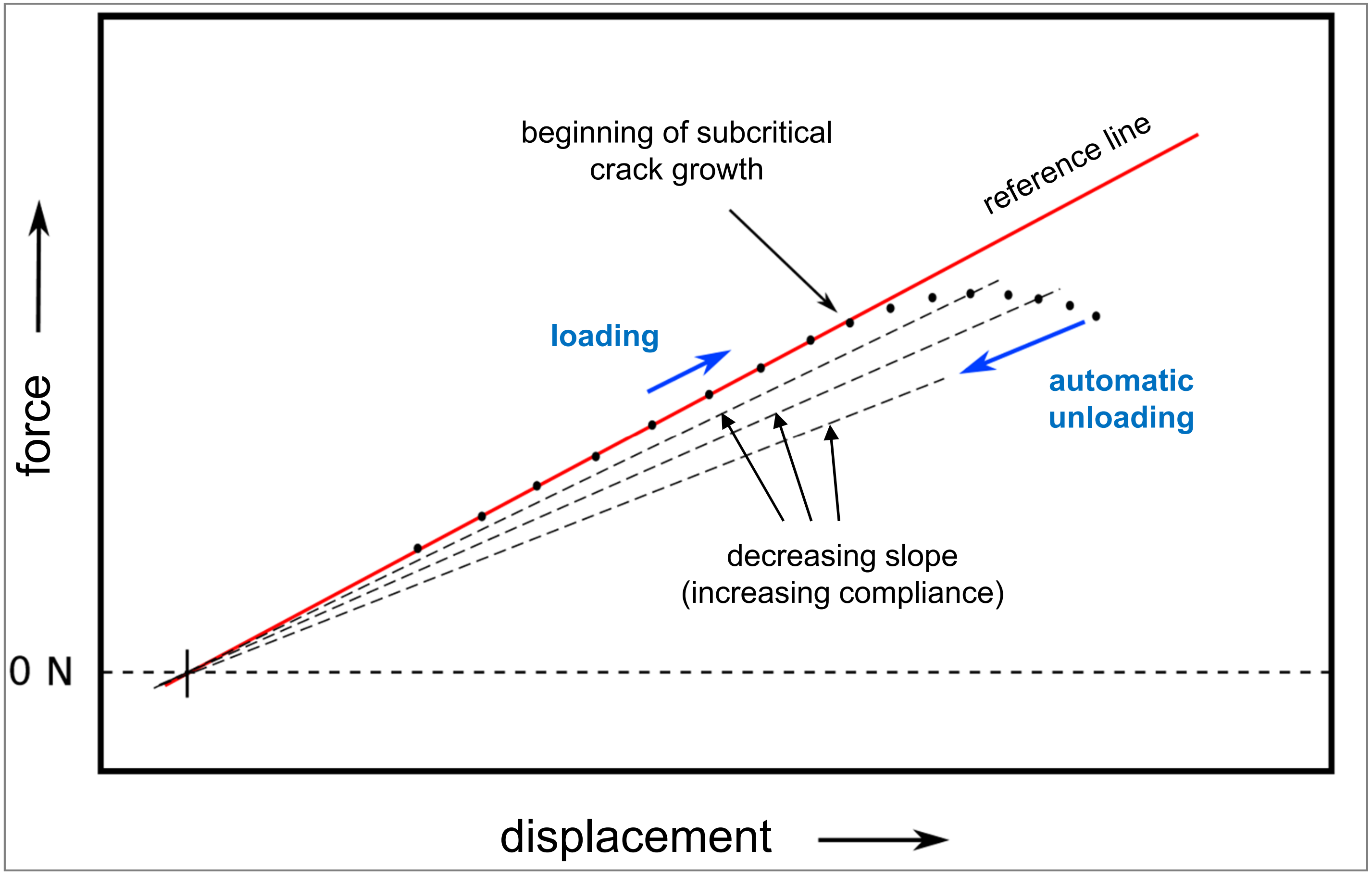
First steps of data evaluation

1. Plot of force-displacement-diagram
2. Selection of appropriate cycles
3. Determination of reference slope



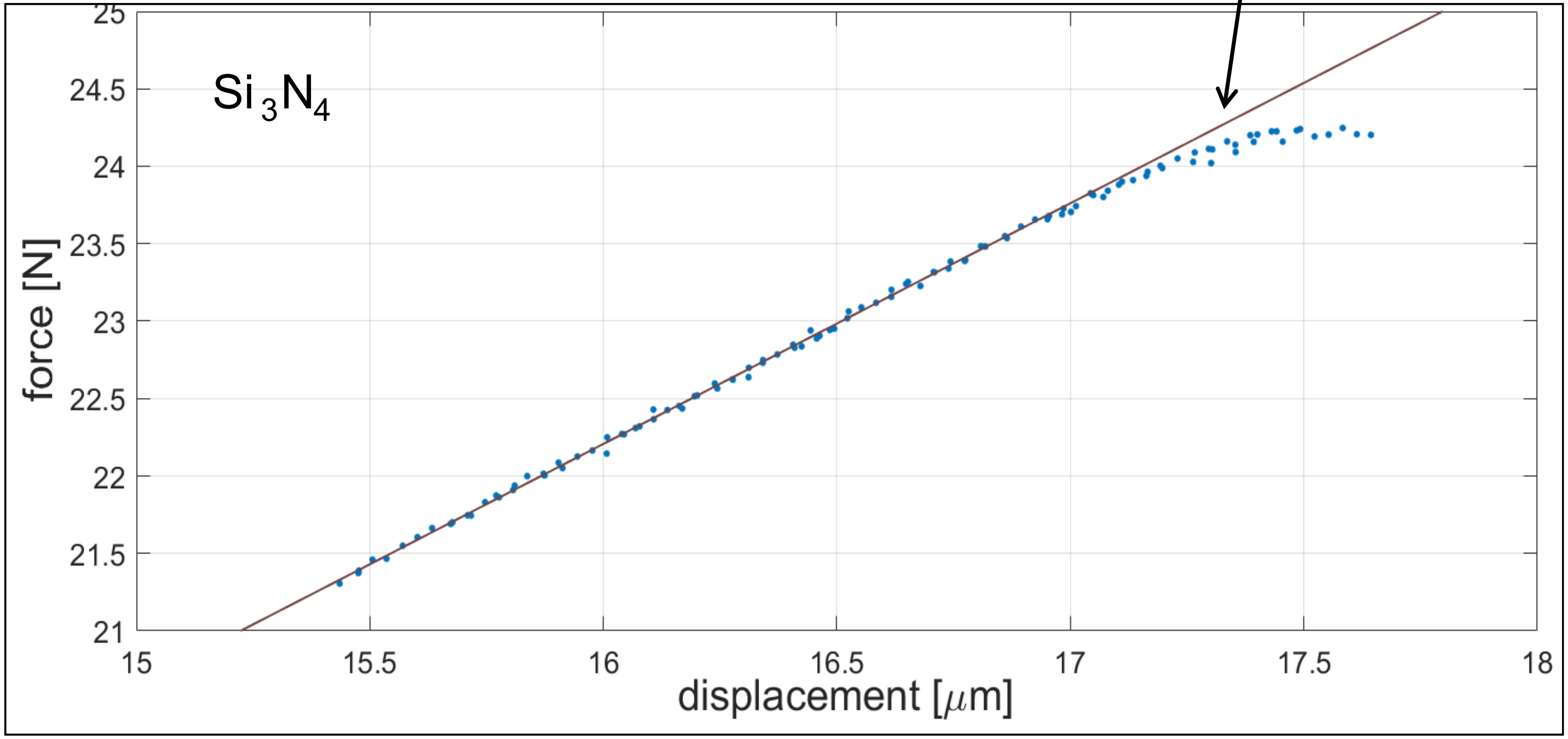
[6] J. Krivohlavek: Optimierung …, bachelor thesis, TUHH (2016)

Measurement principle



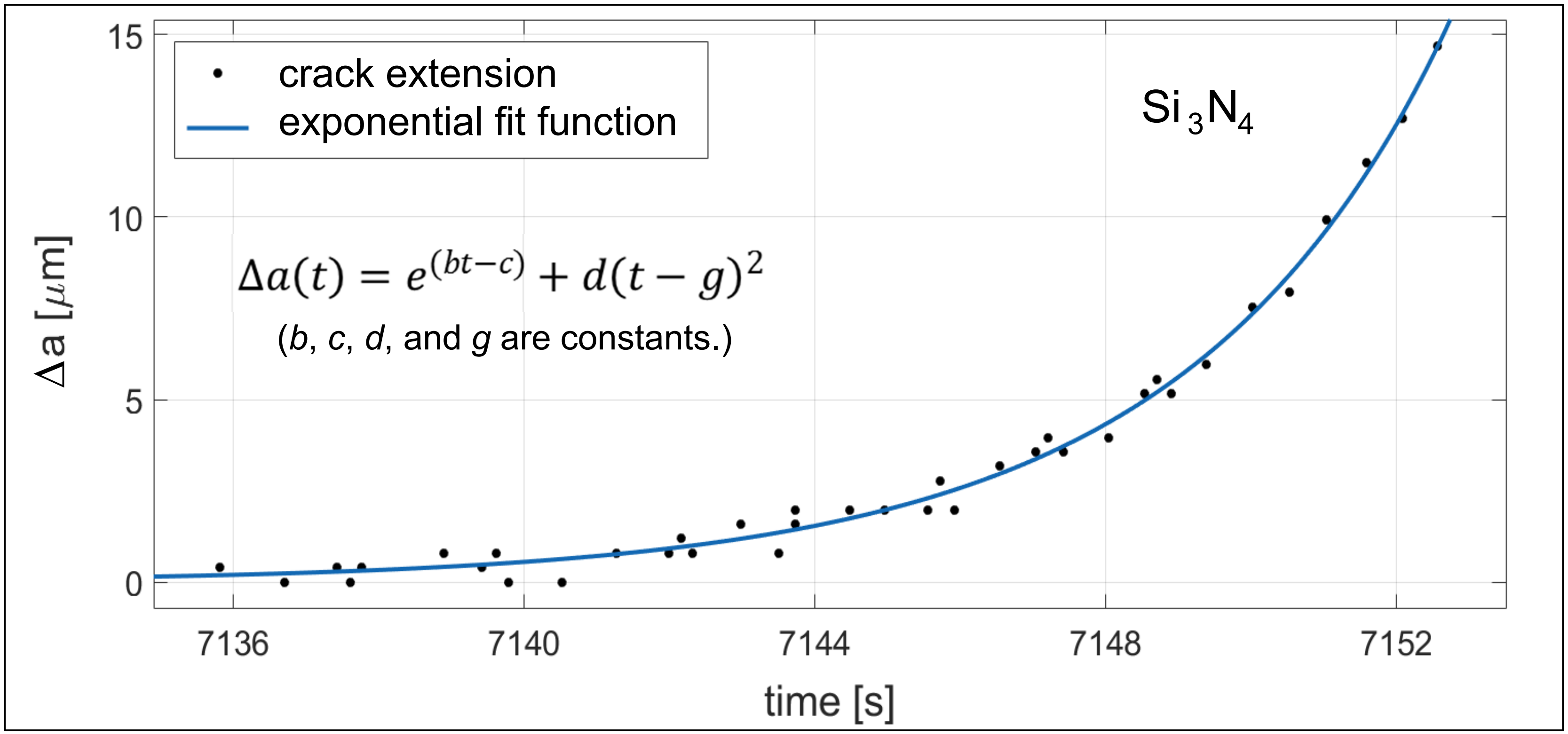
Measured single loading cycle

Change of compliance: $\Delta C(\alpha)$



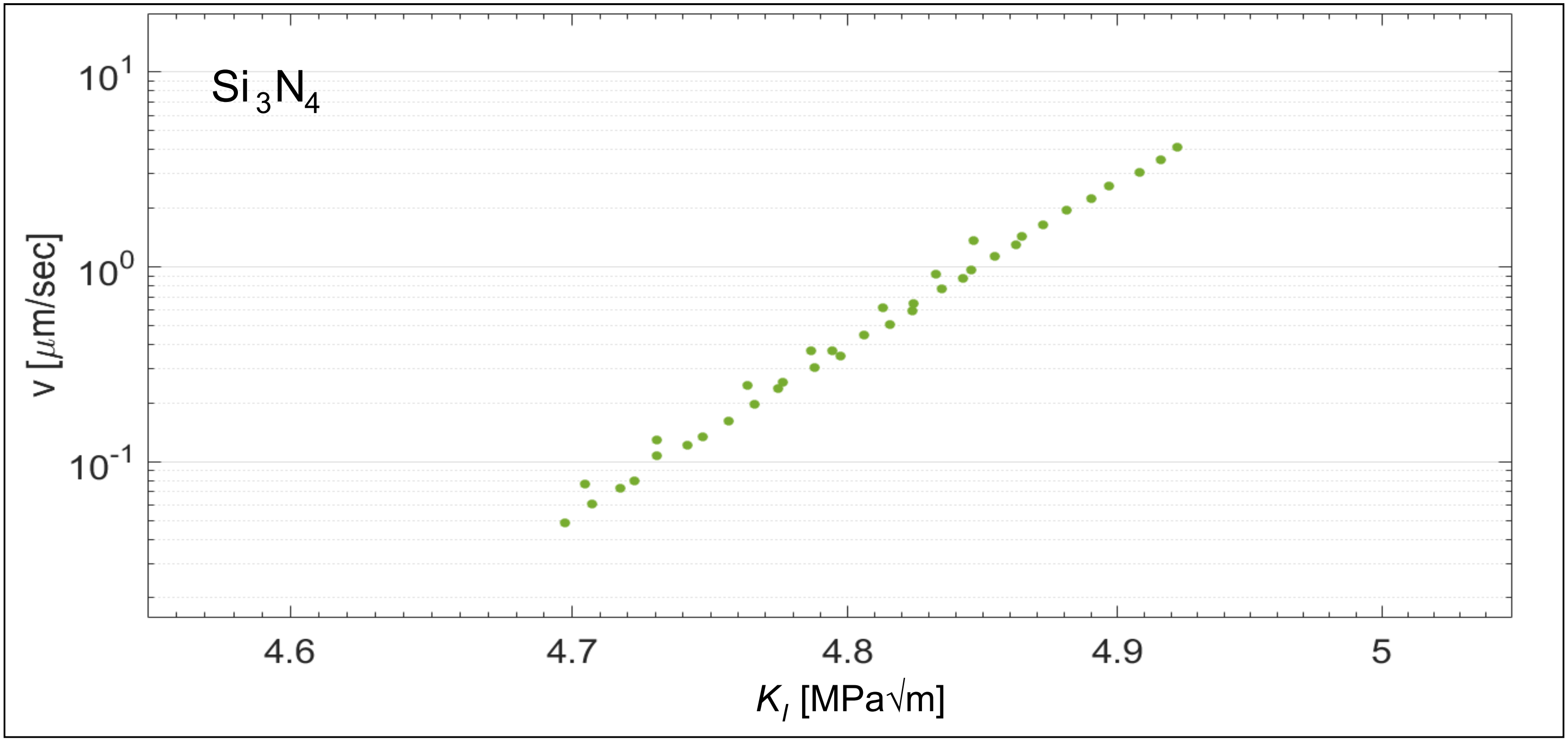
[6] J. Krivohlavek: Optimierung …, bachelor thesis, TUHH (2016)

$$\Delta C(\alpha) = \frac{9(s_2-s_1)^2}{2Ewh^2}\int_{\alpha_a}^{\alpha_b}\frac{\alpha'\left(Y(\alpha')\right)^2}{(1-\alpha')^3}\,d\alpha' \quad \Rightarrow \quad \Delta a = (\alpha_b - \alpha_a)\cdot h$$



[6] J. Krivohlavek: Optimierung …, bachelor thesis, TUHH (2016)

Derivation: $\frac{\partial a}{\partial t} = v$, furthermore, F, a yields K_I

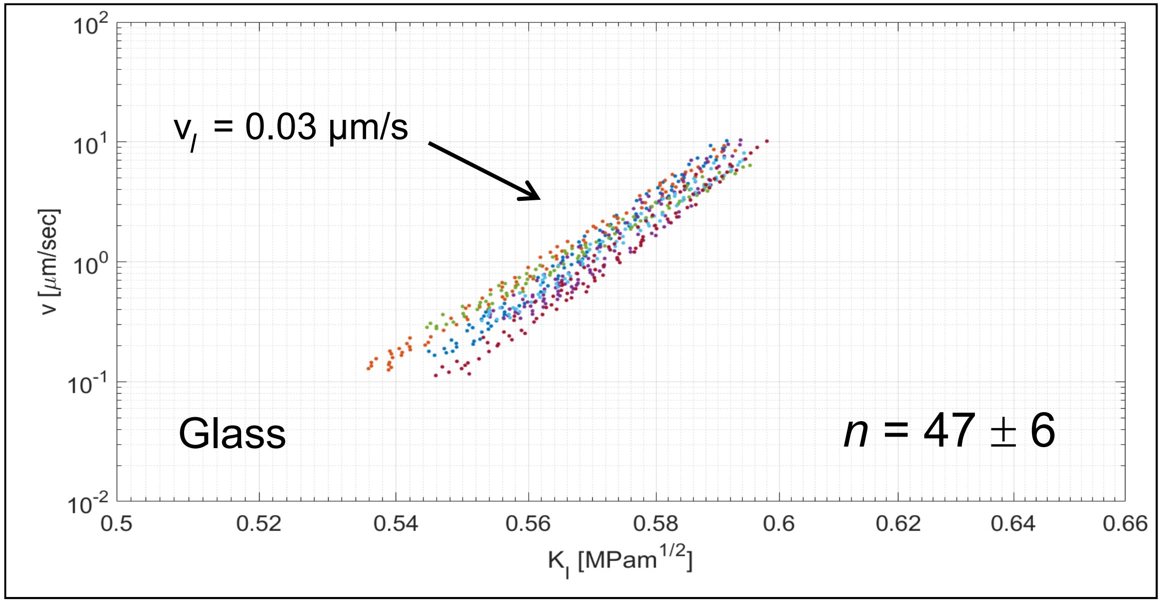


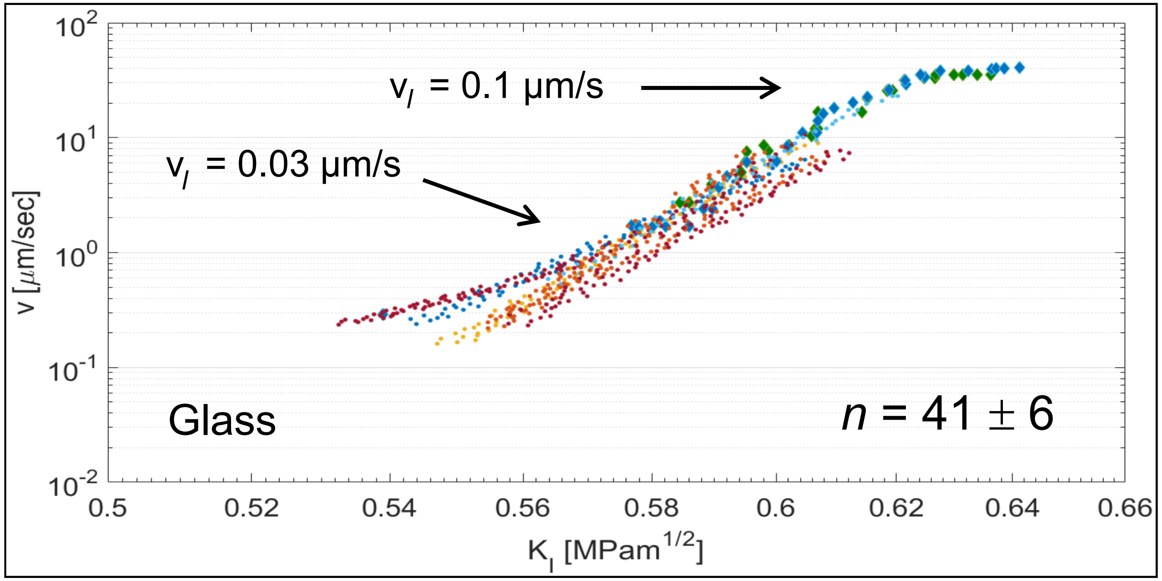
[6] J. Krivohlavek: Optimierung …, bachelor thesis, TUHH (2016)

Summary of evaluation steps (v-K-curves)

1. Determine reference slopes (manual correction)
2. $\Delta C \longrightarrow \Delta a$ (numerical calculation)
3. Fit of exponential curve $\Delta a(t)$
4. Derivative of $\Delta a(t) \longrightarrow$ crack velocity v
5. $a, F \longrightarrow K_I$
6. Logarithmic plot of $v(K_I) \longrightarrow$ v-K-curve

4c. Measurements, v-K-curves



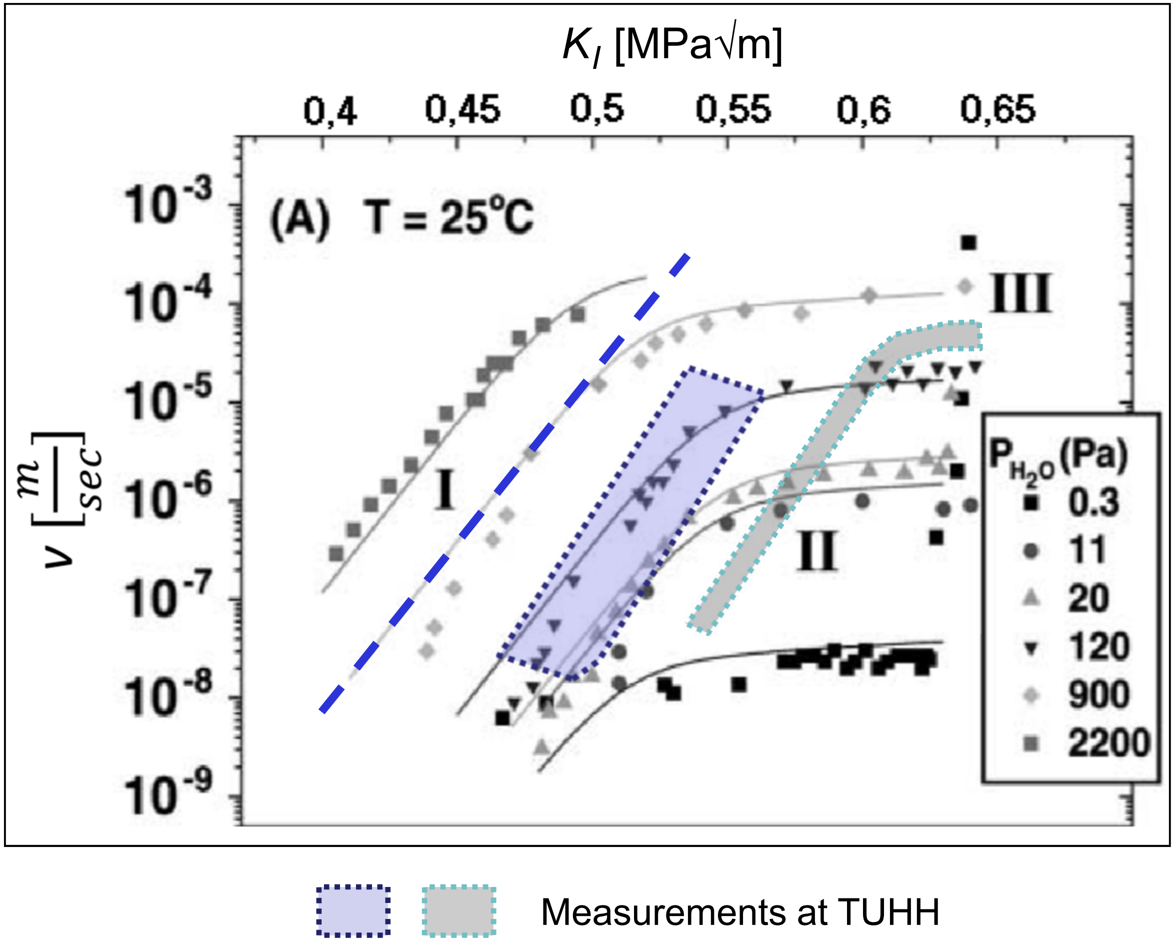


v_l: loading velocity

v : crack tip velocity

[6] J. Krivohlavek: Optimierung einer …, bachelor thesis, TUHH (2016)

Comparison with literature [8]



$n_{Lit.} = 36\ \ (28\%\ \text{hum.})$

$n_{mess} = 41\ \ \&\ \ 47$

$n_{mess} = 50\quad$ (M. Kröchert)

(T = 22–25°C, *hum.* = 20–32%)

→ **Good agreement concerning the exponent *n***

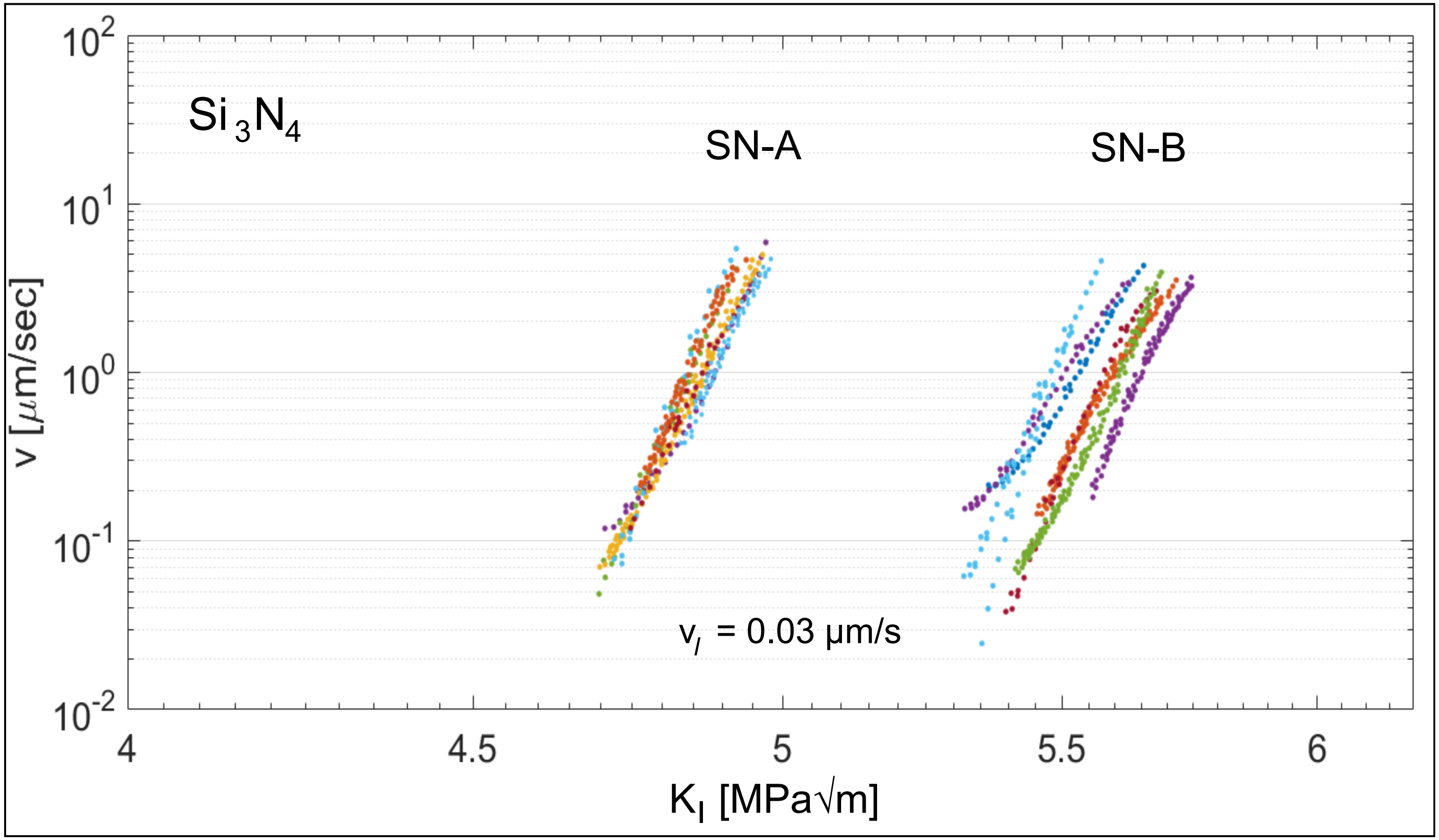
→ **Plateau at correct level**

But:

→ **Horizontal shift, probably due to different glass mat.**

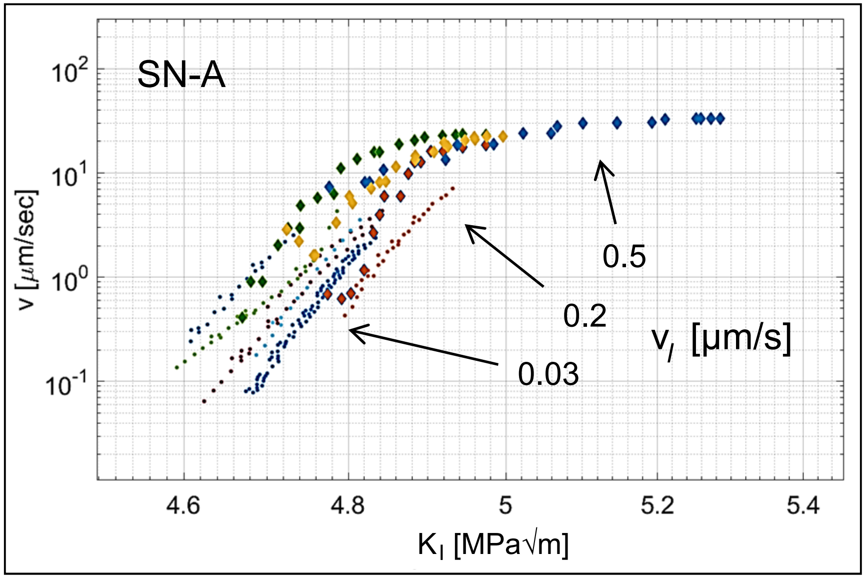
[8] T. I. Suratwala, R. A. Steele: Anomalous Temperatur Dependence of Subcritical Crack … , **J. Non-Cryst. Solids 316/1** (2003) 174

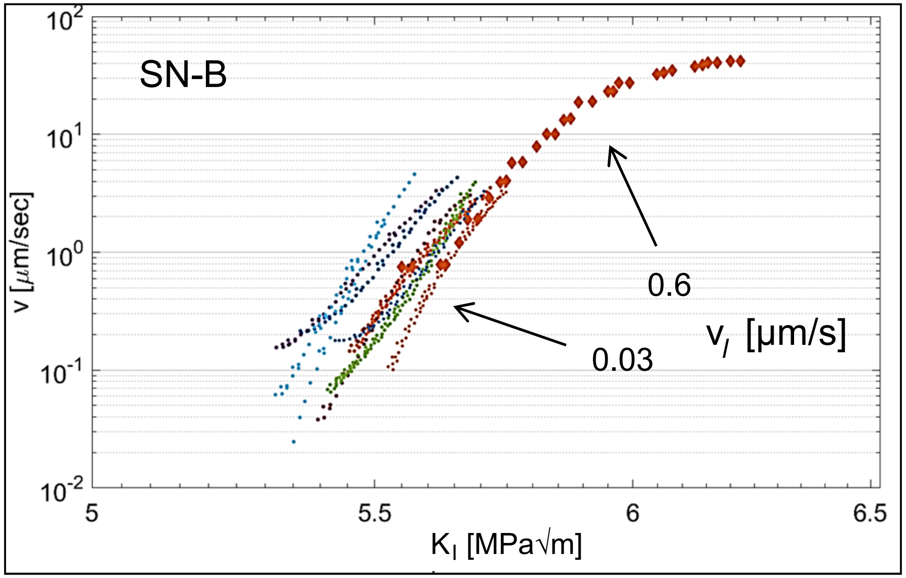
Section of the v-K-curve for low loading velocity v_l



[6] J. Krivohlavek: Optimierung …, bachelor thesis, TUHH (2016)

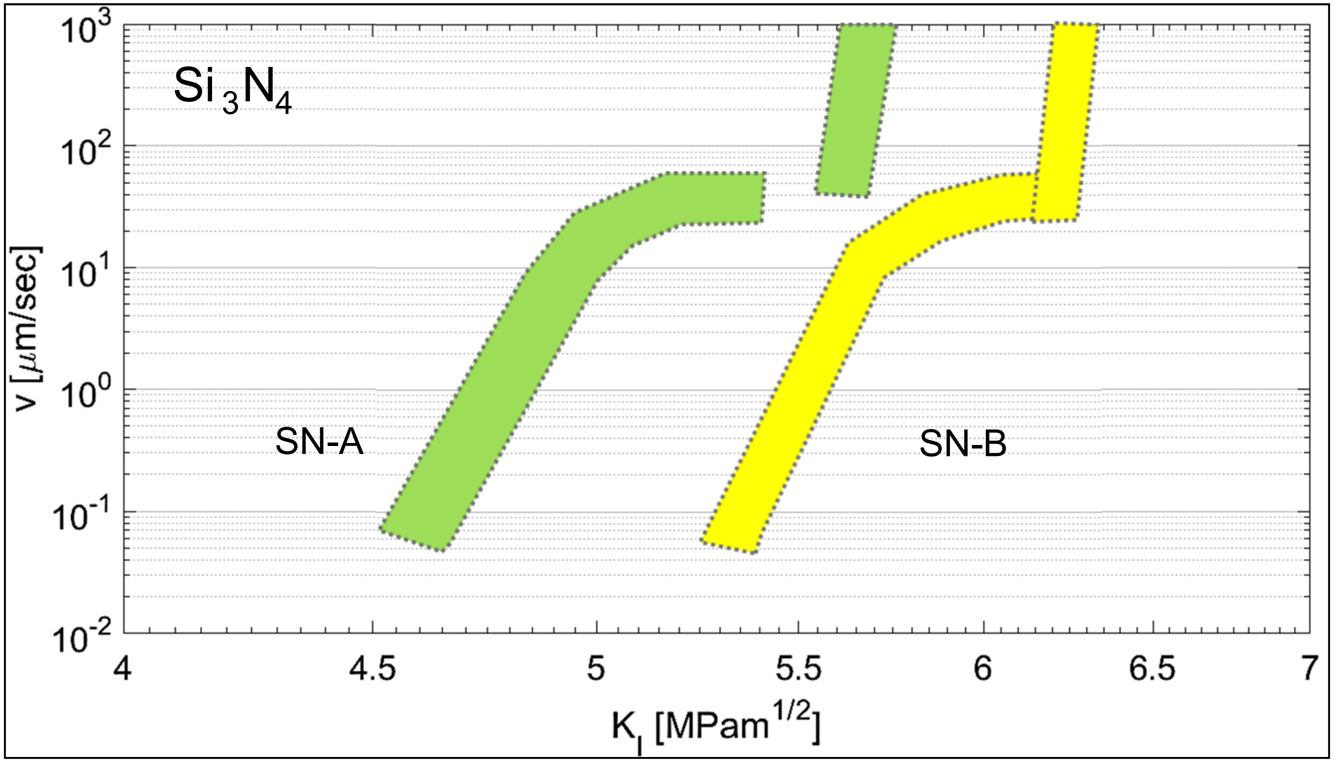
Different loading velocities





[6] J. Krivohlavek: Optimierung …, bachelor thesis, TUHH (2016)

Overview concerning subcritical crack growth in Si_3N_4

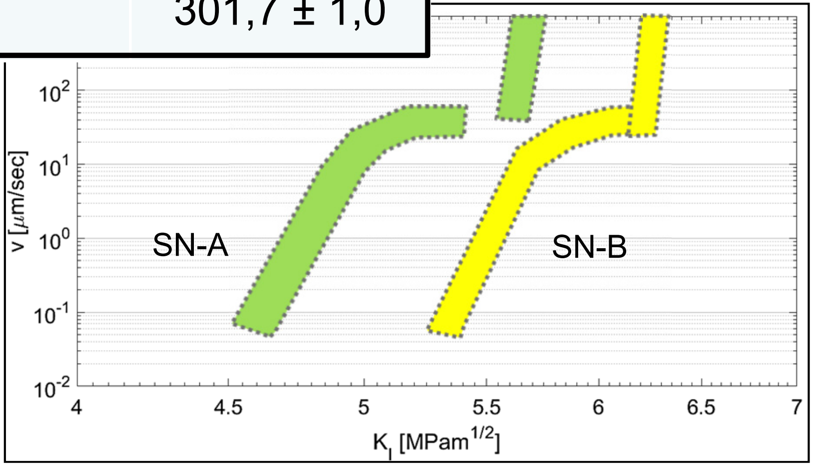


[6] J. Krivohlavek: Optimierung …, bachelor thesis, TUHH (2016)

4c. Measurements, v-K-curves (summary of data)

Material	SN-A	SN-B
Start value of R-curve [MPa√m]	2,5	4,2
Plateau value of R-Kurve [MPa√m]	4,88 ± 0,08	5,71 ± 0,05
Length of steep R-curve increase [µm]	70	100
Exponent *n* (subcrit. crack growth)	88 ± 13	86 ± 18
Plateau at velocity *v* [µm/s]	30	40
K_{IC} (maximum value) [MPa√m]	5,7	6,33
E, measured during loading [GPa]	315 ± 3	302,1 ± 1,8
E, measured during unloading [GPa]	„	301,7 ± 1,0

In principle, all of the data in the table of one material can be measured with a single bending specimen (of the dimensions 3 x 4 x 45 mm^3 or similar). The given data just provide an example of what can be measured.



[6] J. Krivohlavek: Optimierung …, bachelor thesis, TUHH (2016)

If subcritical crack growth exists, the measurement of an R-curve depends on the loading speed. This effect can be observed very well with glass. Higher loading rate means higher K_{IC} because of less time for subcritical crack advance.

On the other hand, the measurement of subcritical crack growth is dependent on how long the crack is, or, in other words, on the position on the R-curve, when subcritical crack growth is recorded.

Thus, the simultaneous measurements of R-curve and v-K-curve influence each other. However, a comparison of different material versions or an optimization of material parameters can be accomplished with this new method by testing different materials under exactly the same experimental conditions.

- **After optimization → Measurements without much exp. effort**
  - E-Module, relativ error approx. 1 %
  - R-curves with steep initial increase
  - v-K-curves, rapid measurement

  } 4-point-bending

- **Advantages**
  - Easy operation and control of stable crack growth
  - measurement of entire v-K-curves (plateau, K_{Ic})
  - Reliable determination of exponent *n* and other quantities
  - Simple specimen preparation (bending bars)

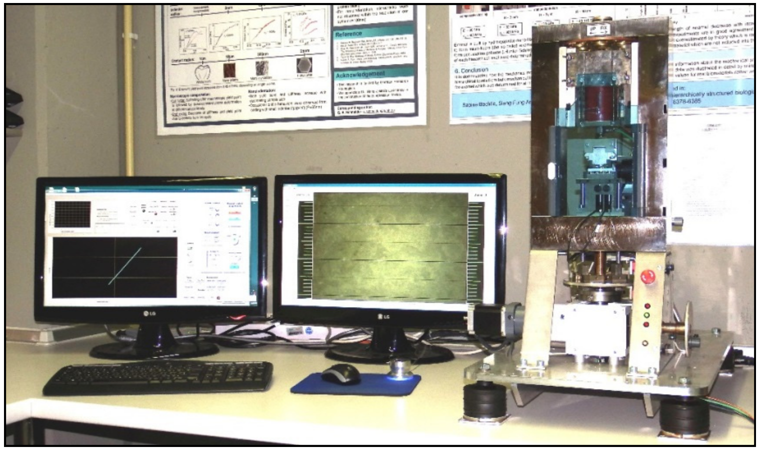
- **Problems**
  - Compliance measurement, manual correction necessary
  - Kind of correction depends on material.

Possible improvements

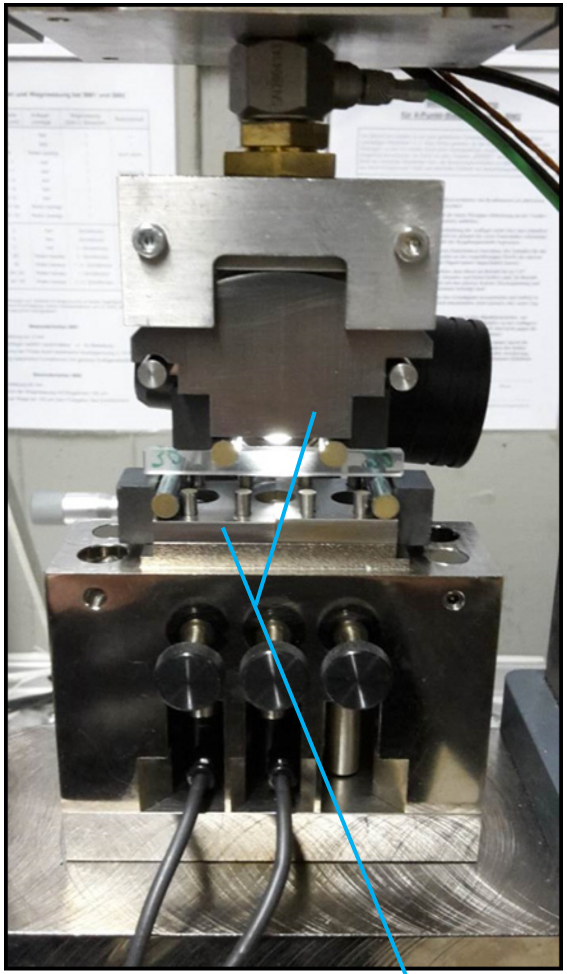
- ➔ Adaption of software (LabVIEW) → correct determination of compliance during the measurement
- ➔ Increase of frequency for data acquisition (real time system)

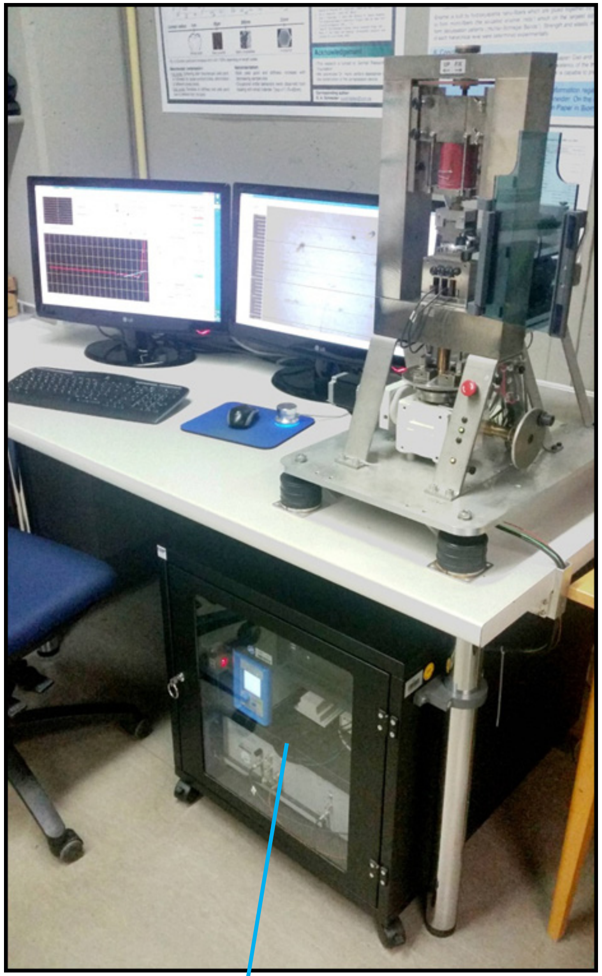
(Remark: Beside basic research we also complete industrial orders.)

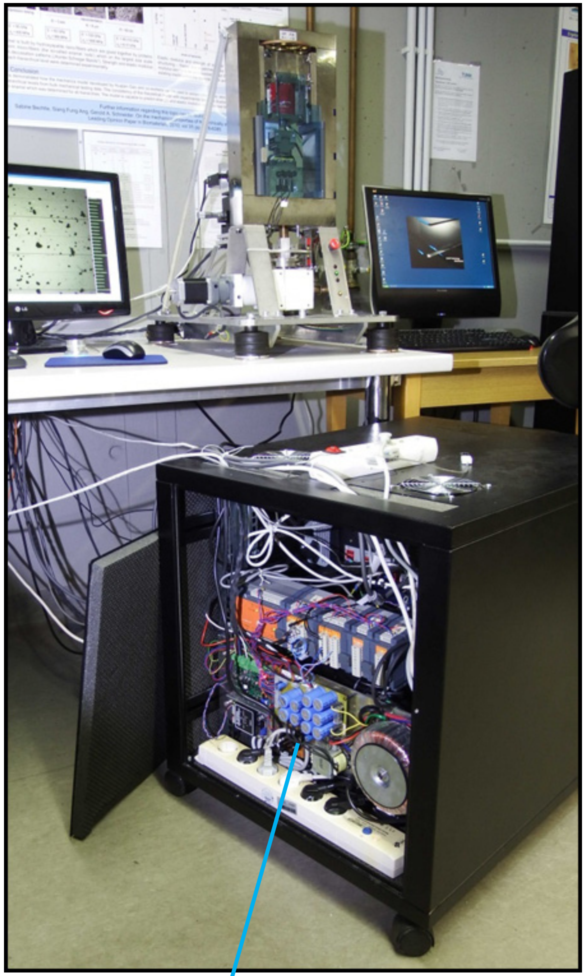
Many thanks for your attention!



Appendix – More about the experimental set-up







4-point-bending load cell

Rack with computer and electronics

The rack is turnable to access the electronics assembly from the back side.