A novel approach to determine the adhesion of hot dip galvanized coatings

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Zinc Coating Adhesion Testing
Standards and Guidelines

- **DIN EN ISO 10684, Appendix E and ASTM A123-17: „Cutting or loosening by means of a knife with considerable pressure”**
  - insufficient in terms of a reproducible adhesion test because there are no defined boundary conditions
- **ASTM A123-84 Impact Testing (Drop Hammer Testing, also IS 2629, JIS 401)**
  - Not suitable for HT-HDG, no correlation with adhesion strength can be derived due to dynamic loading, has been deleted in ASTM
- **DIN 50978 Hammer Testing:**
  - Withdrawn, see drop hammer test. However, it will continue to be listed in a modified form for liability testing in the guidelines of other associations (e.g. Nordic Galvanizers)
- **Forehead trigger test or metallography**
  - Too time-consuming for in-production testing

Status Quo

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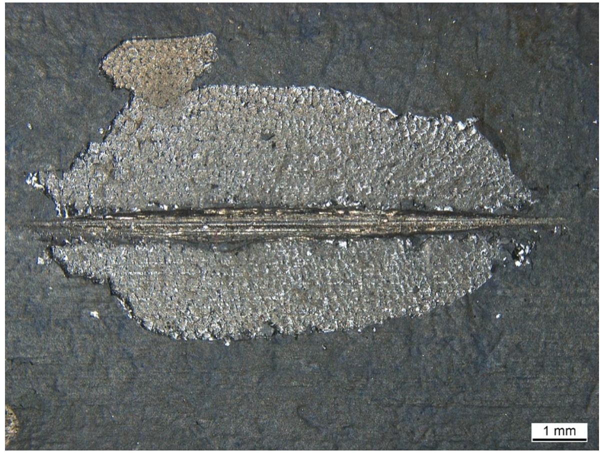
- **Almost 1% of the annual tonnage of hot-dip galvanized fasteners has adhesion problems**, of which around 40% of the adhesion problems are identified by the manufacturer and around 20% during assembly by end customers [1]
- **Currently, there are no suitable standardized test methods for a practical and reliable assessment of the adhesion strength** of hot-dip galvanizing coatings and the associated functional properties, especially during stresses during assembly and operation.
- **Hot-dip galvanizing companies and fastener manufacturers lack a tool** for standard-compliant testing of the quality of their products as well as for process monitoring
- **Existing test approaches are unsuitable for assessing the adhesion strength of zinc coatings**, as these are usually based on dynamic impact stress and therefore only allow statements to be made about the toughness of the coatings. A reliable correlation with the adhesion of the coatings and their function cannot be derived from this.

[1] Internal survey „Gemeinschaftsausschusses Verzinken", March 2019

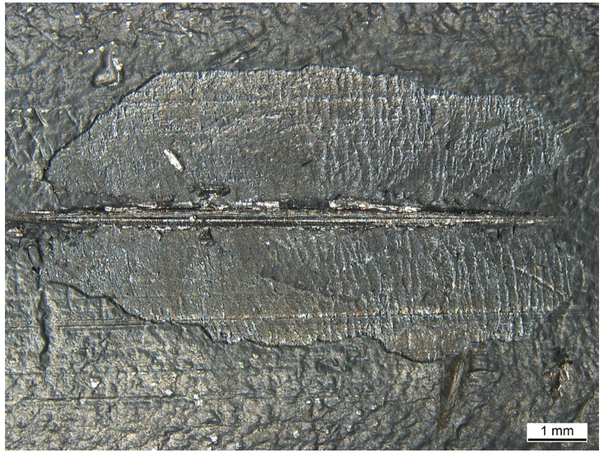
Status Quo

Impact test acc. DIN 50978 for HT-galvanized M16 nuts

OK-part



Not OK-part



- In the case of HT-galvanized nuts M16, it **was not possible to differentiate between components with and without adhesion problems** in the impact test according to DIN 50978, while in the assembly test both batches could be clearly differentiated due to the real stresses.

- An industrial field trial organized by the German Fastener Association (DSV e.V.) with three proprietary test methods (based on cross-cut, bolting and impact test) - carried out by bolt manufacturers and galvanizing companies - showed no correlation with regard to the practical findings [2]

[2] Field trial of Deutscher Schraubenverband, internal, Protocol of 03.07.2019

Testing zinc coating adhesion

Survey in galvanizing plants

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Where do problems arise?

- Focus on HT galvanizing
- for NT galvanizing for heavy components and large layer thicknesses
- when sweeping, partly due to the Kirkendall effect

Test methods used

- no test of adhesion strength
- Impact test according to DIN 50978
- Hitting components against each other
- Cross cut

Objective

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Development of a robust, easy-to-use and meaningful method for testing the adhesion strength of hot-dip galvanizing coatings on fasteners

- **Preparation of a requirement specification** with regard to the adhesion requirements for hot-dip galvanizing coatings, taking into account the stresses during assembly and operation
- **Practical screening of existing methods for the adhesion test** of coatings and zinc coatings in general as well as proprietary methods for adhesion testing for hot-dip galvanizing coatings practiced in the factories
- **Development of practical test methods**, implementation of the specifications
- **Ensuring practical applicability** for fasteners (bolt, nut, washer) of various dimensions
- **Transfer** of the developed method in standards / guidelines, e.g. ISO 10684

Test Material

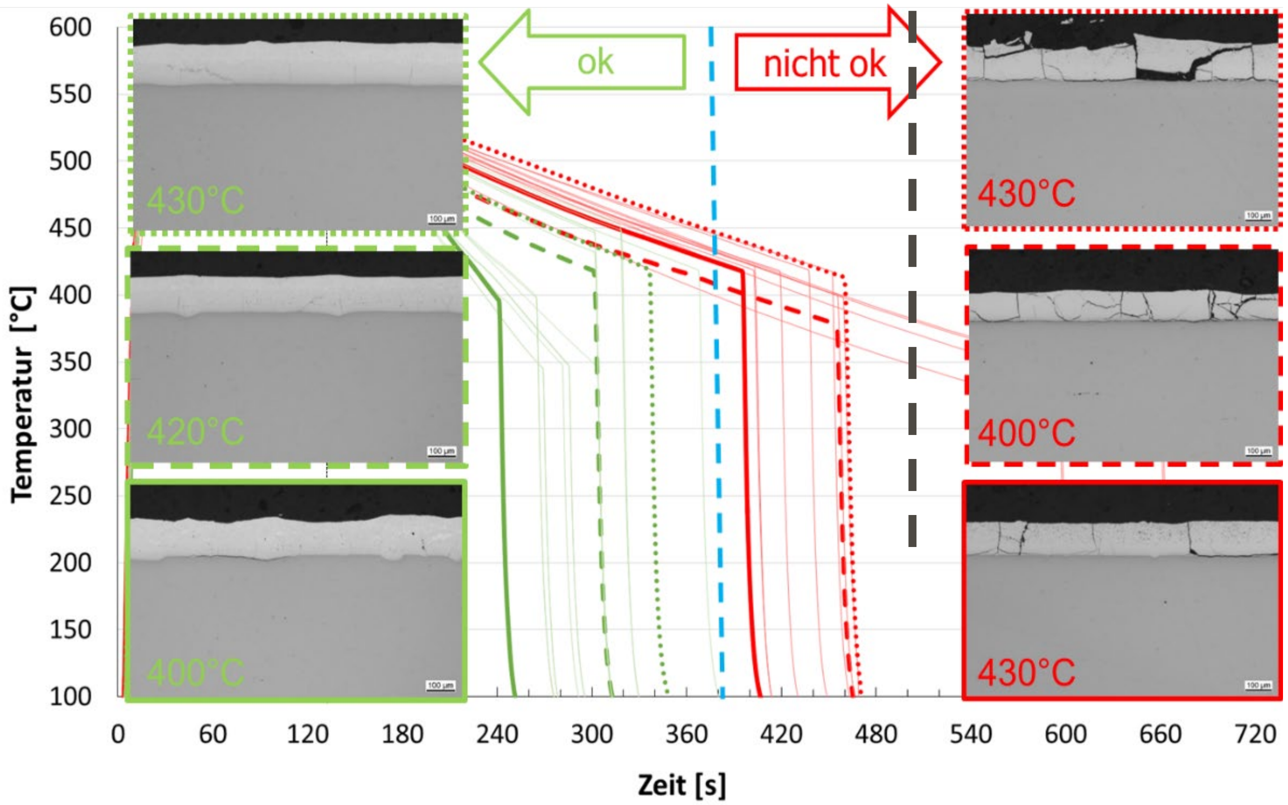
Production of ok and not ok HDG Coatings

Focus on high-temperature HDG
Normal temperature galvanizing as control tests

Galvanizing in industrial plants:

	Bolt	Nut	Washer	Σ
HT-HDG ok	150	150	150	450
HT-HDG not ok	50	50	50	150
Σ	200	200	200	600

Delay of the last quenching step by approx. 8 min is said to produce poorly adhering layers

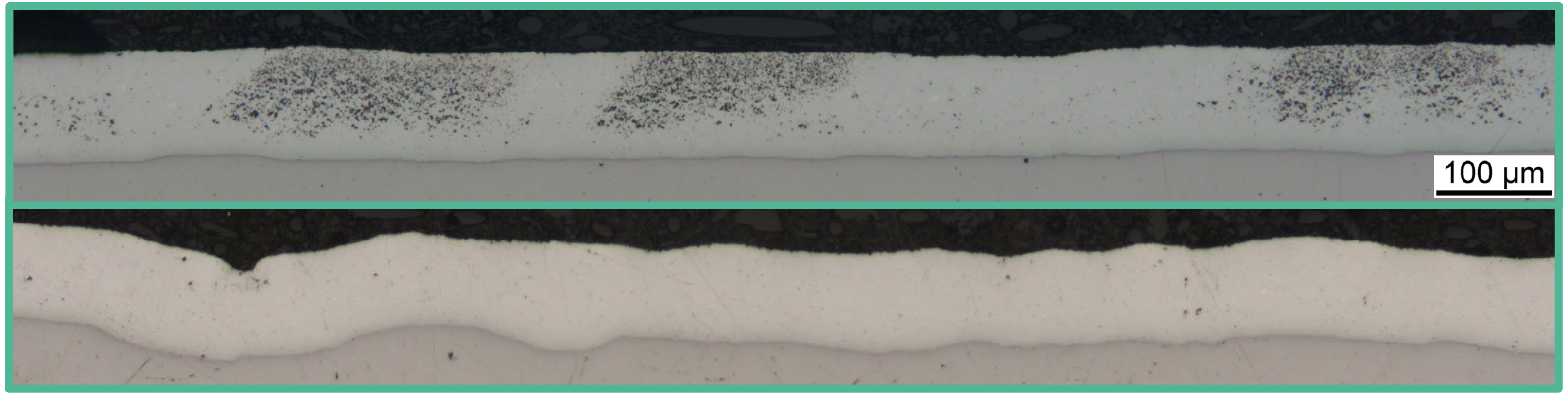


[3] H. Hoche, M. Lander, M. Oechsner, S. Six, S. Friedrich: Examination of Influencing Factors on the Coating Quality of Hot Dip Galvanized High Strength Fasteners, Conference Proceedings Intergalva 17 – 22 June 2018, Berlin, 2018

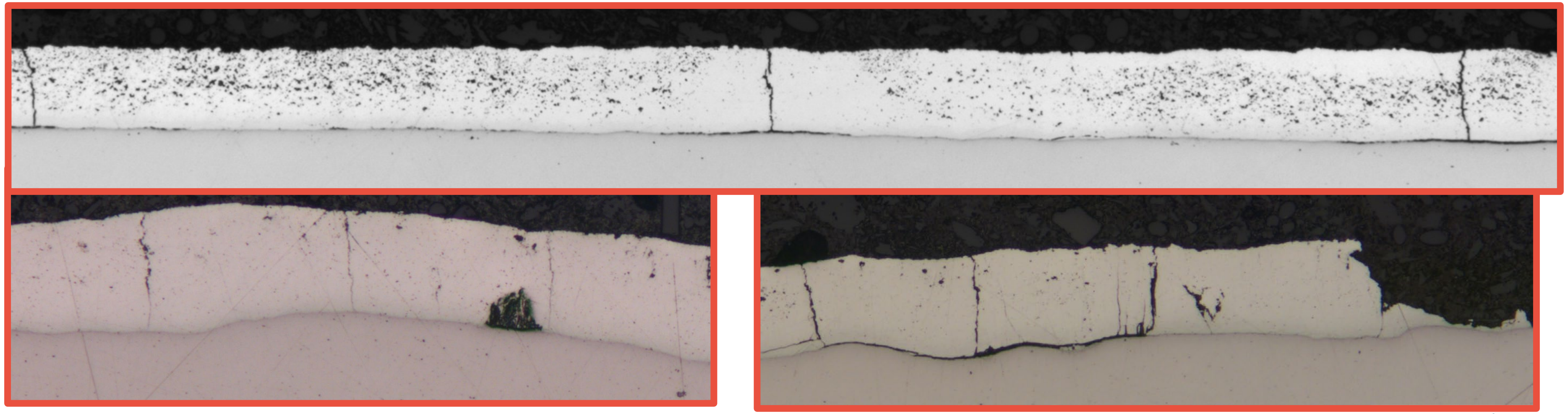
Validation of Test Material

Microspecimens

ok



not ok



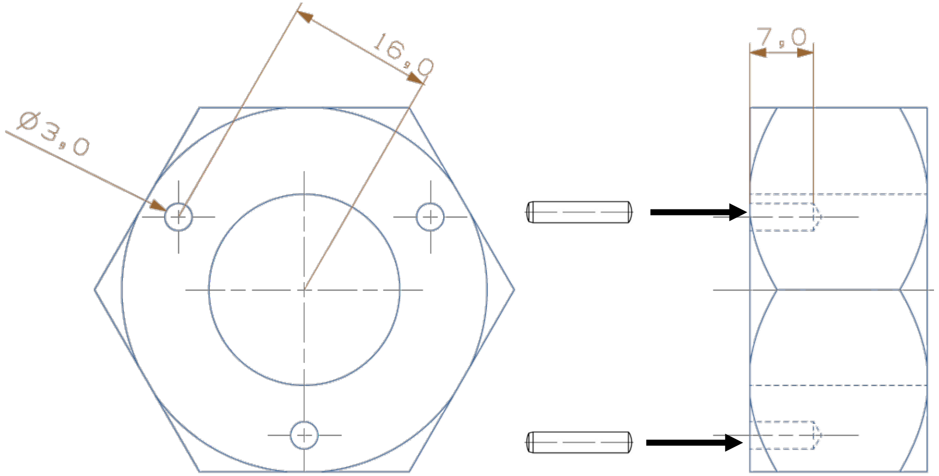
Simulation of the installation conditions

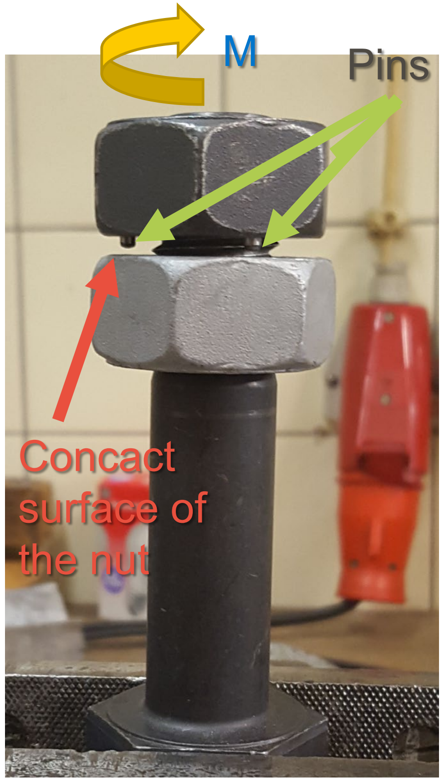
Step 1: Development of a test method

Approach: Compression-shear stress with increasing pressure

Implementation:

- Force transmission via cylindrical pins (DIN 7)
- Increasing stress by tightening the prepared nut
- Increase in stress by tightening the nut





Simulation of the installation conditions

Differentiation ok / not ok is possible !

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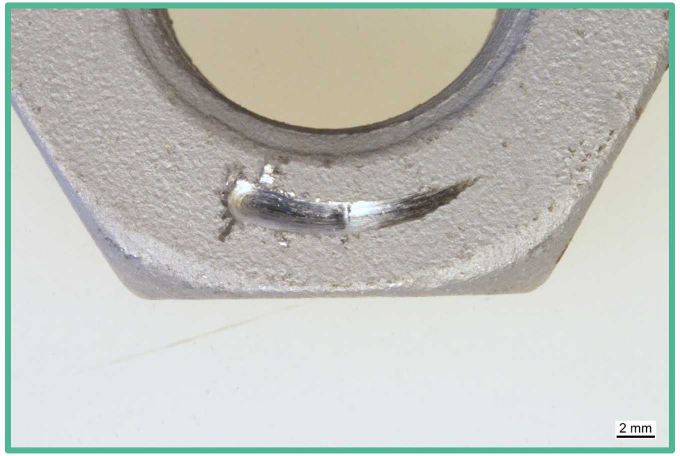
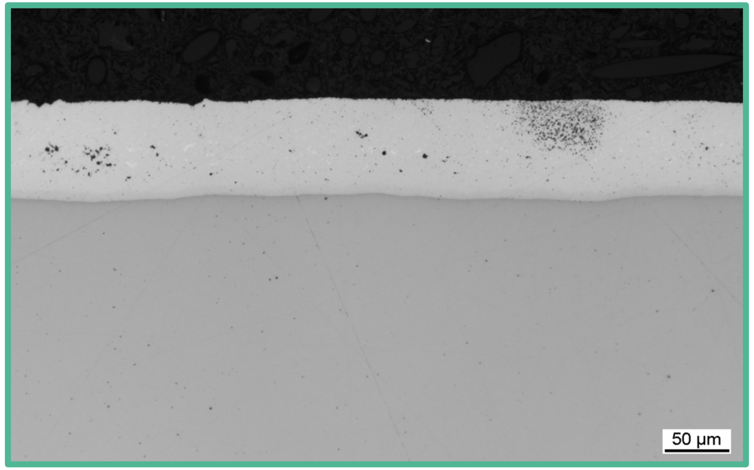
ok





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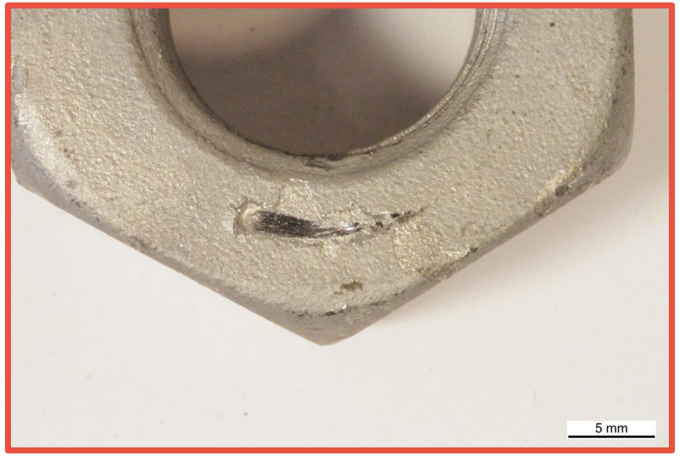

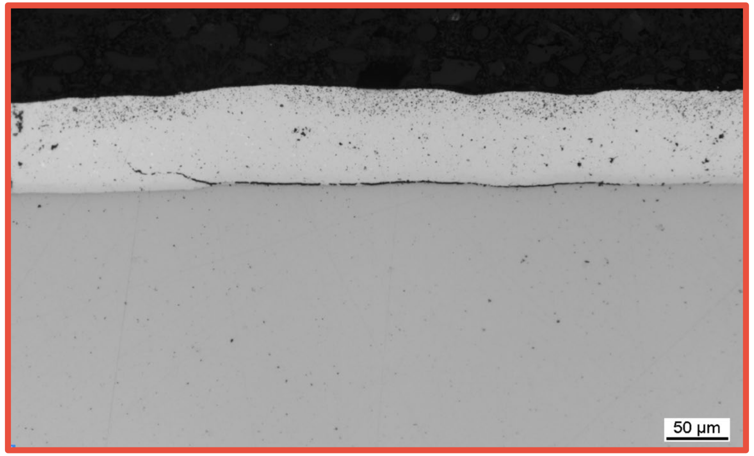

Simulation of the installation conditions

Validation by Microspecimens

- **Differentiation between ok and not ok is possible**
- Evaluation criteria:
  - At least two spalling-free test positions out of three tests
  - Morphology of flaking

- Influencing factors
  - Jerky tigthening
  - Not reproducible gluing of the washers; tilt of the washer results in uneven load by the three pins
  - Solution: Glue replaced by sandpaper

Nuts	
23	96%
0	0%
1	4%

Bolts	
12	100%
0	0%
0	0%

Washer (Glue)	
15	83%
3	17%
0	0%

Washer (Sandpaper)	
6	100%
0	0%
0	0%

Simulation of the installation conditions
Step 2: Finding thresholds for the test parameters

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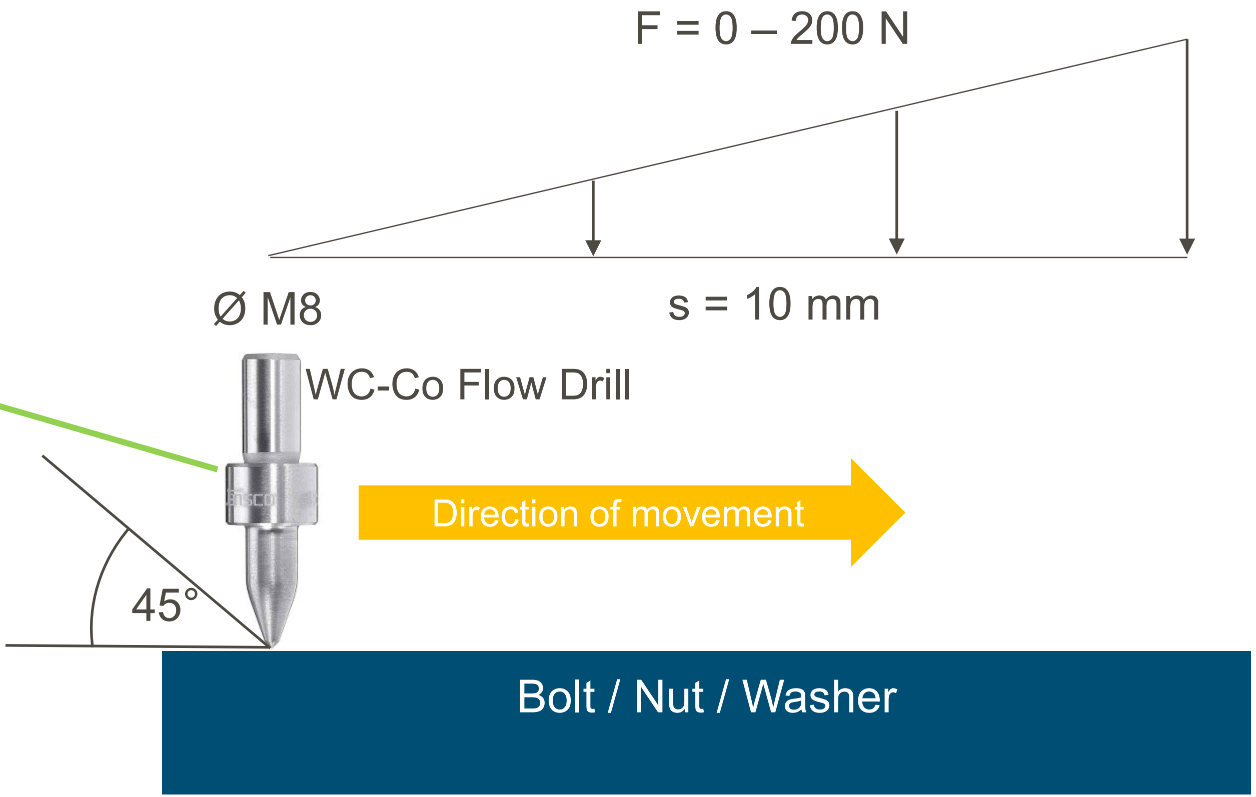

Scratch Tester (DIN EN ISO 20502:2016-11):
- Variation of the Load / Load thresholds
- No jerky tightening
- Wear resistant indenters

Finding thresholds for the test parameters
Washer / Bolt

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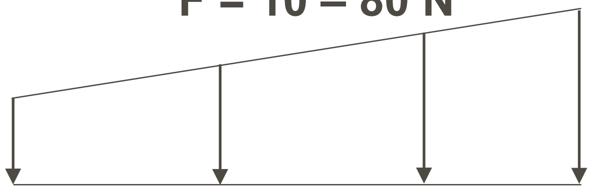
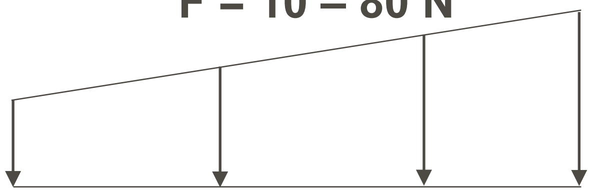
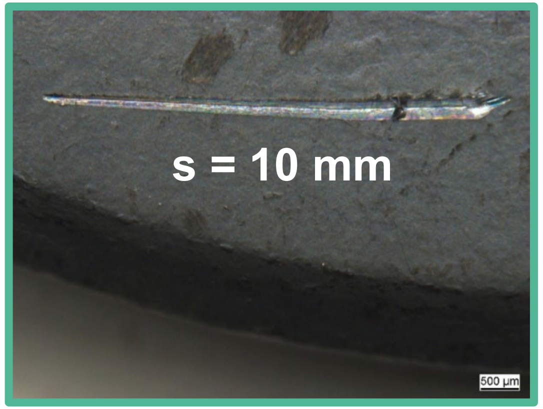
Washer

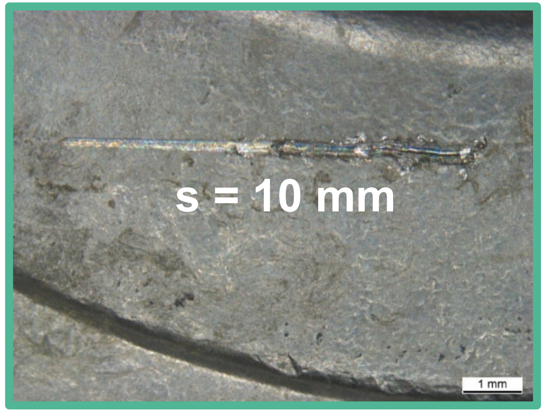


Bolt

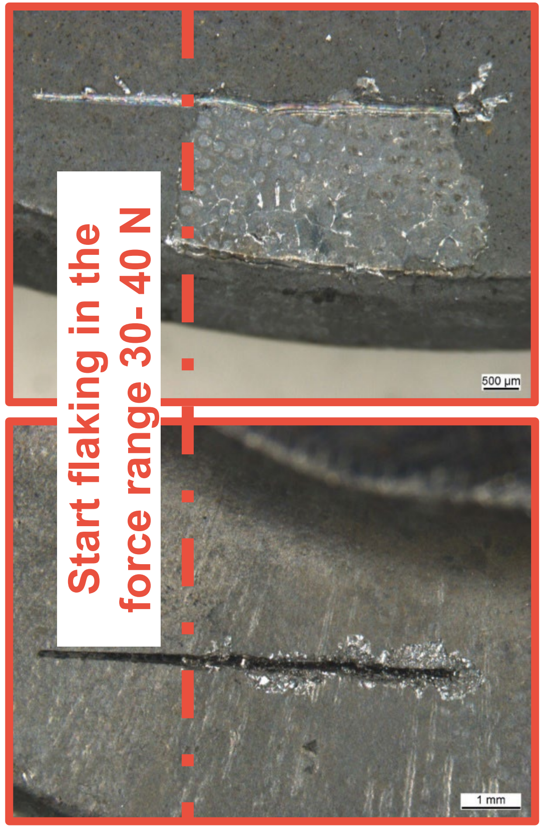


OK





Not OK



Finding thresholds for the test parameters

Nut

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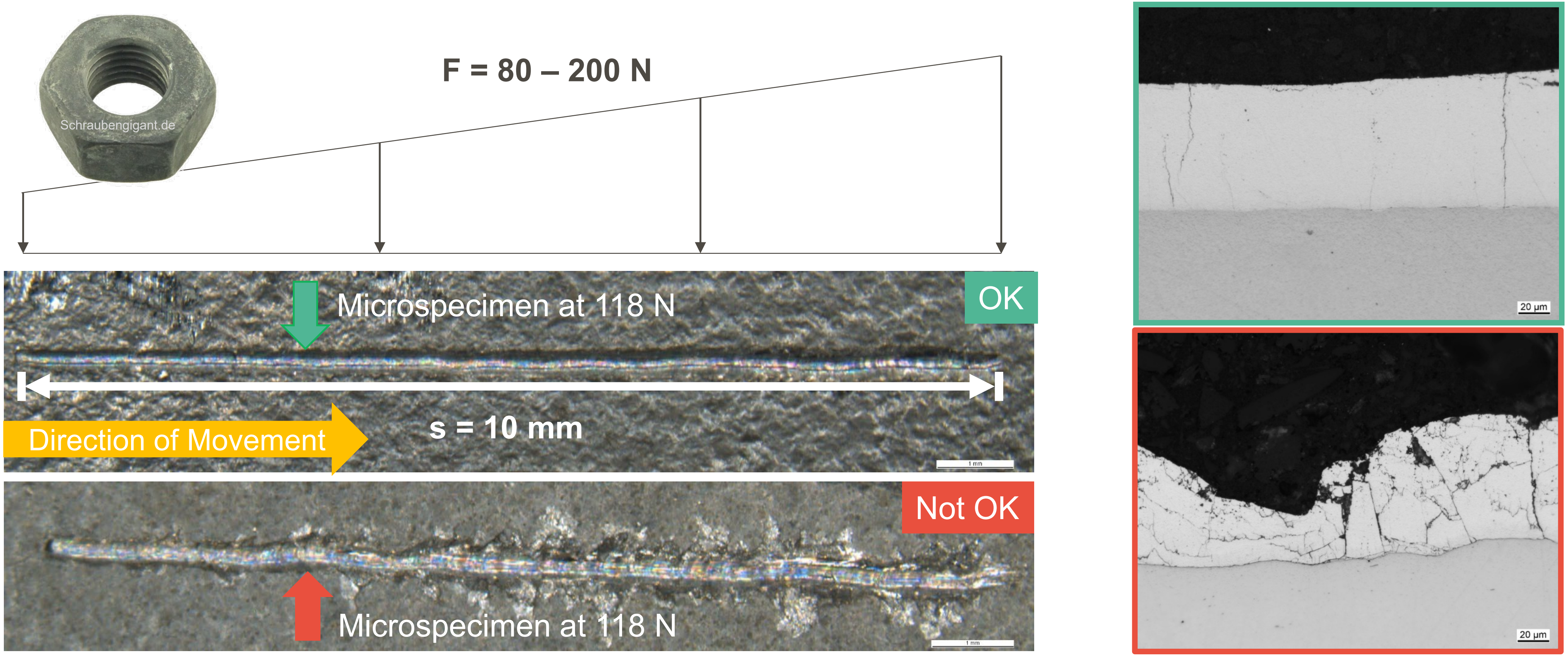

Apperance of not ok HDG adhesion

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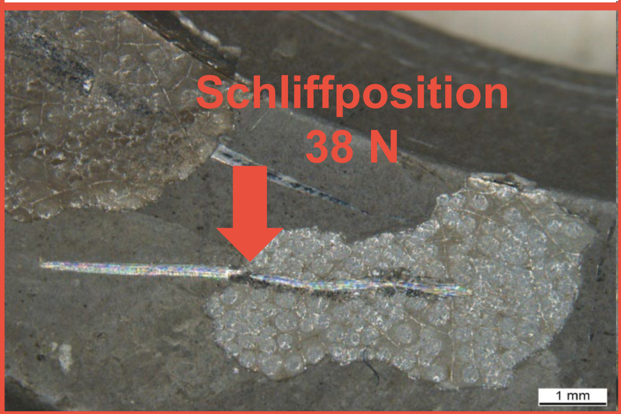
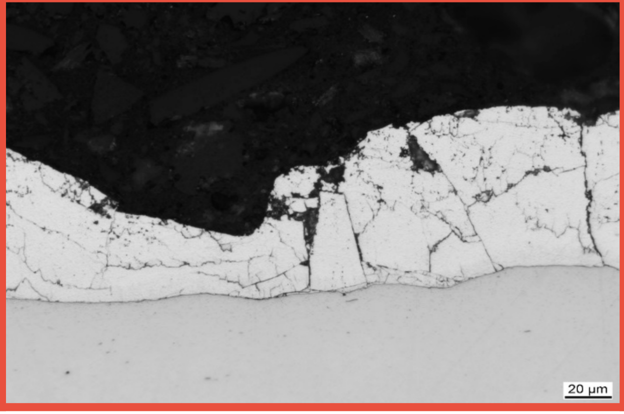
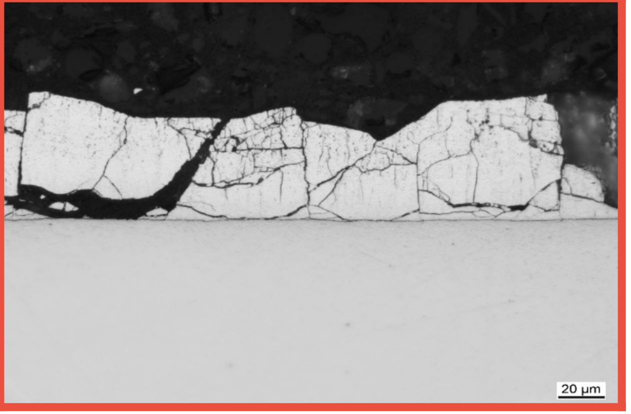
<table>
<tr><th></th><th>Nut</th><th>Washer</th><th>Bolt</th></tr>
<tr><td>Macroscopy</td><td></td><td></td><td></td></tr>
<tr><td>Microspecimen</td><td></td><td></td><td></td></tr>
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Influence of roughness and layer thickness

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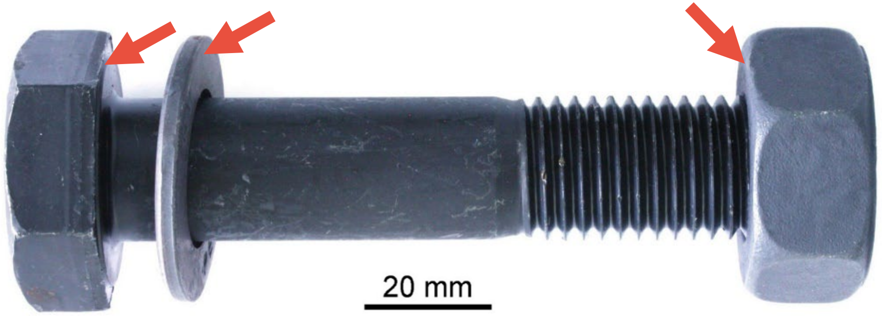
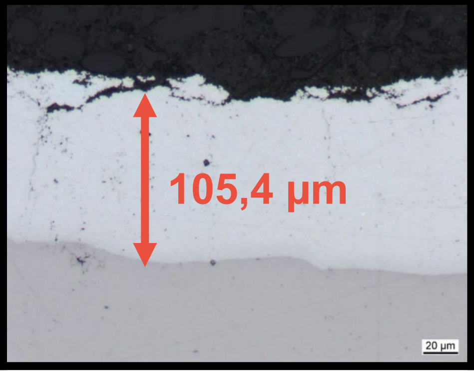
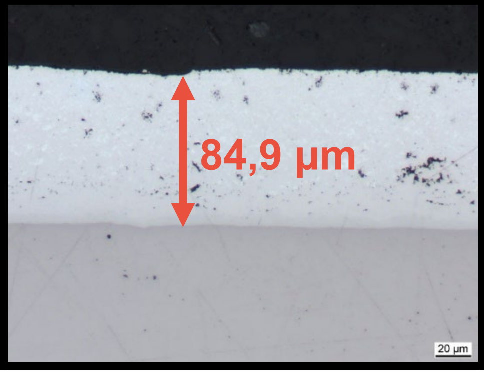
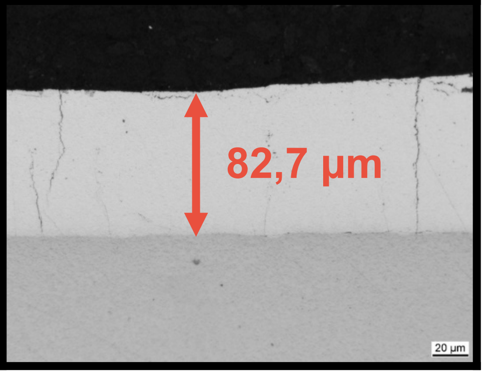
	R_z
Nut	9,5 µm
Washer	6,4 µm
Bolt	5,9 µm

Measurement of roughness on the uncoated bolt



	Nut	Washer	Bolt
Schichtdicke			

Increasing the critical load, which leads to layer flaking, due to higher layer thicknesses of the nut

Is there wear at the flow drill tip?

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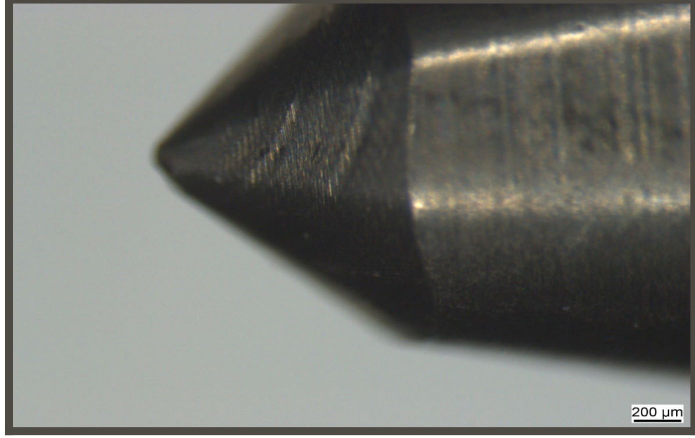
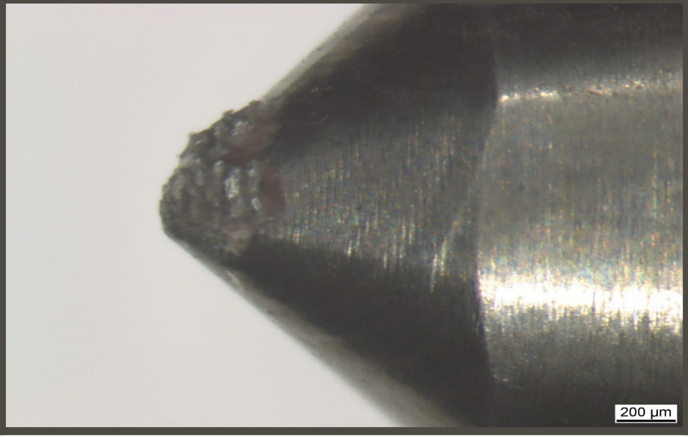
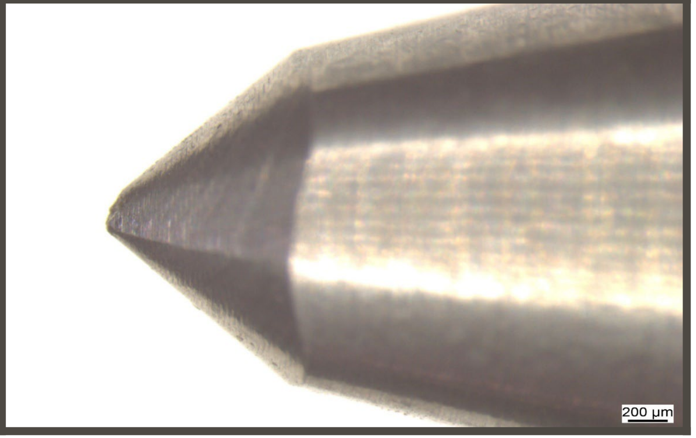
New	50 Scratches	200 Scratches
		

Even after 200 scratches, no significant signs of wear at the tip of the flow drill could be detected with the stereo microscope

Requirements for the test device

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Objective:
Evaluation of Zn coating adhesion

Nut

Variable sizes

- Ok → No flaking ≤ 200 N
- Not ok → Start of flaking ≥ 110 N

Washer

Variable sizes

- ok → No flaking ≤ 80 N
- not ok → Start of flaking ≥ 30 N

Bolt

Variable sizes

- ok → No flaking ≤ 80 N
- not ok → Start of flaking ≥ 30 N

Test device

Schematic drawing of the test device

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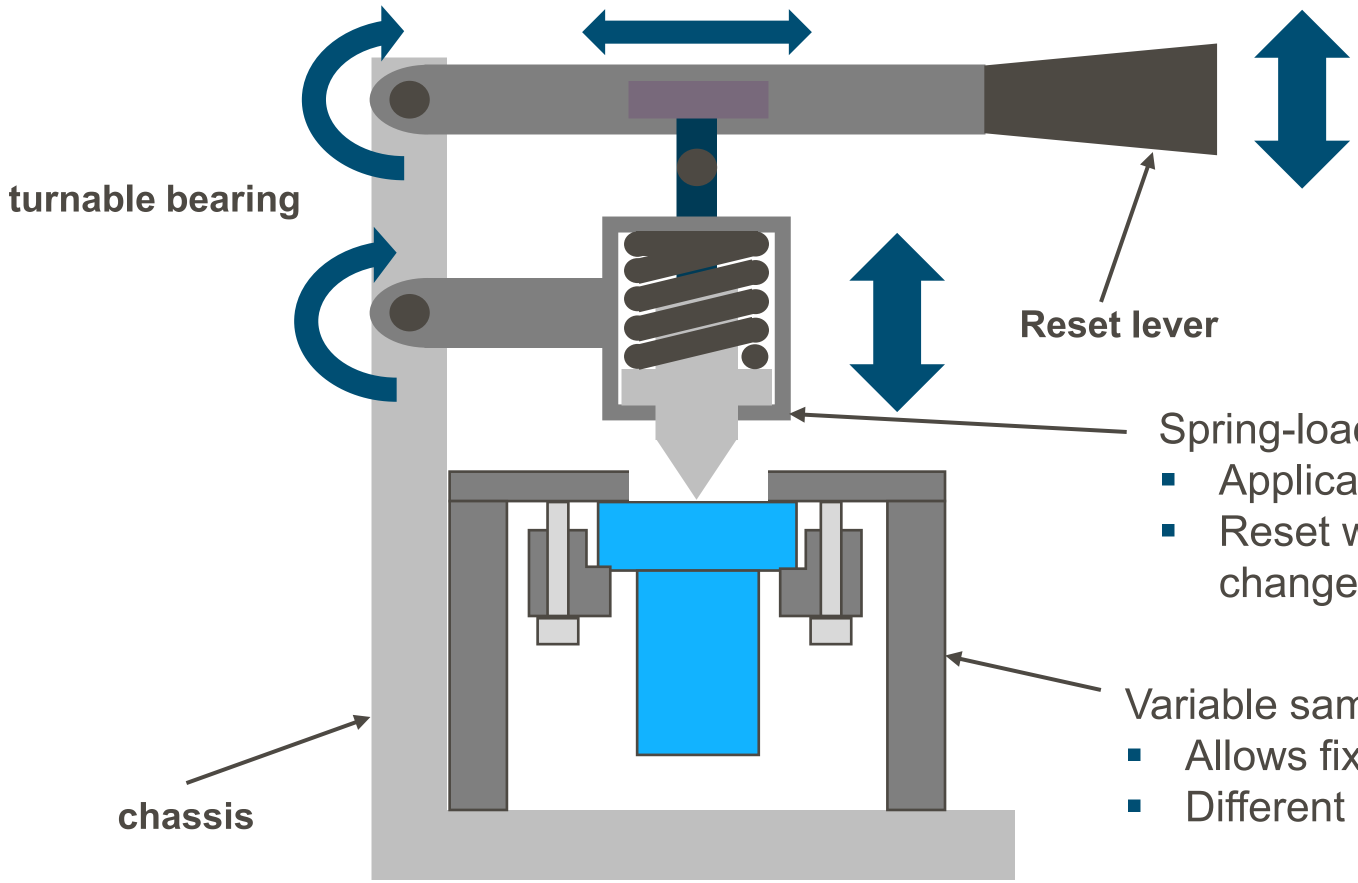

Spring-loaded WC-Co tip
- Application of the test force
- Reset with reset lever simplifies sample changes

Variable sample holder
- Allows fixation of washers, nuts and bolts
- Different sizes

3D modell of the prototype

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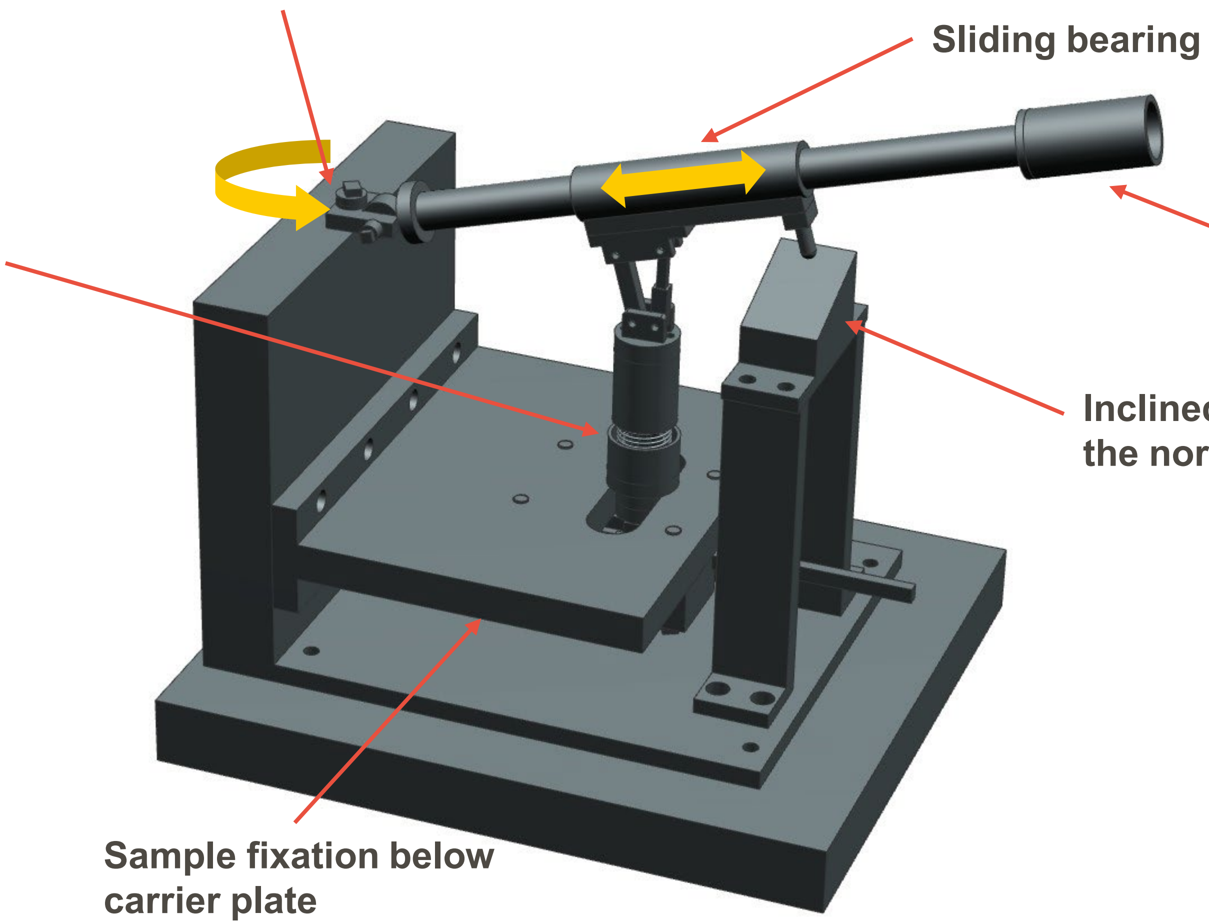


$$\boxed{F_D = D_f \cdot s}$$

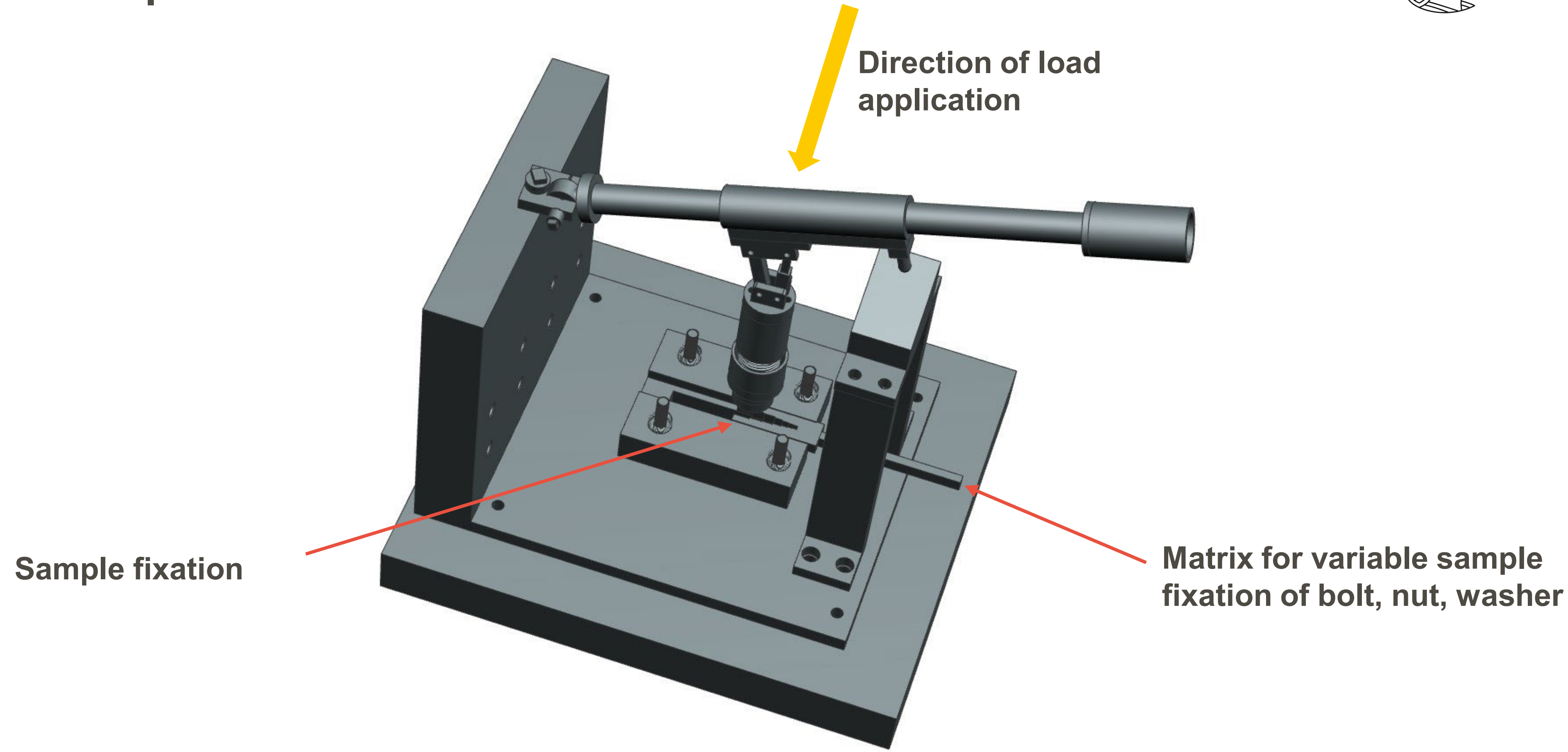
F_D: 40 N (bolt, washer)/ 100 N (nut)

D_f: 900 $\frac{N}{m}$

$$s = \frac{F_D}{D_f}$$

3D model of the prototype

Without carrier plate



Current actions & outlook

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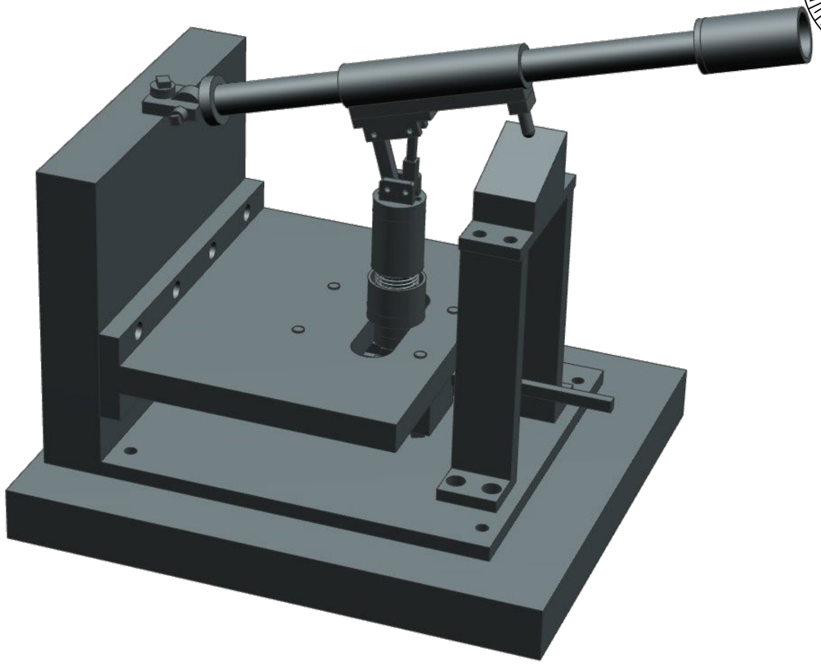
- Procurement of the material
- Refinement of the final design with project board
- Manufacturing of the prototype
- Collecting practical experience in industry
- Ongoing validation and optimization



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