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Full-field strain measurements for microstructurally small fatigue crack propagation using digital image correlation method

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Dear Editor,

Hereby we submit the manuscript entitled “Full-field strain measurements for microstructurally small fatigue crack propagation using DIC method”.

The work is focused on experimental approach for investigation of microstructurally short fatigue crack propagation in polycrystalline alloy with body-centered cubic crystal lattice. Full-field investigation takes into consideration the connection of the strain field distribution, microstructure, crack growth rate, crack path and fracture behavior that provides unique knowledge of the microstructurally short fatigue crack growth mechanism. Such experimental procedure and obtained results can provide additional knowledge for an appropriate prediction of the microstructurally short fatigue crack propagation in other polycrystalline alloys.

We believe this work can attract interest of the journal`s readers and viewers.

On behalf of all co/authors

Sincerely Yours,

Evgenii Malitckii

TITLE:

Full-field Strain Measurements for Microstructurally Small Fatigue Crack Propagation using Digital Image Correlation Method

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KEYWORDS:

Digital image correlation, small fatigue crack, crack growth rate retardation, sub grain level, shear strain localization, strain inhomogeneity.

SUMMARY:

Microstructurally small fatigue crack growth behavior is investigated using a novel methodological approach combining crack growth rate measurement and strain-field analysis to reveal the cumulative deformation field at sub-grain level.

ABSTRACT:

A novel measurement approach is used to reveal the cumulative deformation field at a sub-grain level and to study the influence of microstructure on the growth of microstructurally small fatigue cracks. The proposed strain field analysis methodology is based on the use of a unique patterning technique with a characteristic speckle size of approximately 10 μm . The developed methodology is applied to study the small fatigue crack behavior in body centered cubic (bcc) Fe-Cr ferritic stainless steel with a relatively large grain size allowing a high spatial measurement accuracy at the sub-grain level. This methodology allows the measurement of SFC growth retardation events and associated intermittent shear strain localization zones ahead of the crack tip. In addition, this can be correlated with the grain orientation and size. Thus, the developed methodology can provide a deeper fundamental understanding of the small fatigue crack growth behavior, required for the development of robust theoretical models for the SFC propagation in polycrystalline materials.

INTRODUCTION:

New lightweight solutions are required to improve the energy efficiency of vehicles such as ships. Weight reduction of large steel structures is possible using advanced steel materials. The efficient utilization of new material and the lightweight solution requires high manufacturing quality and robust design methods^{[1],[2]}. A robust design method means structural analysis under realistic loading conditions, such as wave-induced loading in the case of a cruise ship, as well as response

calculations to define deformation and stresses. The allowed stress level is defined based on the strength of critical structural details. In the case of large structures, these are typically welded joints with an inhomogeneous microstructure. One of the key design challenges for new lightweight solutions is fatigue due to its cumulative and localized nature, often taking place at weld notches. For high manufacturing quality, the fatigue behavior is dominated by small fatigue crack (SFC) growth since manufacturing induced defects are very small^{[1][3]}. Thus, the fundamental understanding of small fatigue crack growth in metallic materials is crucial for sustainable use of new steels in high performance structures.

The effective modeling of such a complicated process as fatigue crack propagation in polycrystalline metallic materials is impossible without a clear understanding of the physical processes accompanying the fatigue fracture mechanism. A significant effort from the research community has been focused on investigating fatigue crack propagation using visual observation and statistical analysis. So far, small fatigue crack growth behavior has mainly been investigated by theoretical methods due to the limitations of experimental techniques. The anomalous fatigue crack growth rate retardation for SFCs is usually associated with the grain boundaries (GB)^{4,5,6,7,8,9}. However, the reasons for anomalous SFC growth are still under discussion. The results obtained by theoretical modelling using a discrete dislocation method shows formation of a dislocation wall, or a short low-angle grain boundary caused by dislocations emitted from the fatigue crack tip affecting the fatigue crack growth rate^{10,11,12,13}. Until recently, there has been a challenge in accurate experimental analysis of the small fatigue crack growth behavior. Experimental observations are required for the development of physical principles based computational models.

For analysis of cyclic material deformation behavior at micro-scale it is desirable to have full-field deformation measurements that can be carried out in situ during cyclic loading using standard mechanical testing equipment, with spatial resolution at least an order of magnitude below the characteristic length scale of the microstructure. In order to understand the variations in fatigue crack growth rate, measured strain fields are often linked to electron backscatter diffraction (EBSD) measurements of material microstructure. Carrol et al.¹⁴ provide a quantitative, full-field ex situ measurement of plastic strain near a growing long fatigue crack in a nickel-based super alloy, showing the formation of asymmetric lobes in the plastic wake of the propagating fatigue crack. At higher magnification, electron microscopy digital image correlation (DIC) revealed strain inhomogeneities associated with strain localization on the slip bands, with twin and grain boundaries affecting the fatigue crack growth behavior. However, the used ex situ measurement approach is not able to capture the strain field during fatigue crack propagation. An experimental study of plastic blunting during long fatigue crack propagation was performed by Peralta¹⁵ using in situ DIC for commercial purity Ni (99.6%). Results revealed that the accumulation of plastic deformation was dominated by shear along the slip bands that extended ahead of the crack and were inclined with respect to the crack growth direction. The observed strain localization at the slip bands is probably caused by overloading, since the low stress intensity factor values result in a mixed nature of the deformation (shear and normal strain)^{14,15}. A heterogeneous strain field distribution at the sub-grain level has been observed for coarse grained aluminum alloy¹⁶ and

duplex steel¹⁷, where the activation of the dislocation slip systems was associated with Schmid's law^{16,17}.

A recent study performed by Malitckii¹⁸ manifests that anomalous SFC growth behavior is controlled by strain inhomogeneities related to the grain structure or, in particular, by accumulation of shear strain localization zones ahead of the crack. With high-quality micro-scale patterns and grains larger than 100 μm , optical microscopy DIC enabled in situ sub-grain deformation measurements for the first time. However, in Malitckii¹⁸, the novel methodology applied to measure plastic strain field in situ over hundreds of thousands of load cycles was not presented or discussed in detail. Therefore, the objective of this paper is to introduce this new experimental approach for studying small fatigue crack growth behavior in polycrystalline materials in the high cycle regime. The novelty of the approach consists of in situ full-field strain measurement using a unique pattern technique, in addition to crack growth rate measurement. Because this method uses optical image sensors it enables capturing thousands of frames during the fatigue test. Electron backscatter diffraction (EBSD) is used for microstructural characterization and combined with DIC measurements to reveal the impact of grain boundaries on small fatigue crack growth retardation¹⁸. The approach is applied for the measurement of small fatigue crack propagation in bcc 18%Cr ferritic stainless steel¹⁸ simulating the behavior of the structural steel for large structural applications. In this paper, we explain the main steps of the measurement procedure and provide a summary discussion of the main finding.

PROTOCOL:

1. Specimen preparation and annealing

1.1. Mill the original ferritic stainless-steel plates with a thickness of 3 mm (see **Table of Materials**) to form the plate with characteristic size of about 200 mm x 15 mm x 1 mm.

1.2. Place the produced steel plate into the quartz tube and pump (see **Table of Materials**) it until the pressure of about 10^{-6} mbar.

1.3. Provide argon gas (see **Table of Materials**) into the quartz tube until the pressure reaches about 0.2 mbar.

1.4. Seal the quartz tube with the specimen inside by heating the quartz tube up to the melting temperature¹⁹.

CAUTION: The sealing procedure is hazardous. Use appropriate precautions such as proper eye protection, etc.²⁰.

1.5. Anneal the steel plate sealed inside of the quartz tube using the chamber furnace (see table of materials) at temperature of 1200 °C for 1 h and quench in water.

NOTE: The annealing procedure increases the average grain size of the studied steel up to 349 μm without extensive formation of chromium carbide particles²¹.

CAUTION: The annealing procedure is hazardous. Use appropriate precautions and follow the instructions of the chamber furnace manual.

1.6. Cut notched specimens (with thickness of 1 mm) from the annealed plate of the studied ferritic steel using electrical discharge machining (EDM, see **Table of Materials**). The scheme of the specimen is shown in **Figure 1**.

CAUTION: The EDM cutting procedure is hazardous. Use appropriate precautions and follow the instructions of the EDM manual.

1.7. Grind and polish the specimen surface.

1.8. Grind the specimen surfaces using grinding machine with emery paper (**Table of Materials**) until the surface of the specimen is uniform.

1.9. Polish the specimen surfaces using the polishing machine with 3 μm and 1 μm diamond paste (see **Table of Materials**) for 10 min each.

1.10. Polish the specimen surface using 0.02 μm colloidal silica vibratory polishing (see table of materials) for about 4 h; this is required for EBSD analysis.

2. Fatigue pre-cracking

2.1. Experimentally define the displacement controlled fatigue test parameters.

2.1.1. Adjust the displacement limits ϵ_{\min} and ϵ_{\max} of the servo hydraulic machine (see **Table of Materials**) so that the σ_{\min} and σ_{\max} are in range of about -50 MPa and 300 MPa, respectively.

CAUTION: The servo hydraulic machine is hazardous. Use appropriate precautions and follow the instructions of the servo hydraulic machine manual.

2.1.2. Examine the initial crack formation after 2,000, 5,000 and 10,000 cycles using optical microscopy (see **Table of Materials**) to define the optimal number of fatigue cycles and avoid extensive crack growth.

2.2. Subject the specimen to displacement controlled uniaxial cyclic loading for defined amount of cycles.

2.3. Examine the initial crack formation after defined amount of cycles using optical microscopy. Initial cracks with lengths up to about 20 μm are produced at the notch tip.

2.4. Increase the number of the fatigue loading cycles if the initial crack was not produced.

2.5. Replace the specimen if the initial crack length exceeds 50 μm .

3. Microstructural characterization

3.1. Clean the pre-cracked specimen.

3.1.1. Clean the pre-cracked specimen with acetone for 20 min using the ultrasonic bath (see **Table of Materials**).

3.1.2. Clean the pre-cracked specimen with ethanol for 20 min using the ultrasonic bath (see **Table of Materials**).

3.2. Mark the studied area using Vickers microindentations as shown in **Figure 2a**.

3.2.1. Follow the instructions of the Vickers microindenter (see **Table of Materials**) to perform the microindentation marks.

3.2.2. Insert the specimen into the micro Vickers hardness tester (see **Table of Materials**).

3.2.3. Set the indentation force at 500 N.

3.2.4. Adjust the position for the first Vickers indentation mark at about 500 μm sideways from the notch tip. Prepare the second indentation at another side.

3.2.5. Adjust the position for the third indentation mark at about 500 μm sideways and about 400 μm away from the notch tip.

3.3. Analyze the microstructure of the steel from the side surface of the specimen in the vicinity of the notch using electron backscatter diffraction (EBSD) analysis (see **Table of Materials**).

3.3.1. Follow the instruction manual of scanning electron microscope to perform EBSD analysis.

3.3.2. Set the magnification at 200x.

3.3.3. Adjust the position of the specimen under EBSD detector. Ensure that the notch tip and three Vickers microindentation marks are within the framework of the EBSD scanning (see **Figure 2b**).

3.3.4. Set the step size of the EBSD scanning at 2 μm . Scanning duration is about 1 h.

4. Decoration with a pattern

4.1. Clean the specimen surface with ethanol (see **Table of Materials**) for 10 min using the ultrasonic bath.

4.2. Dry the specimen using a fan.

4.3. Clean a microscope slide using a paper napkin soaked with ethanol (see **Table of Materials**).

4.4. Deposit a thin layer of ink on the glass surface of the microscope slide. A permanent marker provides uniform layer of ink on the glass surface by hand.

4.5. Press down on the silicone stamp with the pattern on the glass surface to transfer a layer of ink to the stamp surface.

4.6. Press down on the silicone stamp covered with the ink on the specimen surface.

4.7. Check the speckle pattern quality using optical microscopy. An example of the speckle pattern is shown in **Figure 3**. See references^{22,23} for details of pattern and microcontact printing.

4.8. Ensure that the speckle pattern size is at least 10 times smaller than the grain size of the studied material.

NOTE: Perform the steps 2, 3 and 4 in sufficient time to avoid the ink drying. Define the drying time experimentally.

5. Fatigue testing with DIC

5.1. Set the specimen into the servo hydraulic machine (see table of materials).

CAUTION: The servo hydraulic machine is hazardous. Use appropriate precautions and follow the instructions of the servo hydraulic machine manual.

5.2. Adjust the load-controlled fatigue test parameters using $R = 0.1$ ($\sigma_{\min} = 35$ MPa, $\sigma_{\max} = 350$ MPa) and test frequency of 10 Hz using the control software of the fatigue machine.

5.3. Set up an optical microscope with 16x precision zoom lens (see **Table of Materials**) for optical observation of the specimen notched area.

5.4. Equip the optical microscope with a digital camera with resolution of 2,048 pixels x 1,536 pixels.

5.5. Adjust the magnification of the optical microscope manually.

5.5.1. Ensure that the whole notched area of the specimen fits into the image area of the digital camera.

5.5.2. Ensure that the pixel size is at least 5 times smaller than the pattern size.

5.6. Run the fatigue test and synchronize with the image recording system.

5.6.1. Capture the images during temporary (10 s) stops of the fatigue test in intervals of 500 cycles.

5.6.2. Ensure that the load is held constant with an average stress of about 210 MPa during image acquisition.

5.7. Continue the fatigue testing until the crack length approaches a critical value or net-section plasticity starts to dominate.

6. Results analysis

6.1. Use the obtained raw images to perform the crack growth rate (CGR) and DIC analysis using a commercial software (see **Table of Materials**).

6.1.1. Use the operation manual to perform CGR analysis. Note that the crack growth rate analysis is possible to perform using the commercial software automatically or manually.

6.1.2. Perform the CGR analysis manually using the raw image dataset by measurement of the crack length increment after each 500 cycles.

6.2. Analyze of the shear strain deformation for the studied area using commercial software.

6.2.1. Use the operation manual to perform shear strain deformation analysis.

6.2.2. Ensure that correlation mode in time series settings of the software is chosen to be “relative to the first”.

6.3. Perform Schmid factor and grains misorientation analysis of EBSD data using the open source MTEX toolbox (see **Table of Materials**).

NOTE: Details about Schmid factor and grains misorientation analysis are available in user guide of MTEX toolbox²⁴.

6.4. Perform cumulative analysis of the obtained results.

NOTE: The cumulative analysis is discussed in Ref. ¹⁸.

6.4.1. Use Vickers microindentation marks to match the grain boundary map, misorientation map and Schmid factor map on top of the shear strain deformation field¹⁸.

6.4.2. Define the correlation between CGR, strain field and microstructure (misorientation and Schmid factor maps)¹⁸.

REPRESENTATIVE RESULTS:

Using the proposed methodology, we can analyze the sub-grain deformation field accumulating during small fatigue crack propagation under cyclic loading. The characterization is performed at sub grain level showing tiny features of the material behavior under fatigue loading even within a single grain. In particular, formation of shear strain localization fields was observed as shown in **Figure 4**. A number of tests were performed to verify the observed phenomena.

The deformation field is easily combined with the grain boundary image for a comprehensive characterization of the features responsible for the anomalous growth behavior of the small fatigue cracks (see **Figure 5**). Cumulative analysis of the deformation field, microstructure, crack growth rate and crack path reveal a dependence between the small crack growth rate retardation and accumulation of the shear strain localization zone¹⁸, as shown in the video.

FIGURE AND TABLE LEGENDS:

Figure 1: Schematic view of the fatigue test specimen of the studied ferritic stainless steel (dimensions are in mm).

Figure 2: SEM image of the side surface of the ferritic stainless steel specimen in the vicinity of the notched area (a) and its inverse pole figure (IPF) map with IPF key in the inset (b). The alignment of the DIC strain field and EBSD image was performed with help of Vickers microindentations shown by dashed circles (a).

Figure 3. Optical microscopy of the specimen side surface decorated with a pattern.

Figure 4. Intermittent accumulation of the shear strain localization zones during small fatigue crack growth.

Figure 5. Two examples (a and b) of the combined view of the shear strain field and microstructure of the studied steel tested in fatigue.

Figure 6. Custom-made pneumatic machine for pattern decoration of the specimens.

DISCUSSION:

A novel in situ measurement approach is introduced to measure the cumulative deformation field at a grain micro-scale level. In order to demonstrate the approach capability, the microstructurally small fatigue crack propagation behavior is studied in ferritic stainless steel with 18% chromium. The studied steel was provided in the shape of hot rolled plate with a thickness of 3 mm (see **Table of Materials**) and average grain size of about 17 μm ²¹.

A successful measurement requires that an initial fatigue crack is produced at the notch tip of the specimens for further propagation behavior analysis. In order to study a microstructurally small crack, the length of the initial crack should be significantly smaller than the grain size of the studied steel. Fatigue testing is displacement controlled to prevent crack growth after fatigue crack initiation. It was found that fatigue crack initiation time decreases significantly with the decrease of stress ratio (R). Thus, only 10,000 cycles were required for fatigue crack initiation in the specimens tested with R-ratio -0.16, while with R-ratio 0.1, the fatigue crack did not initiate even after 100,000 cycles. The use of the load ratio $R=-0.16$ allows to increase the stress range from 315 MPa to 350 MPa, having still smaller maximum stress for pre-cracking than that of actual fatigue testing.

The intermittent small fatigue crack growth is usually associated with the microstructure. In particular, grain boundaries are widely considered as microstructural features responsible for small crack growth retardation^{4,5,6,7,8,9,10,11,12}. The dislocation formulation in the boundary element by Hansson et al.¹³ shows that the low-angle grain boundaries lying in the way of the crack path can result in both an increase and decrease of the crack growth rate; however, the high-angle grain boundaries do not affect the crack growth rate. The physical reasons causing the anomalous crack growth behavior are not well known. In order to reveal the microstructural features causing the small crack retardation, a microstructural characterization was performed before fatigue testing of the specimen. The polishing procedure described in step 1 is crucial for reliable microstructural analysis using EBSD. In step 3, just before EBSD analysis, the cleaning of the specimen in ethanol is only allowed, since acetone vapor is hazardous for EBSD detector.

In order to reveal deformation processes within individual grains, the size of the speckle pattern must be significantly smaller than the grain size of the studied steel. Since the average grain size of the steel after annealing is about 350 μm , the characteristic size of the speckle pattern required for DIC calculation was chosen to be approximately 10 μm ^{22,12}. The speckle pattern size must be at least 10 times smaller than the grain size of the studied steel for proper implementation of step 5. The surface of the specimen is decorated with a speckle pattern using a silicone stamp. We use a custom-made pneumatic tool (see **Figure 6**) for fast and precise operation of the stamp.

Small fatigue crack propagation behavior is studied during load-controlled fatigue testing of the pre-cracked specimens using the R-ratio of 0.1 ($\sigma_{\min} = 35 \text{ MPa}$, $\sigma_{\max} = 350 \text{ MPa}$) and the frequency of 10 Hz. Fatigue testing follows together with digital image correlation (DIC) measurement. The area of interest is monitored using an optical microscope, 16x Precision Zoom Lens, with a resolution of 2 $\mu\text{m}/\text{pixel}$. Images are captured during temporary (10 s) stops of the fatigue test in intervals of 500 cycles. During image acquisition, the loading is held constant, with an average stress of approximately 210 MPa, in order to have equal loading conditions for all images, stabilize plastic deformation, and avoid fatigue crack closure and extensive creep accompanied with min and max of loading force, respectively. The novelty of the method is based on high-resolution in situ DIC image recording that allows to reveal tiny deformation zones forming during small fatigue crack growth. The success of the experiment depends on the proper implementation of the pre-cracking procedure, selection of image capture interval and

magnification to prevent the blurring of small features such as the observed shear strain localization zones. Thus, proper selection of camera resolution, optical magnification and speckle pattern size as described in step 5 of the protocol can be crucial for investigation of the strain localization phenomena. However, morphology of the shear strain localization zones is still unclear and needs further improvements of the speckle pattern and resolution of the image recording equipment.

The methodological approach described in this paper is suitable for crack growth analysis of small fatigue cracks in coarse-grained materials. A combination of crack growth rate measurement and strain-field analysis at the sub grain level helps to reveal the mechanism that are responsible for anomalous growth of the small fatigue cracks¹⁸, in addition to the widely observed grain boundary effects on SFCs. Deeper understanding of the fatigue fracture mechanisms makes development of new theoretical approaches possible and thus, enables design of lighter and more energy efficient structures in the future.

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DISCLOSURES:

The authors have no competing financial interests to disclose.

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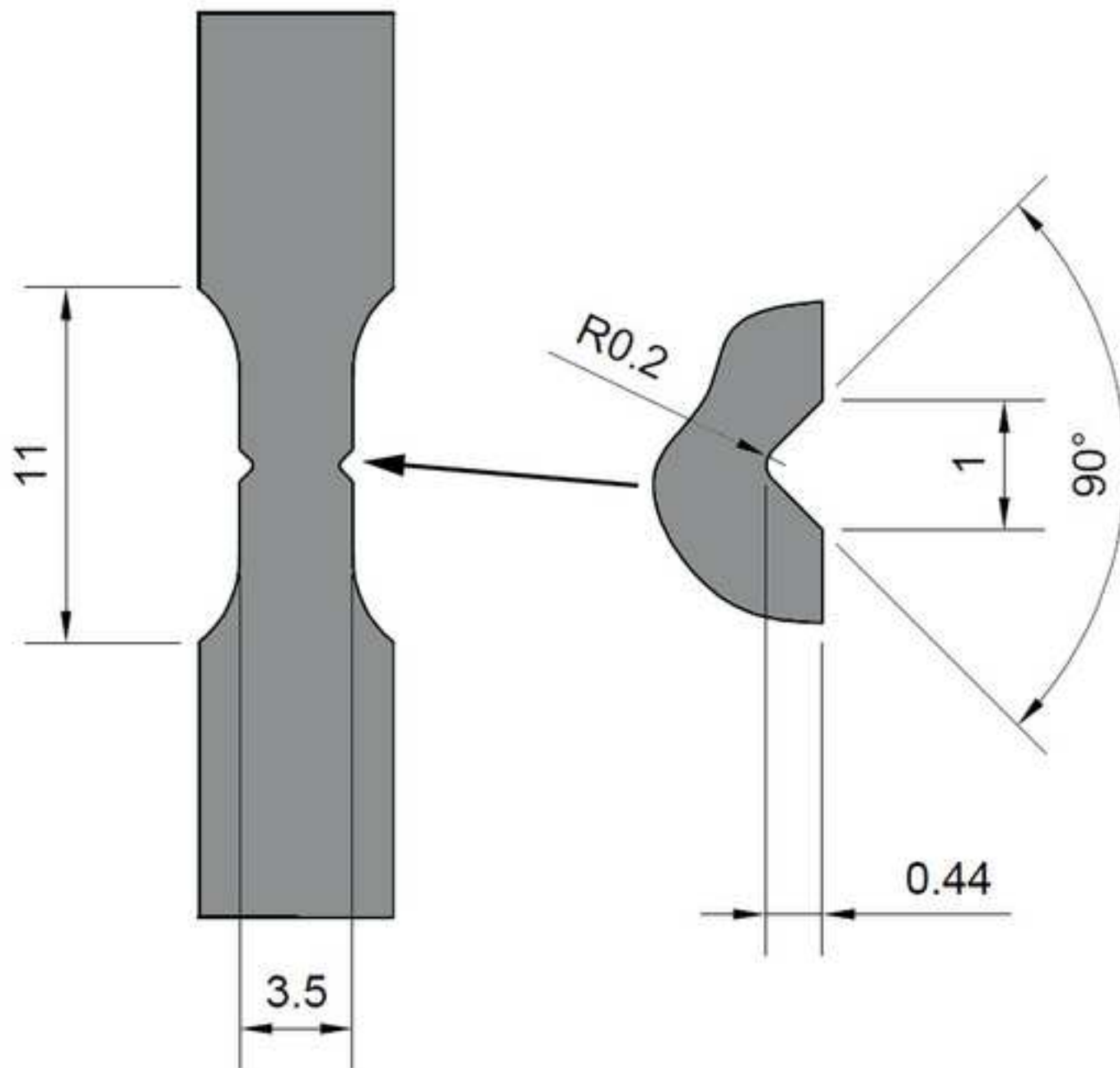
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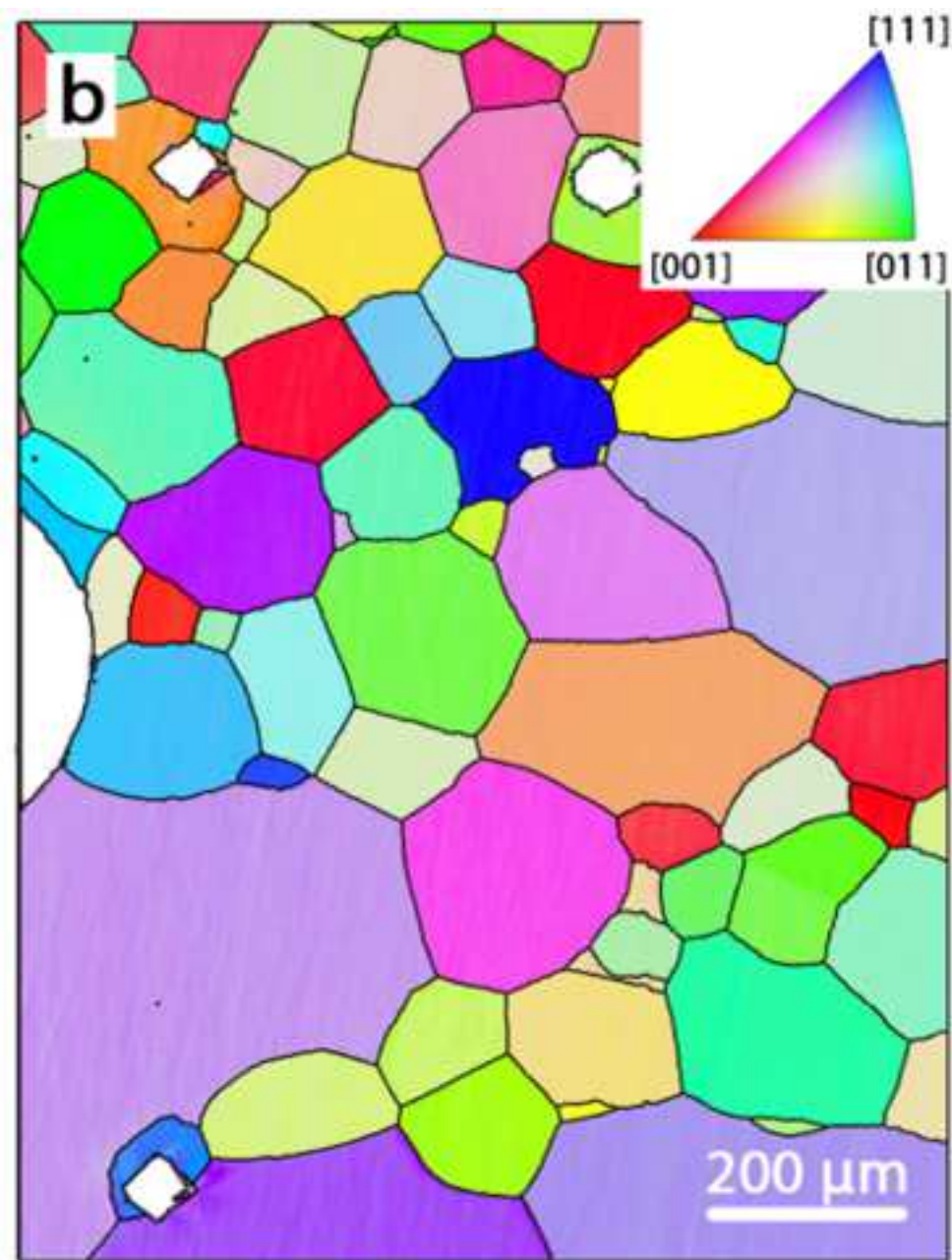
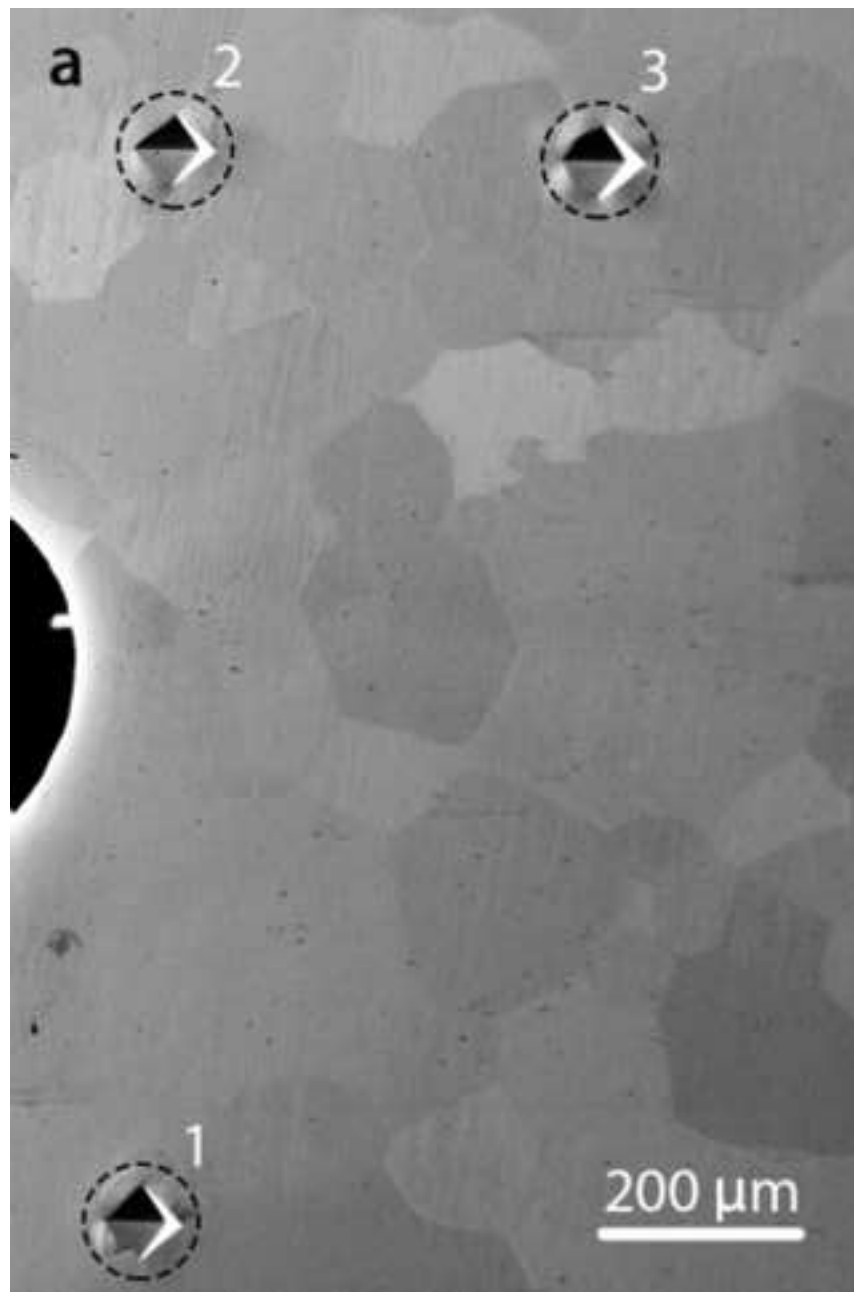
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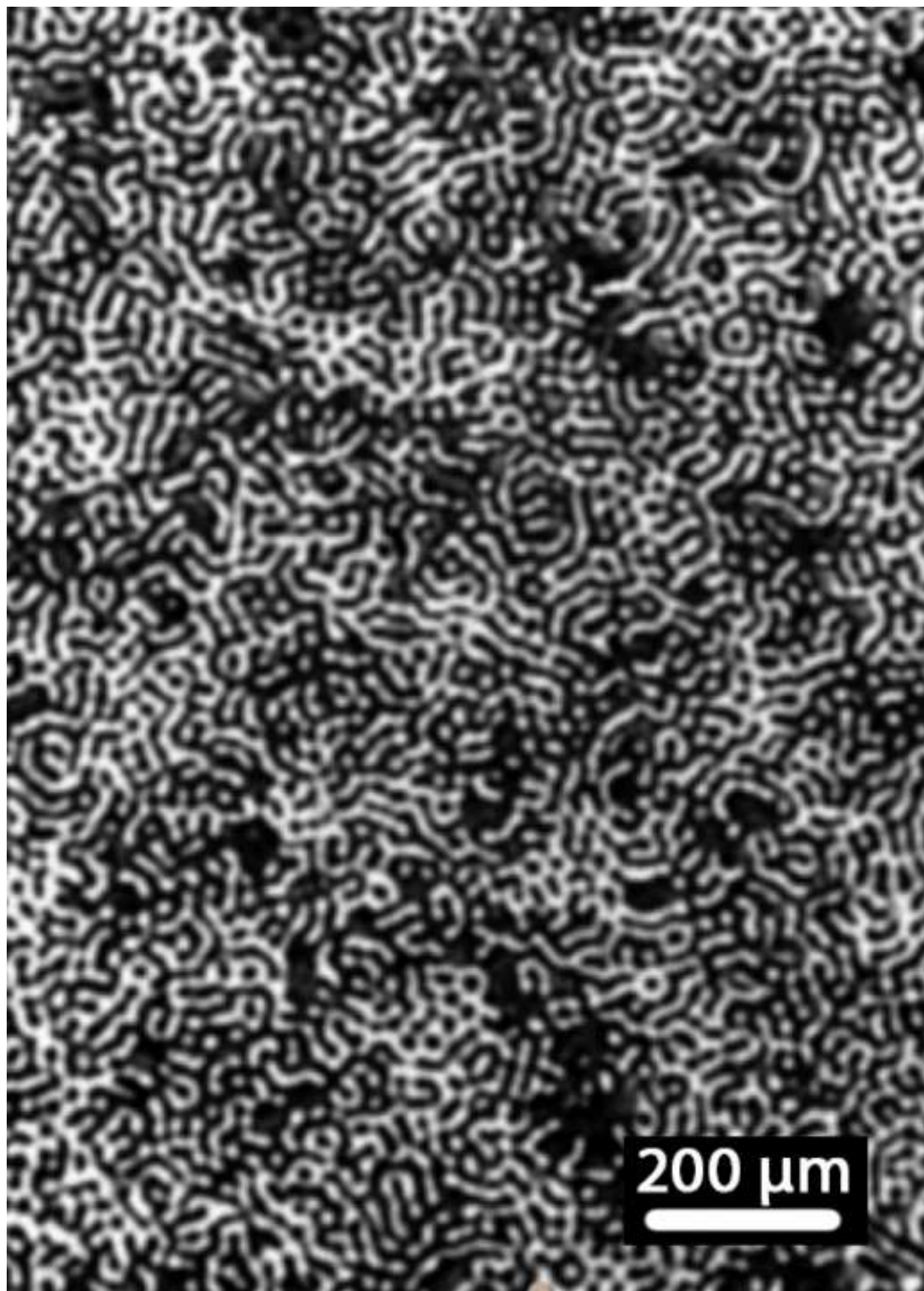
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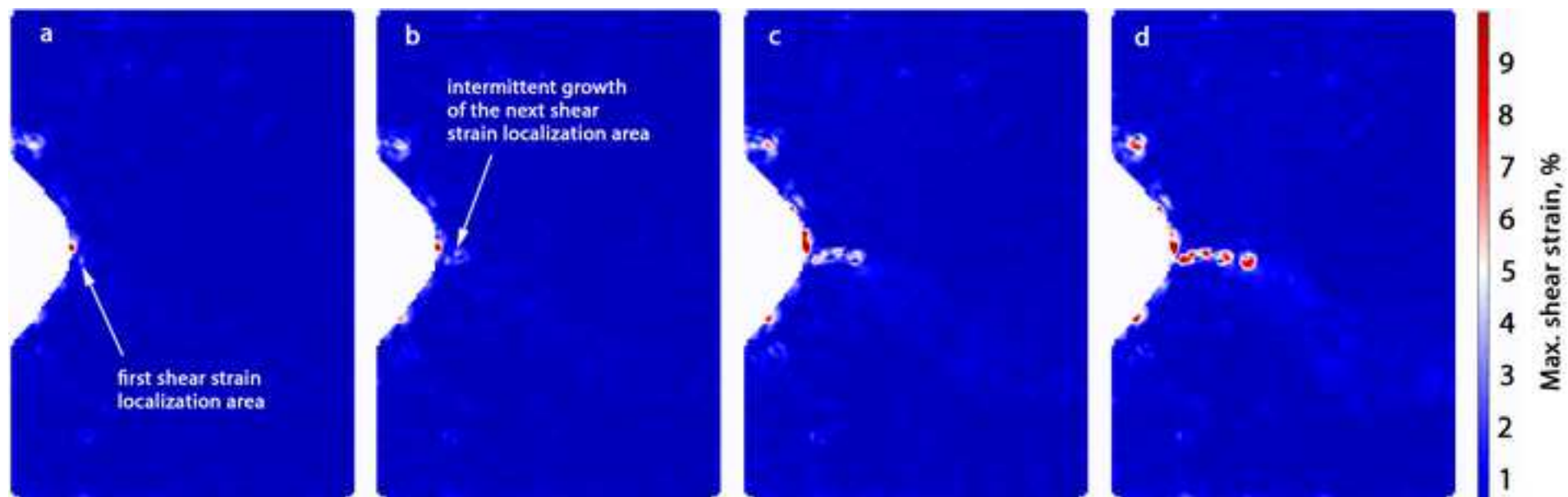
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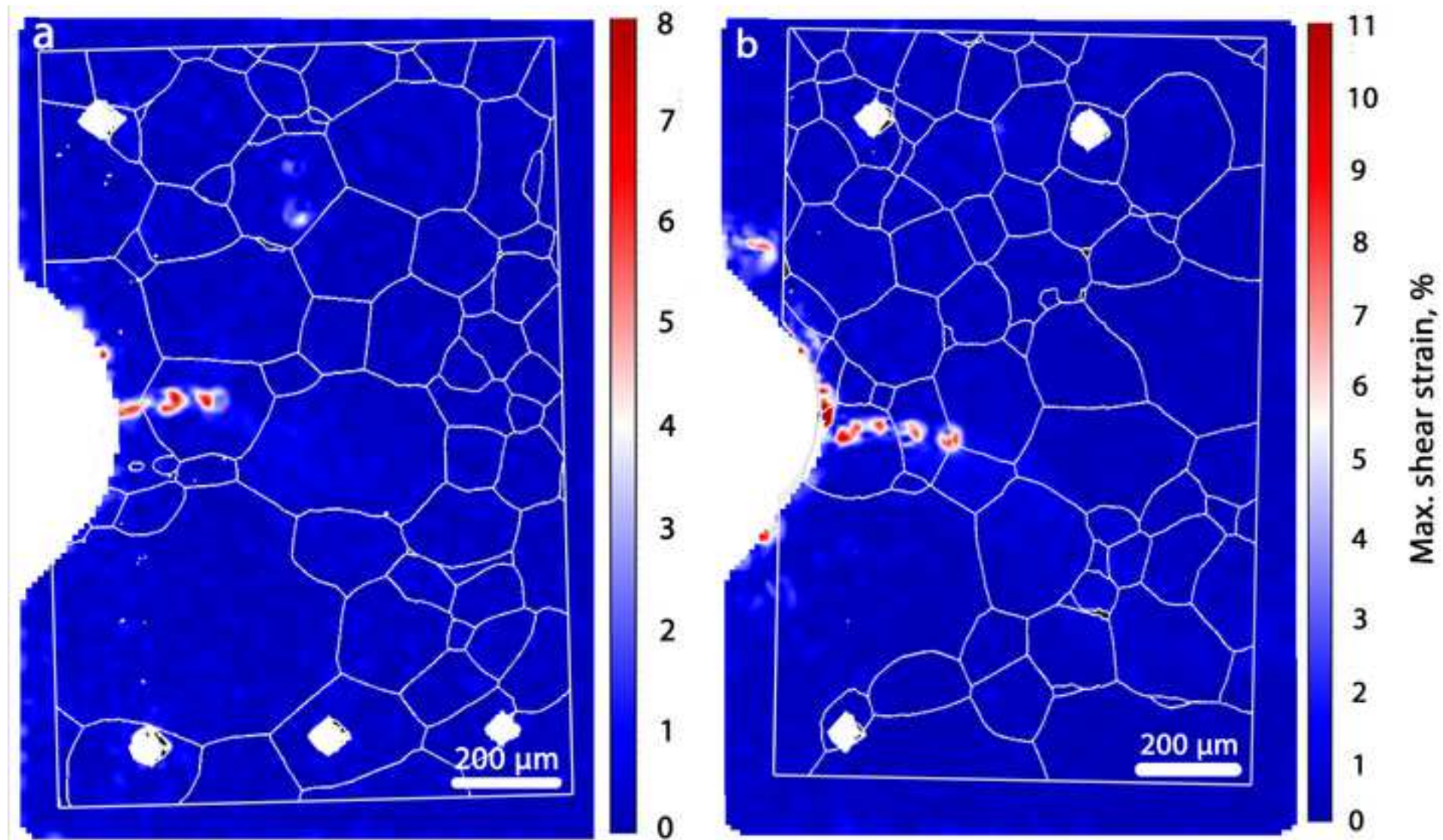
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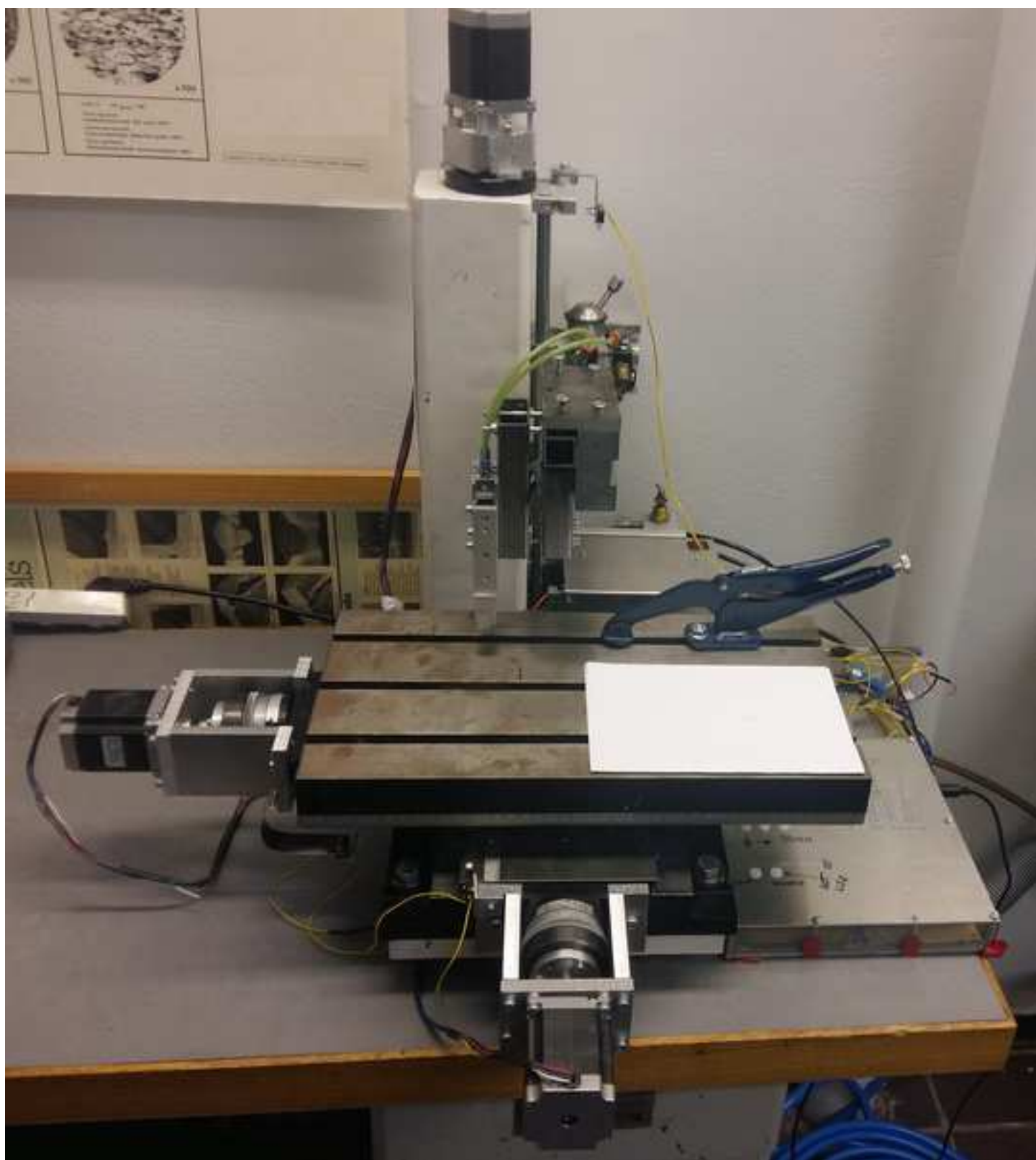












| Name of Material/ Equipment | Company | Catalog Number | Comments/Description |
|--|---|--|--|
| Acetone | Sigma-Aldrich | STBH7695 | Acetone pyrity ≥ 99.5 % |
| Argon gas | Oy AGA Ab, Industrial Gases (Finland) | UN 1006 | Gas purity ≥ 99.9999 % |
| Chamber furnace | Lenton | 4934 | heat range 20-1200 °C |
| Commercial software DaVis 8 | LaVision Inc. | | Commercial software used for crack growth rate and strain field analysis |
| Custom-made pneumatic stamping tool | Aalto University | | Made in Aalto University |
| Diamond paste | Struers Inc. | DP-Mol. 3 μm , DP-Nap. 1 μm , | Paste for polishing |
| Emery paper | Struers Inc. | FEPA P #800, FEPA P #1200, FEPA P #2500 | Paper for grinding |
| Ethanol | Altia Industrial | ETAX Ba | Ethanol pyrity ≥ 99.5 % |
| FEG-SEM scanning electron microscope | ZEISS | ULTRA 55 | EBSD analysis |
| Ferritic stainless steel | Outokumpu Stainless Oyj (Finland) | Core 441/4509 (ASTM UNS S43940) | 3 mm rolled plate |
| For Vacuum pump | Leybold-Heraeus | D4B/WS | |
| Grinding machine | Struers Inc. | LaboPol-21 | Hand grinding |
| MasterMet 2 Non-Crystallizing Colloidal Silica Polishing Suspension | Buehler Inc. | 40-6380-064 | 0.02 μm colloidal silica |
| MatLab software | MathWorks Inc. | | MatLab software used as a platform for MTEX toolbox |
| Milling machine | 3ΦC Stankoimport (Moscow, USSR) | 6P82Ш #22 | Aalto University machining services |
| Micro Vickers hardness tester | Buehler Inc. | 1600-6400 | |
| MTEX software | Open source | | Open source toolbox based on MatLab for analysis of the EBSD data (http://mtex- toolbox.github.io/) |

| | | | |
|-------------------------|------------------------|------------------------|-----------------------|
| Optical microscope | Nikon Corporation | EPIPHOT 200 | |
| Polishing machine | Struers Inc. | LaboPol-5 | Hand polishing |
| Servo hydraulic machine | MTS system corporation | 858 Table Top System | |
| Turbomolecular pump | Leybold-Heraeus | Turbovac 50 | |
| Vibratory polisher | Buehler Inc. | VibroMet 2 | Automatic polishing |
| Wire-cut EDM | TamSpark Oy | Charmilles robofil 400 | wire diameter 0.15 mm |



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Full-field strain measurements for microstructurally small fatigue crack propagation using DIC method.

Author(s):

E. Malitzkii, H. Reme, P. Lehto, S. Bossuyt

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Article Title:

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Hereby we submit the revision for the manuscript “Full-field strain measurements for microstructurally small fatigue crack propagation using digital image correlation method”. We thank the reviewers for the instructive comments of the manuscript.

Response to Editorial and production comments

Following suggestions:

1. Please take this opportunity to thoroughly proofread the manuscript to ensure that there are no spelling or grammar issues.

The manuscript was proofread and corrected.

2. Please revise lines 134-136, 164-167, 196-199 to avoid previously published text.

The lines was revised and corrected as shown in lines 280-283, 299-301, 250-253.

3. Please avoid abbreviations in the title.

The abbreviation in the title of the manuscript was removed.

4. Please provide an email address for each author.

Email address was provided for each author.

5. Please spell out each abbreviation the first time it is used.

Each abbreviation were spelled out the first time it is used.

6. Please use SI abbreviations for all units: L, mL, μ L, h, min, s, etc.

SI abbreviations were used for all units.

7. Please include a space between all numerical values and their corresponding units: 15 mL, 37 °C, 60 s; etc.

Space between all numerical values and their corresponding units was included.

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The commercial language was removed from the manuscript and replaced using generic terms followed by reference “(see table of materials)”.

9. Please adjust the numbering of the Protocol to follow the JoVE Instructions for Authors. For example, 1 should be followed by 1.1 and then 1.1.1 and 1.1.2 if necessary. Please refrain from using bullets, dashes, or indentations.

The numbering of the Protocol was adjusted.

10. Please revise the protocol text to avoid the use of any personal pronouns (e.g., “we”, “you”, “our” etc.).

The protocol text was revised and all personal pronouns was removed.

11. Please revise the protocol to contain only action items that direct the reader to do something (e.g., “Do this,” “Ensure that,” etc.). The actions should be described in the imperative tense in complete sentences wherever possible. Avoid usage of phrases such as

“could be,” “should be,” and “would be” throughout the Protocol. Any text that cannot be written in the imperative tense may be added as a “Note.” Please include all safety procedures and use of hoods, etc. However, notes should be used sparingly and actions should be described in the imperative tense wherever possible. Please move the discussion about the protocol to the Discussion.

The Protocol was revised. The Protocol steps were rewritten in imperative mode. Safety procedures were added with appropriate references.

12. The Protocol should be made up almost entirely of discrete steps without large paragraphs of text between sections. Please simplify the Protocol so that individual steps contain only 2-3 actions per step and a maximum of 4 sentences per step. Use sub-steps as necessary. Please move the discussion about the protocol to the Discussion.

The Protocol steps were simplified reducing the number of sentences per individual step and using sub-steps. The discussion about protocol was moved to the Discussion.

13. Please add more details to your protocol steps. There should be enough detail in each step to supplement the actions seen in the video so that viewers can easily replicate the protocol. Please ensure you answer the “how” question, i.e., how is the step performed? Alternatively, add references to published material specifying how to perform the protocol action. See examples below.

More details were added to the protocol steps. References [19], [20], [23], [24] were added to specify the actions of the Protocol steps.

14. Line 140: Please describe how to use Vickers microindentations.

The procedure of microindentation and use of the Vickers marks was described in more details as shown in lines 155-163, 229-231.

15. Line 143: Please describe how to perform electron backscatter diffraction (EBSD) analysis.

The procedure of EBSD analysis was described in lines 164-173.

16. Line 144: Please describe how this is done.

The procedure was specified in lines 224-226.

17. Line 153: Please specify the purity of ethanol.

Purity of ethanol was specified in table of materials.

18. Line 154: Please describe how.

The procedure was specified in lines 179-180.

19. Line 171: Please describe how to run the fatigue test.

The procedure was specified in lines 193-197.

20. Figure 1: Please indicate the unit for the numbers shown in the figure.

The unit was specified in the figure name. Please see the line 248-249.

21. Figure 2 and Figure 4: Please use the micro symbol μ instead of u.

Figures 2 and 4 were corrected.

22. Discussion: As we are a methods journal, please also discuss critical steps within the protocol, any modifications and troubleshooting of the technique, and any limitations of the technique.

The critical steps were discussed in lines 268-271, 286-288, 292-294.

23. For in-text references, the corresponding reference numbers should appear as superscripts after the appropriate statement(s) in the text (before punctuation but after closed parenthesis). The references should be numbered in order of appearance.

The references were updated and placed after appropriate statements.

24. Please ensure that the references appear as the following: [Lastname, F.I., LastName, F.I., LastName, F.I. Article Title. Source. Volume (Issue), FirstPage – LastPage (YEAR).] For more than 6 authors, list only the first author then et al. See the example below: Bedford, C.D., Harris, R.N., Howd, R.A., Goff, D.A., Koolpe, G.A. Quaternary salts of 2-[(hydroxyimino)methyl]imidazole. Journal of Medicinal Chemistry. 32 (2), 493-503 (1998).

The style of references was updated according to the comment.

25. References: Please do not abbreviate journal titles.

The journal title abbreviations were removed.

26. Table of Equipment and Materials: Please sort the items in alphabetical order according to the Name of Material/ Equipment.

The items of table of materials were sorted in alphabetical order.

Changes to be made by the Author(s) regarding the video:

1. 01:21, 01:44, 03:46, 04:18, 05:07, 05:23, etc.: Please use the micro symbol μ instead of u.

The micro symbol u was replaced by μ in video.

2. 04:44: Different from the video, the written protocol does not have step 6. Please include it in the manuscript.

The Step 6 was included in Protocol in lines 212-233.

3. The video must have chapter title cards to show the video is changing from one section to the other.

Chapter title cards were added in the video.

4. Branding concerns

• 0:01 - The university logo should be removed from the opening title card. It can remain at the end of the video.

The university logo was removed from the opening title card.

• Please remove all commercial language references from the video and use generic terms instead. All commercial products should be sufficiently referenced in the Table of Materials and Reagents. Examples of commercial sounding language are: Outokumpu Stainless Oyj, LaVision, MatLab, etc

Commercial language was removed from the video.

5. Text/formatting issues

• 5:23 - A chapter title card that reads "Representative Results" should be added here. This will be necessary for the chaptering feature on our website.

The title card "Representative results" was added to the video.

6. Please upload a revised high-resolution video here:

<https://www.dropbox.com/request/BwkGIFoIbsq1n8HFrAtN>

The updated high resolution video was uploaded using the link.

Response to Reviewer #1 comments

Major Concerns:

Page 1, lines 32-34: « At the same time, the intensity of the shear strain localization seems to be dependent on the grain orientation » This information appears only in the abstract and is not discussed in the paper nor in the video. Could the authors discuss this aspect in the paper as they present results about grain orientation (cf Fig. 2)

Target of the present work is to describe the method used to study the mechanism of small fatigue crack propagation described in details in Reference [18] of the manuscript. In order to avoid misunderstanding we changed the emphasis by modification of Abstract.

Page 4:

Lines 151-152: How is the silicone stamp fabricated? Do the authors compare several pattern before choosing this one? Please provide more information as I doubt that the stamp is so easy to obtain and as it seems to me that the pattern with such a spatial resolution is the most important part of the proposed methodology.

The silicone stamp used in the present work is the result of the ongoing research in our university. All information related to pattern quality and stamp fabrication process can be found in Reference [22] and [23] of the manuscript. The manuscript was updated with respect to the pattern properties (lines 184-186, 289-292) and additional Reference [23] included.

Page 5:

Lines 174-176: Please provide more details about DIC with LaVision with at least the spatial resolution of DIC and the images that were used for correlation, i.e. the reference and deformed images that allow to measure a cumulative strain. How is the crack growth rate computed: crack tip identification from the raw image or from DIC? In the video it is said that LaVision is used? Is there a fitting of the crack length as a function of the number of cycles? The CGR is not presented into details in the paper but it is shown in the video so it would be useful to detail how it is obtained.

The information about spatial resolution of DIC and images as well as the correlation method was described in lines 200-204, 289-296, 299-301. The CGR can be calculated automatically or manually using LaVision software. We use the raw images directly from the camera to calculate the CGR. Since the quality of the raw images is not always good enough to perform the automatic CGR analysis the best and most accurate solution is to make it manually. The procedure is described in lines 213-219.

Page 6: The discussion emphasizes the observation of strain localization zones ahead of the crack tip and suggests there is a link between these zones and "anomalous growth of the small fatigue cracks". On has to look at the video to discover how this link works. It would be nice to have some explanation in the paper. Besides the authors do not explain where these high shear strain localization stem from. Do the authors observe a link between the highly strained area and the grain orientation as indicated lines 33-34?

The present paper is targeted to reveal the experimental approach that allows to study the small fatigue crack growth behavior. The detailed analysis of the relationship between the observed shear strain localization zones, crack growth rate and microstructure was performed and published previously (see Reference [18] of the manuscript). Step of the results analysis was added in Protocol (lines 212-233)

Video:

3min39: Maps of Schmid factor and grain orientation are shown although they are not used after.

In the video, we try to reveal the steps that need to be performed for further cumulative analysis of the results described in reference [18]. Objective of the manuscript is now presented more clearly in Abstract and Introduction of the manuscript (lines 25-35, 92-102).

5min51: The relationship between crack growth rate and strain localization is discussed but not the link between the grain boundaries and grain orientation and strain localization. It would be nice to discuss a little bit more the relationship between short fatigue crack growth and the microstructure that is presented sooner in the video.

Discussion about the relationship between SFC growth and the microstructure is closely related to the studied material. Since we discuss the method, the detailed analysis of the results performed for one particular material is not the objective of this paper. However, the detailed analysis of the link between CGR, strain localization, grain boundaries and grain orientations of the 18%Cr ferritic stainless steel is available in reference [18] and discussed in lines 212-233 of the manuscript.

Minor Concerns:

Page 2, line 52: « such a complicate processes » → such a complicated process

The mistake was corrected. Please see line 51.

Page 3:

Line 126: the sentence has no verb: « 2 Optical monitoring of the initial crack formation after 10 000 cycles. »

The sentence was corrected. Please see line 139.

Line 128: « 3 Repeat the fatigue loading cycle if an initial crack was not produced. ». Please rephrase the sentence as to have subject - verb - complement and be consistent with the previous items.

The sentence was rewritten. Please see line 146.

Page 4, Line 169: Why were the images acquired at a 210MPa stress level which is neither the minimum nor maximum stress of the fatigue cycle? Please justify.

The image acquisition was performed at an average stress of about 210 MPa in order to stabilize plastic deformation. This avoids fatigue crack closure and extensive creep accompanied with min and max of loading force, respectively. Please see lines 302-305.

Page 5:

Line 195: I guess the dimensions in Fig. 1 are in mm. Please indicate it.

The dimensions were indicated. Please see lines 248-249.

Line 202-203: The range of the strain map is not visible. Please use a larger font size.

The font size of the strain map was increased.

Response to Reviewer #2 comments

Manuscript Summary:

The small fatigue crack growth of bcc Fe-Cr ferritic stainless steel has been studied using

digital image correlation. The paper is not the first on small fatigue crack growth assessment and is just presenting shortly a standard DIC test. There is almost no scientific analysis of the obtained results. This work can be only classified as a letter with limited novel scientific outcome. The authors are expected to improve the discussion of the results and don't postpone this to their future articles. I'm sorry but I cannot accept this article in its current form. It requires major revision before second submission.

Experimental investigation of the small fatigue crack growth behavior is a complicated task due to the limitations of the experimental techniques. The DIC measurement performed at high magnification using scanning electron microscopy evidences strain localization in vicinity of the crack, however, the ex-situ measurement approach blurs out the details of the deformation field accompanying the short crack propagation (see Ref. [14] of the manuscript). The novelty of the approach consists of in-situ full field strain measurement that allows to reveal the transitional details of the strain field evolution during SFC propagation. The proposed approach is novel also due to the unique patterning technique with spatial resolution at least one order of magnitude below the characteristic length scale of the microstructure. Since the JoVE is the journal of methods, in our manuscript we focused on detailed explanation of the experimental procedure of the small fatigue crack growth study over hundreds of thousands of load cycles using DIC, crack growth analysis and microstructure analysis. The scientific analysis of the results obtained using this methodology is already published in ref. [18] of the manuscript. The manuscript was considerably rewritten to emphasize better the main objective of the study.

Major Concerns:

- The authors have mentioned in the text "The most promising tool for analysis of cyclic material deformation behavior at micro-scale is the digital image correlation (DIC) technique.". how do you prove this claim? Did you compare different methods to come to this conclusion? Or it's your imagination? Please provide citation for this statement.

In order to avoid unclear sentence, the text was rewritten in lines 65-68.

- provide the country and city for "Outokumpu Stainless Oyj".

Outokumpu Stainless Oyj is located in Tornio, Finland. The information about location is added in table of materials.

- how did the authors choose the displacement range for pre-cracking the test specimens? Did you use any standard? Please add more details.

The displacement range was chosen experimentally so the σ_{min} and σ_{max} are in range of about -50 MPa and 300 MPa, respectively. The maximum tensile stress should not be over the yield point of the material to avoid extensive plastic deformation. Manuscript was rewritten in lines 134-147, 271-277.

- how the initial pre-crack of 1 μm was detected? Did you use a potential drop? Or only DIC. Please add a picture of pre-cracked specimen with 1 μm pre-crack.

Definitely, the crack with only 1 μm length is difficult to observe. We specified the range from 1 μm to 20 μm to show the pre-crack length we typically accept for our tests. The crack with length of about 1 μm is usually difficult to distinguish using optical microscopy, so the pre-cracking test was repeated to get the pre-crack visible. The manuscript text was rewritten in lines 144-145 to exclude the misunderstanding. The typical pre-crack with length of about 20 μm is shown in Fig. 1.

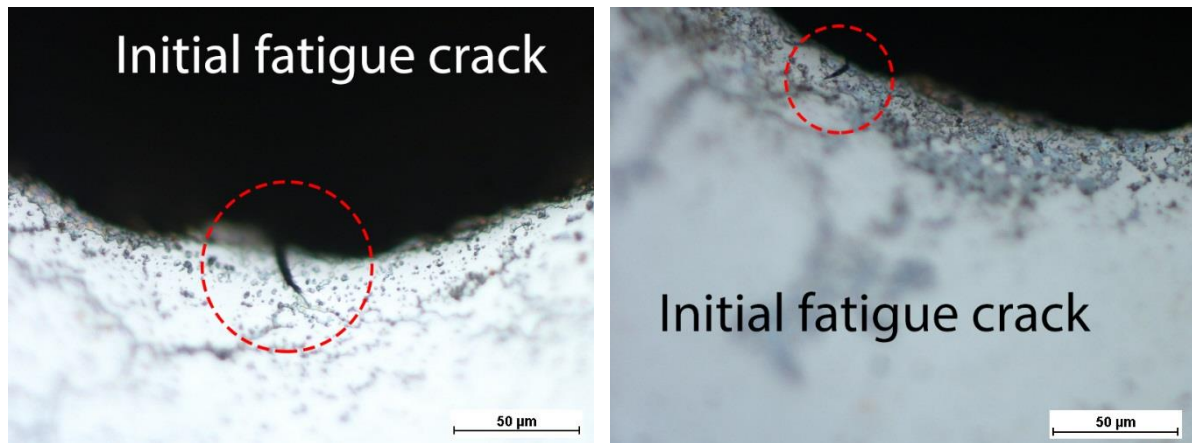


Figure 1. Optical micrograph of the initial fatigue crack produced by pre-cracking procedure.

Minor Concerns:

- Fig. 4 is not well-described, please add some sound scientific description of the observed pattern.

The silicone stamp used in the present work is the result of the ongoing research in our university. All information related to pattern quality and stamp fabrication process can be found in Ref. [22] and [23] of the manuscript (lines 184-188).

Thank you for the substantial comments.

On behalf of authors
Sincerely Yours,
Evgenii Malitckii