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Toughening mechanisms in nacre-like alumina revealed by in-situ imaging of stress

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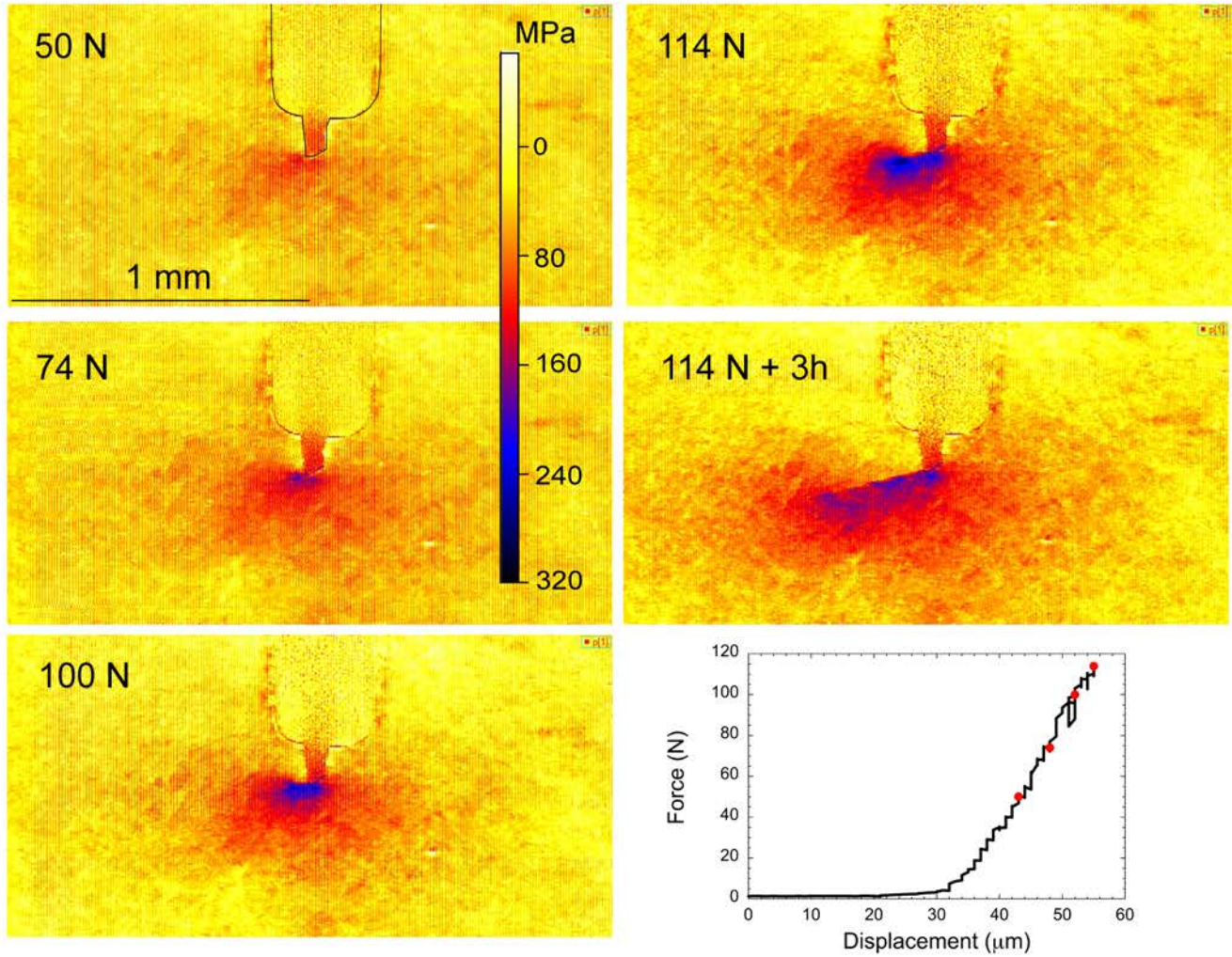
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Abstract. Nacre-like aluminas are bio-inspired, ceramic-based composites that display high toughness and strength. Their toughness arises from their brick-and-mortar microstructure. We must understand the role of several microstructural features over their mechanical properties. Techniques that allow microstructural imaging during mechanical tests are therefore desired. Here, we use both *in situ* fluorescence spectroscopy to image the stress field around cracks propagating in samples, and *in situ* scanning electron microscopy to image the crack-microstructure interactions. Stress concentrates around locally disordered zones where the crack is pinned while crack propagation is delayed. *In situ* imaging shows that obstacles to crack propagation are either larger-than-average alumina platelets or bundles of alumina platelets misaligned with respect to the majority of the platelets. Such microstructural heterogeneities are therefore important to impede crack propagation in nacre-like alumina. The approach proposed here can be used to understand the structure/properties relationships of other types of materials.

Keywords: *mechanical behavior, toughness, scanning electron microscopy, fluorescence spectroscopy, ceramic composites*

32 Graphical abstract



33

34 Images of stress and crack propagation in single-edge notched beam of nacre-like alumina submitted to
35 increasing force

36

37 **1. Introduction**

38 Strength and toughness are mutually exclusive properties of materials in general and ceramics in
39 particular. Designs found in biological structural materials such as nacre may provide useful
40 guidelines to alleviate this trade-off (Ritchie, 2011). Nacre-like alumina, made of brittle constituents
41 only, combines the hardness and strength of standard alumina materials, and comparatively high
42 toughness. This toughness (damage resistance) is achieved mostly by the deviation of cracks along the
43 platelets interface in their brick-and-mortar microstructure (Bouville et al., 2014). While the deviation
44 of cracks in brick-and-mortar microstructures is well documented, several other toughening
45 mechanisms can exist in biological or bio-inspired materials (Bouville, 2020; Kakisawa and
46 Sumitomo, 2011). Discrete elements modeling (DEM) has been implemented to understand the
47 toughening mechanisms of nacre-like alumina and provide guidelines of microstructural features to
48 implement to improve the damage resistance of these composites (Radi et al., 2020). Establishing
49 quantitative links between nano- to microscopic structures and bulk mechanical properties determined
50 in standardized fracture tests requires additional mechanical modeling at various scales and methods
51 that enable imaging the effects of microstructures on the mechanical behavior. For instance, structural
52 defects (such as local misalignments, variable platelet size) of real microstructures spanning few tens
53 of micrometers can be modeled by DEM, which suggests that they may have a significant effect on
54 crack propagation (Radi et al., 2021). To validate these results, we developed *in situ* spectroscopic and
55 scanning electron microscopy (SEM) imaging of stress field and microstructure during a bending test
56 on nacre-like alumina.

57

58 **2. Materials and methods**

59 Samples were prepared as standard single-edge notched beams (SENB), and mirror polished
60 before testing (Saad et al., 2020). $3 \times 6 \times 36$ mm samples were used for fluorescence spectroscopy and
61 SEM characterization. The fluorescence of trivalent chromium (Cr^{3+}), a ubiquitous impurity in

62 alumina-based polycrystalline ceramics, was used to map stresses in the composite (Pezzotti et al.,
63 2012; Portu et al., 2005). Even in very low quantities (< 0.5 wt%), Cr^{3+} causes an intense fluorescence
64 characterized by two sharp and intense lines, noted R_1 and R_2 , whose energy shifts with applied stress
65 (Feher and Sturge, 1968; He and Clarke, 1995).

66 We used a Horiba™ LabRam HRevolution Raman spectrometer equipped with a motorized
67 stage for mapping the fluorescence signal excited with a 532-nm solid-state diode laser from SENB
68 submitted to a four-point bending test. The laser is focused through a microscope equipped with a
69 long-working distance x50 Mitutoyo™ objective down to a spot of about $1\mu\text{m}$. The high fluorescence
70 signal allows a typical recording time τ of the order of one millisecond or less to obtain an intense
71 enough fluorescence spectrum at each point and determine peak position and stress. X-Y maps of the
72 sample lateral surface were obtained in the SWIFT™ mode where the sample is scanned at constant
73 speed in the X direction (perpendicular to the notch, see Figure 1 left) under the laser, and signal-
74 processed through the CCD detector to record individual points every τ . Spatial resolution is
75 determined by the product of the scanning speed and reading time τ in the X direction, and by step-by-
76 step motion in the Y direction (along notch). Identical steps were chosen in both X and Y directions to
77 avoid potential distortion in the image, and set to 5 and $2\mu\text{m}$ for low- and high-resolution images,
78 respectively. Low-resolution images covering a $1 \times 2\text{mm}^2$ field of view and high-resolution images
79 covering $300 \times 750\mu\text{m}^2$ were acquired in about one minute each around the crack. One image with
80 steps of $0.5\mu\text{m}$ over a $400 \times 150\mu\text{m}^2$ area was acquired in 40 minutes at the end of the test
81 (corresponding to a long crack propagation but without macroscopic failure of the sample) for
82 comparison with post mortem imaging by SEM. The stress was maintained at a set value at each step
83 while maps were acquired. With the optical setting and characteristics of the laser-material interaction,
84 the actual resolution is better than $2\mu\text{m}$.

85 The R_1 position shifts by $\Delta\nu$ with the applied stress σ as:

86

87

$$\Delta\nu = \Pi\sigma \quad (1).$$

88

89 $\Pi = 1/3 \partial\nu/\partial P = 2.5 \text{ cm}^{-1}.\text{GPa}^{-1}$ is the average piezospectroscopic coefficient with P the hydrostatic
 90 pressure, $\Pi_c = 1.5 \text{ cm}^{-1}.\text{GPa}^{-1}$ is the piezospectroscopic coefficient along the c direction and $\Pi_a =$
 91 $3.0 \text{ cm}^{-1}.\text{GPa}^{-1}$ is the average piezospectroscopic coefficient perpendicular to the c direction of
 92 corundum lattice (Feher and Sturge, 1968; He and Clarke, 1995). In the nacre-like microstructure the
 93 macroscopic applied tensile stress is parallel to the basal plane of most alumina platelets (or corundum
 94 structure), but the local stress field near the notch is not uniaxial. The average value of $2.5 \text{ cm}^{-1}.\text{GPa}^{-1}$
 95 for the piezospectroscopic coefficient was used here, which is sufficient for imaging and following the
 96 spatial and temporal changes in stress. Stress fluctuations in homogeneous areas yield an estimated
 97 uncertainty on relative values of stress of 40 MPa from pixel to pixel, and less than 15 MPa for 3×3
 98 pixel squares, *i.e.* over about 15 microns for low resolution images, and 6 or 3 microns for higher
 99 resolution images.

100 The sample tested *in situ* with fluorescence spectroscopy was characterized post operando with a
 101 SUPRA 55VP SEM. Due to the elastic spring back, the crack was not open anymore and its exact
 102 trajectory in the complex microstructure was determined by comparison with the high-resolution
 103 fluorescence image. A complementary SEM *in situ* analysis was performed on another SENB sample.
 104 The SUPRA SEM allows working at low acceleration voltage without metallization of the surface,
 105 which preserves the microstructure. In these conditions, back-scattered electrons are subjected to
 106 electron channeling that enhances the contrast between phases, highlighting the nacre-like
 107 microstructure (Lafond et al., 2021).

108

109 **3. Results**

110 Starting from an initial state with negligible stress, the evolution of the stress map calculated
 111 from large fluorescence maps (Fig. 1) shows, the tensile stress field that develops around the crack tip

112 as applied force increases (Fig. 1). Tensile stresses are detected while the sample is still in the elastic
113 domain, and well before the crack initiation. The stress field is asymmetric with the side at higher
114 stress (left of the notch in Figure 1) eventually fracturing with a crack initiating at about 70° of the
115 notch plane for an applied force of 110 N. Before the crack can be observed at the surface of the
116 sample, the area under tension remains constant and does not increase in size, only the value of the
117 calculated stress increases with the loading force to exceed locally 300 MPa. While the crack extends,
118 the tip of the uncracked ligament is subjected to important tensile stresses, with average values up to
119 250 MPa. The tensile nature of the stress field generated at the bottom of the notch is coherent with
120 the SENB configuration

121 At an applied force of 110 N, the first high-resolution image recorded (Fig. 2) shows a stress
122 accumulation pinned on a mechanical asperity along the crack (point 1). The force was increased to
123 114 N, and the sample was then maintained at a constant force for several hours. High-resolution
124 maps display the evolution of the stress field, which highlight mechanical asperity effects during sub-
125 critical crack propagation. The crack first propagated towards a second mechanical asperity (point 2)
126 after the first low-resolution map was recorded at 114 N (about 10 minutes), and did not evolve with
127 the force maintained at 114 N for at least 1.5 hours. The stress concentration zone then transferred to
128 point 3 after 3 hours (Fig. 1), due to progressive cracking of mechanical bridges that hindered crack
129 propagation at point 2. The microstructure did not evolve when the last map was recorded after 4
130 hours at 114 N (Fig. 2). The separation between each mechanical asperity is roughly 100 μm, from
131 defect 0 (notch tip) to defect 3.

132 Progressively, the stress transfer along the crack is accompanied by a decrease of the tensile
133 stress field around the crack tip, with final values around 250 MPa. Further propagation of the crack
134 would require building a sufficient stress field at the tip of the crack to break the asperity on which it
135 is now pinned. The sub-critical propagation is promoted by the initial high tensile stress surrounding
136 the crack tip at point 1. When the loading force is maintained constant, and as long as the stress at the

137 crack tip exceeds the fracture properties of the interface, the crack propagates by jumping from one
138 defect to the other. It took about 2 hours to jump from point 2 to point 3.

139 To better visualize the stress distribution around the crack tip after sub-critical crack
140 propagation, a higher resolution fluorescence map was recorded in the area containing the crack while
141 the load was maintained at 114 N for 40 minutes, for comparison with microstructural SEM imaging.
142 The contrast between the area under tension and compression reveals the crack path and can be
143 overlaid with the SEM image of the same area (Fig. 3).

144 The crack path up to its tip (point 3) can be located from a sharp contrast between the
145 mechanically relaxed areas (yellowish tones) above the crack and those under tensile stress below it. A
146 major crack is outlined by yellowish tones next to reddish ones, and secondary cracks branch on it and
147 separate reddish areas from bluish ones. Between the notch and the crack tip, the points where crack
148 pinning and stress accumulation was noted during the test (Fig. 2) correspond to specific defects in the
149 microstructure as observed from the surface. At point 1, the crack had to bypass a cluster of misaligned
150 platelets, resulting in a diverted trajectory and stress contrast that contours both sides of the cluster. At
151 point 2, branching is observed with a secondary crack seemingly appearing below a larger-than-average
152 alumina platelet. At point 3, the crack is again pinned on a cluster of misoriented alumina platelets, and
153 a very local stress concentration is observed before the experiment was stopped after 4 hours at a force
154 of 114 N.

155 The nature of microstructural defects involved in crack deviation and branching was further
156 characterized using *in situ* SEM imaging of another SENB sample under stress. Similar behaviors and
157 features were observed with fast crack propagation steps separated by arrests. With *in situ* imaging, the
158 active cracks are open and can be directly visualized (Fig. 4) whereas in a posteriori characterization,
159 cracks are closed and their exact location in the complex contrasts of the microstructure is more
160 difficult to determine (Fig. 3). Although the sample used for *in situ* SEM was different from the one
161 used in fluorescence mapping, similar microstructural features are associated to crack deviation and

162 branching.

163

164 **Discussion and concluding remarks**

165 When SENB four-point experiments are performed on non-textured ceramics, the crack initiates
166 and propagates along the notch plane. Strong crack deviation from the notch plane here is due to the
167 anisotropic microstructure of the material. This crack deviation is linked to the contrasted mechanical
168 properties between alumina platelets (brick) and the aluminosilicate glass phase (mortar) (Bouville et
169 al., 2014; Henry et al., 2020; Jackson et al., 1988; Kakisawa and Sumitomo, 2011). Optimization of
170 brick-and-mortar materials has thus far relied on mechanical tests on bulk samples and numerical
171 models that both assume structural homogeneity from the microscopic to the macroscopic level. This
172 may be true for the well-ordered nacre. However, the actual structure of nacre-like ceramics is more
173 complex, with heterogeneity in alumina platelet size and imperfect orientations during the fabrication
174 (Saad et al., 2020). Simulations based on actual microstructures have shown the importance of local
175 imperfections of the microstructure on the toughness of the composite (Marcinkowska, 2018; Radi et
176 al., 2021). Imaging of local stress around the propagating crack highlights the interaction of the crack
177 with the imperfections of the microstructure.

178 Fluorescence maps revealed important stress gradients near the tip of the notch, with variations of
179 up to 300 MPa in a few micrometers. This is lower than the strength of platelets around 1 GPa
180 (Doitrand et al., 2020; Feilden et al., 2017), but higher than the yield strength of aluminosilicate
181 glasses used as mortar along the interfaces (Bouville et al., 2014; Saad et al., 2020), with values lower
182 than 100 MPa (Sheikh et al., 2019). Even if a calculation of local stresses considers hydrostatic
183 stresses and is based on the hypothesis of isotropy in piezospectroscopic coefficient (thereby not
184 capturing the texture of this material), the stress values are coherent. These calculated stress values
185 likely reflect the effects of the textural heterogeneities on local mechanical properties. In particular,
186 bundles of alumina platelets oriented at high angles with respect to the main orientation of platelets in

187 the microstructure are likely to play a role because platelets cannot be broken along a mechanically
188 strong direction of the platelets. Thus the crack has to deviate along an easier path, the weaker
189 aluminosilicate interphase (Figs. 3 and 4). This is consistent with the results of numerical simulations
190 that used the observed microstructures to show cracks propagate at higher stress around bundles of
191 misoriented platelets, thus contributing to toughening the composite (Radi et al., 2021). These defects
192 likely contribute to the increasing toughness (R curve) of the nacre-like alumina. High-stress gradients
193 are clearly associated with defects, in spite of the intrinsic limitation that the observation is performed
194 on the surface of a 3D sample.

195 *In situ* fluorescence mapping of stress and SEM imaging during mechanical tests prove useful to
196 identify local mechanisms and microstructural features that contribute to toughening in these
197 bioinspired composites. Future improvements will focus on measurements that can be combined on
198 the same sample and use the orientation information provided by EBSD to locally analyze the stress
199 from the scale of the individual components (here the alumina platelets) to the scale of the aggregate
200 (platelets bundles) and of the bulk sample. Besides using fluorescence, stress can also be characterized
201 using Raman spectroscopy in non-luminescent materials. This approach could be applied to other
202 advanced ceramics like zirconia, or natural ceramics like apatite and calcium carbonates (Loh et al.,
203 2020; Perrichon et al., 2017; Pezzotti and Sakakura, 2003; Pezzotti et al., 2012; Wei et al., 2020; Zhu
204 and Pezzotti, 2011).

205

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211 **Conflict of interest.** The authors declare no conflict of interest.

212

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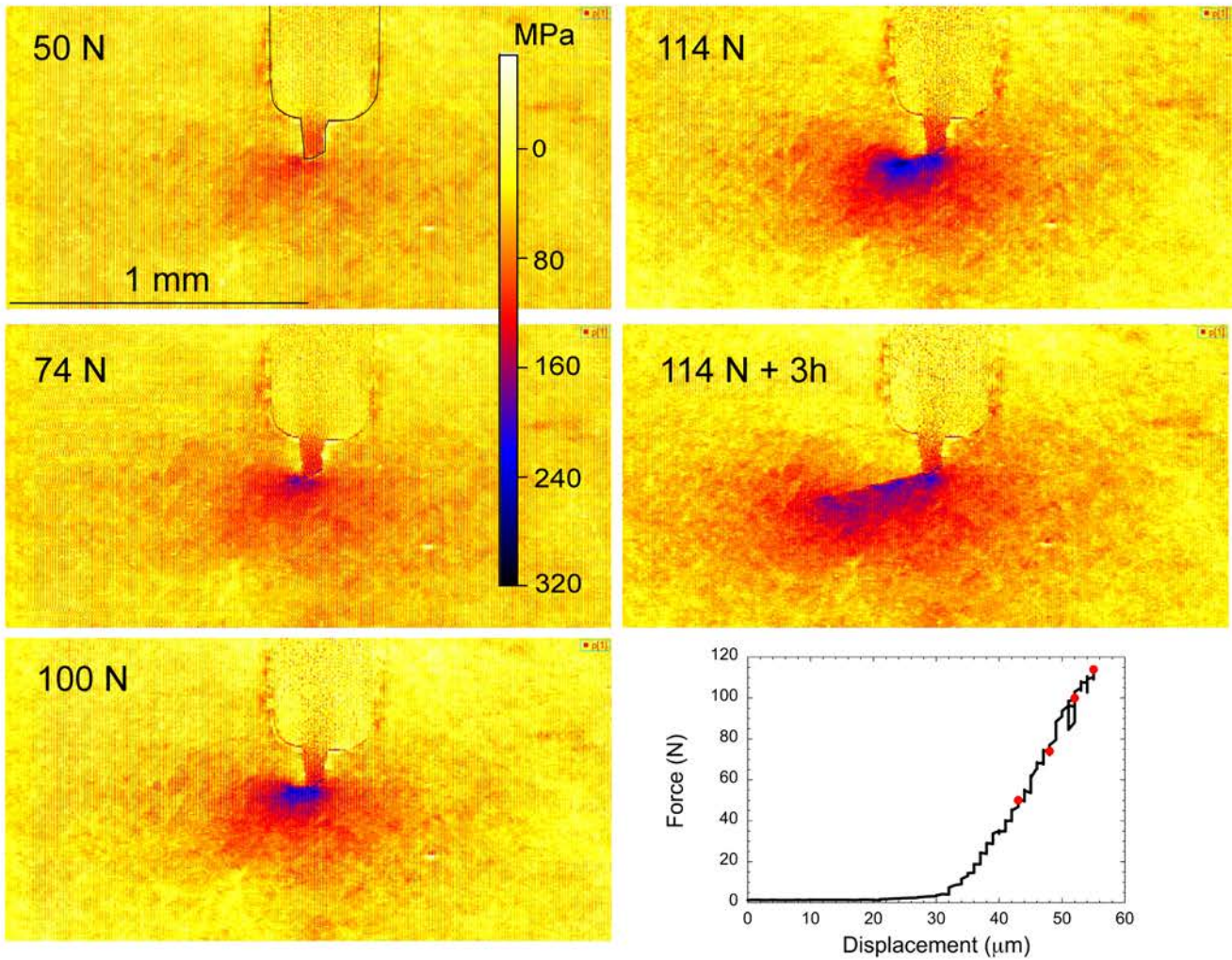
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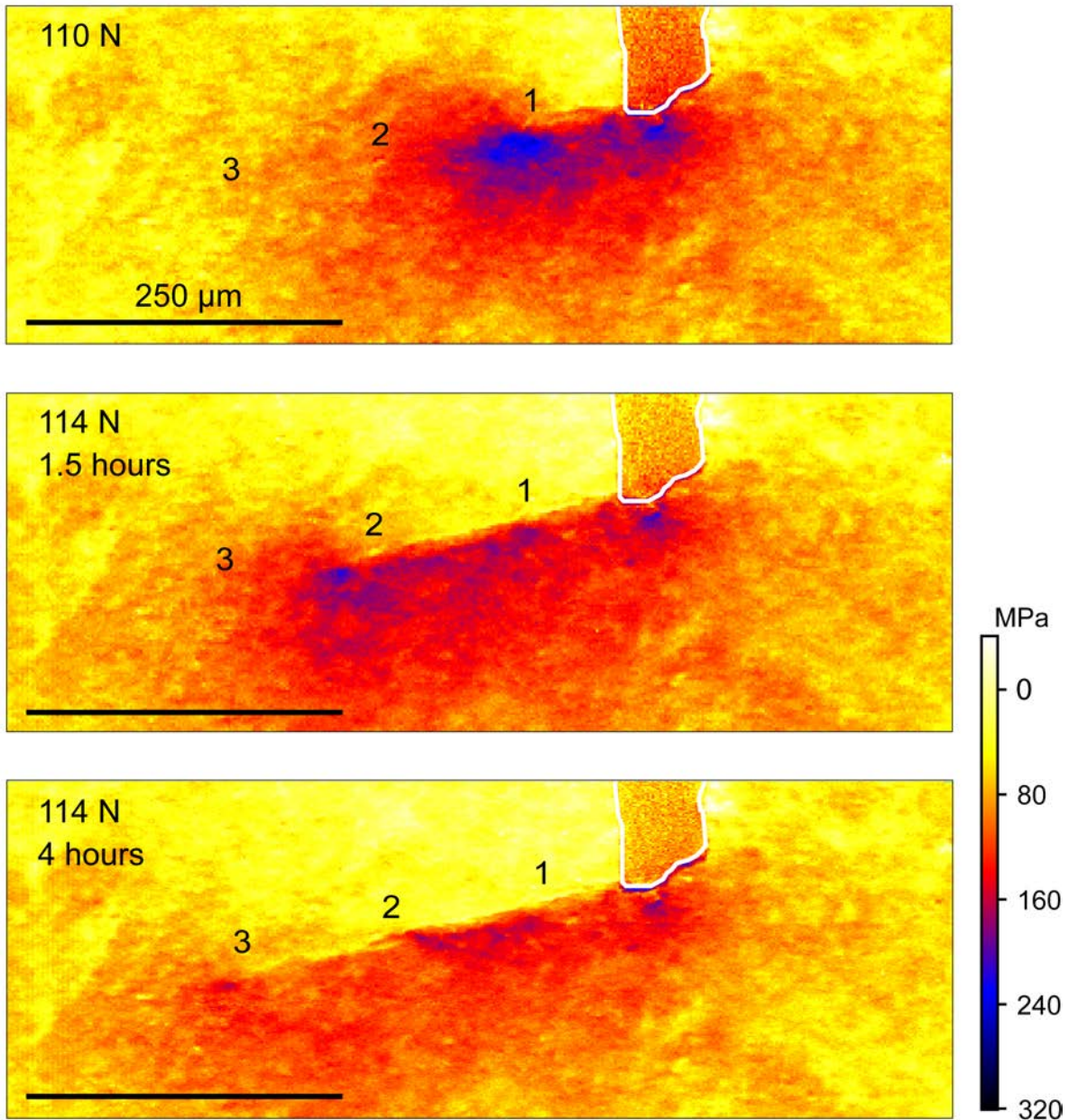
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272 *Figure 1. Evolution of the stress measured using Cr^{3+} fluorescence in SENB under increasing force*
 273 *(values in N) up to crack initiation and propagation at 114N. Corresponding points are indicated by*
 274 *the red dots on the force-displacement curves (bottom right). The stress field locates and concentrates*
 275 *on the left side of the notch materialized by the thin black outline on the 50N stress map. As the load*
 276 *increases, the stress locally reaches values of ~250 MPa. The thin black line outlines the position of the*
 277 *notch. © (2022) H. Saad et al. (10.6084/m9.figshare.20072111) CC BY 4.0*
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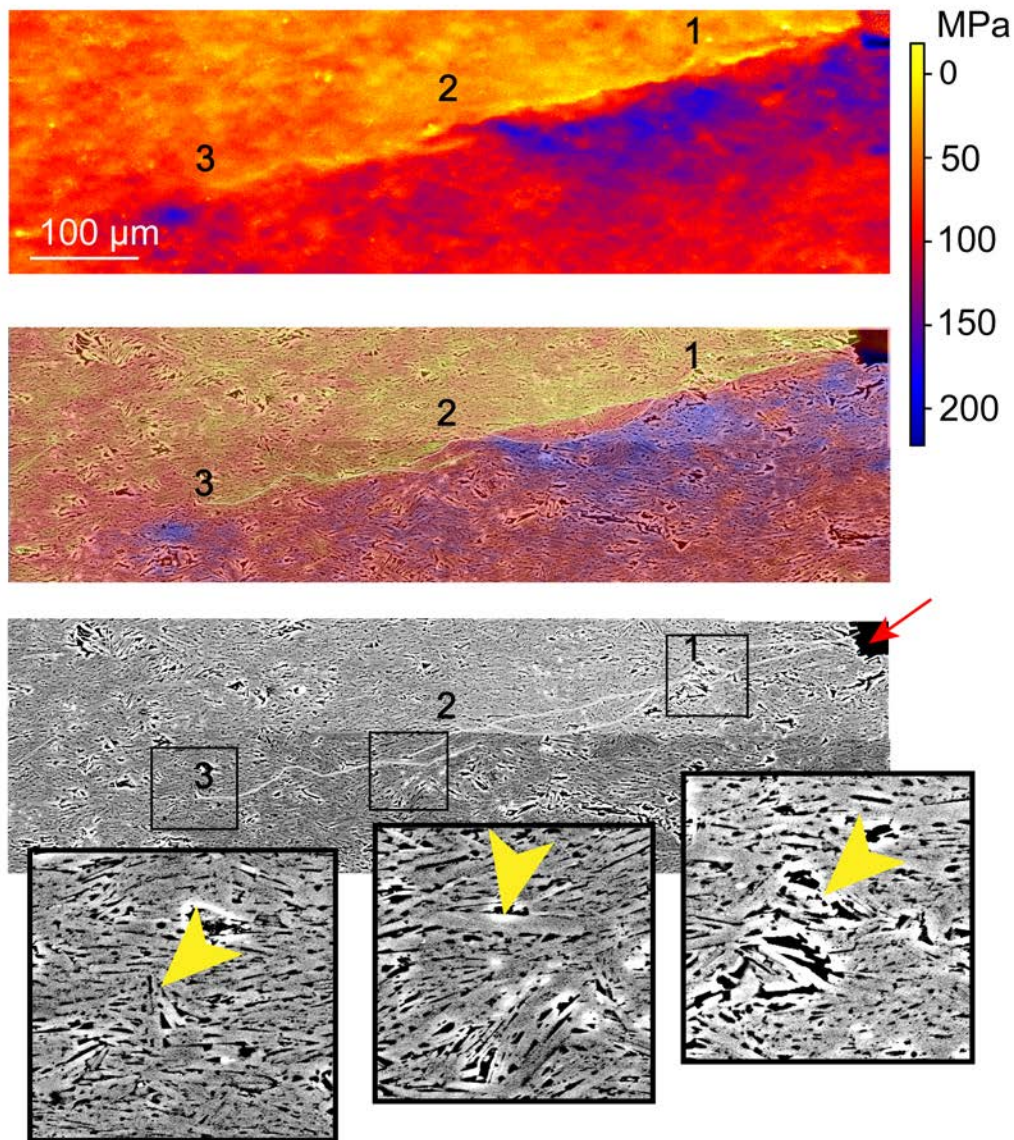
281 *Figure 2. Sub-critical crack propagation. The high tensile stress field present at the crack tip is*
 282 *highlighted. Crack propagation is accompanied by a reduction of the stress intensity at the crack tip.*

283 *The stress field is pinned on mechanical asperities labeled 1 to 3 that contribute to the crack*

284 *propagation resistance of the material. The white line outlines the position of the notch. © (2022) H.*

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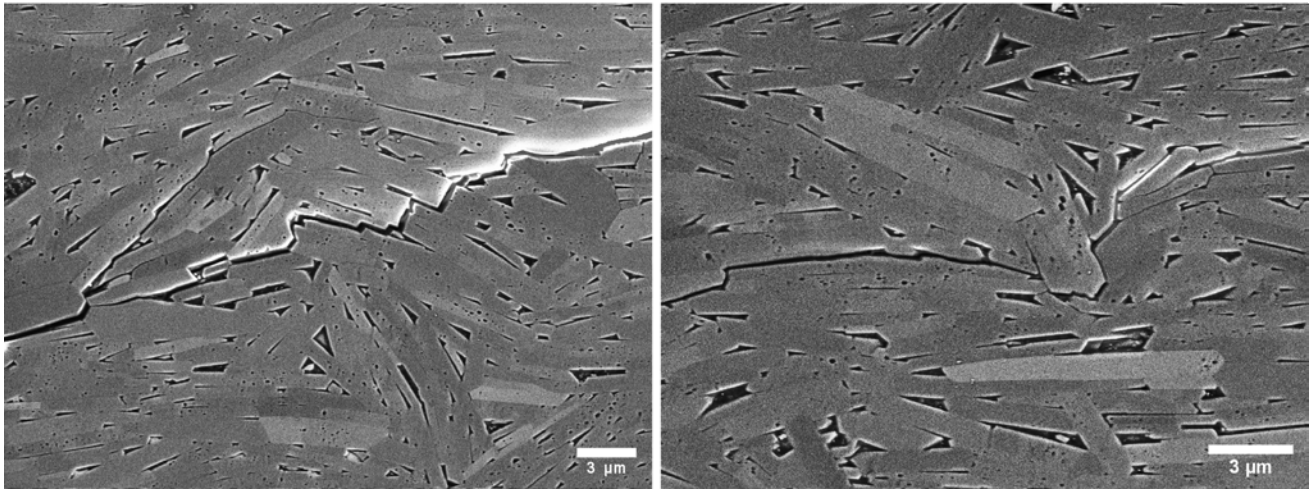
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288 *Figure 3. Crack path and stress field obtained by mapping the stress field (top) is overlaid with the*
 289 *SEM mosaic image of the same area (bottom). Microstructural defects labeled 1 to 3 are the same as*
 290 *in Fig. 2. Enlarged SEM images reveal that defects (yellow arrows) that cause toughening are either*
 291 *large alumina platelets (point 2) or bundles of highly misoriented alumina platelets (1 and 3). The*
 292 *color scale is modified from Fig. 2 to enhance contrast. The notch tip (red arrow) appears black in*
 293 *the upper right corner of the SEM image. White lines on SEM images highlight the main cracks*
 294 *identified from sharp contrasts in the stress map. © (2022) H. Saad et al.*
 295 *(10.6084/m9.figshare.20072111)*

296



297

298 *Figure 4. In situ SEM images of the open crack show crack branching (left) or deviation (right) around*
299 *interlocking bundles of alumina platelets. These defects slow down crack propagation, and are*
300 *associated with the mechanical asperities and crack pinning revealed by fluorescence imaging (Figs. 2*
301 *and 3). © (2022) H. Saad et al. (10.6084/m9.figshare.20072111)*

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