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1 A New Method to Rapidly and Accurately Assess the Mechanical Properties of Geologically
2 Relevant Materials

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8 Abstract

9 A new indentation-based method was developed that will impact and facilitate the
10 elastic property measurements of rocks and minerals, especially those possessing unusual
11 deformation behavior including brittle materials and those with complex architectures. The
12 novel feature employed is a metallic film that uniformly transfers the load from the indenter
13 tip to the sample. The film also absorbs the damage caused by the penetrating indenter,
14 shielding the material from highly localized deformation that can impact its response to
15 loading. Many geologically relevant materials have resisted traditional indentation testing
16 because they are either brittle in nature or possess highly anisotropic architectures, such as
17 layered or lamellar structures. In both cases, the highly localized deformation from direct
18 indentation significantly affects the indenter unloading stiffness, from which the elastic
19 properties are determined. The indirect indentation method developed here, demonstrated
20 accurate determination of the elastic properties of many common geologic materials as well
21 as materials that have resisted elastic characterization such as galena and talc.

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24 **Introduction**

25 The elastic properties of rocks and minerals have long been central to understanding and
26 predicting the mechanical behavior of the Earth from a regional to a planetary scale. Recent
27 decades have seen the development of many skillful techniques capable of measuring elastic
28 properties, including their dependence on temperature and/or pressures relevant from the
29 Earth's crust to its core. These include electromagnetic radiation (EMR), acoustic emission
30 (AE), micro-seismic (MS), and many others, see (Angel et al., 2009) for a recent review.
31 Given these significant advances, the accuracy of deformation models continues to depend on
32 accurate property inputs, which should be reflective of the regional rocks and minerals
33 relevant to the model. In this regard, the local environmental conditions that these materials
34 form and exist in can influence their properties as a function of composition, structure,
35 hydration, and other characteristics (Ersoy and Waller, 1995; Na et al., 2017; Raisanen, 2004;
36 Sun et al., 2017; Tandon and Gupta, 2013). Thus, the mechanical behavior of many geologic
37 relevant materials can vary appreciably from region to region (Atkinson, 1976; Blackman et
38 al., 2002; King Hubbert, 1951; Meyers and Chawla, 2009; Riecker, 1984), warranting the
39 need for simple, rapid and accurate tests to provide this information. Indentation has long
40 been a popular technique to carry out elastic property measurements due to the minimal
41 sample preparation and the rapid collection of numerous data points, important to robust
42 statistical analyses (Oliver and Pharr, 1992; Oliver and Pharr, 2004). It also enables unrivaled
43 spatial mapping of elastic properties to ascertain variations in sample composition,
44 microstructure, and phases (Constantinides et al., 2006; Randall et al., 2009). An additional
45 benefit is that indentation does not require specific expertise or access to special facilities and
46 can be performed at most universities and research institutions. The basic premise involves
47 pressing a sharp diamond tip of well-known geometry into the material while independently
48 recording the load and indentation depth. The resistance to penetration is represented by

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49 hardness (H) while the elastic behavior (E) is related to the slope of the unloading curve, the
50 unloading stiffness (S), as in the following relations (Oliver and Pharr, 1992):

51
$$H = \frac{P}{a} \quad \text{and} \quad S = \frac{dP}{dh} = E \frac{2}{\sqrt{\pi}} \sqrt{a}, \quad (1)$$

52 where p is the applied indenter load, a is the contact area between the indenter and sample, h
53 is the indenter displacement into the sample surface and E is the elastic modulus of the
54 sample. By oscillating the indenter tip during penetration, both properties can be monitored
55 continuously as a function of depth, termed continuous stiffness (Pharr et al., 2009). The
56 typical indentation test ranges from the nanometer to micron scale and can infer local
57 variations in elastic behavior including; compositional and structural changes, different
58 material phases, interfaces, and many other varying characteristics. (Hintsala et al., 2018).

59 The extraction of the elastic modulus from indentation can be problematic for materials
60 that are brittle or that possess unusual deformation behavior (Chen et al., 2018; Pharr and
61 Bolshakov, 2002), which many rocks and minerals do. For example, brittle materials that
62 cannot plastically strain in response to indenter penetration instead generate cracks or other
63 defects. These form during the loading cycle and dissipate energy through the creation of new
64 surface. They have a significant effect on the unloading stiffness (S) as they consume stored
65 elastic energy that would normally push back on the indenter as it is withdrawn. Thus, the
66 extracted elastic modulus is measured to be lower than it actually is. Many geologic materials
67 possess complex architectures whose deformation can alter the contact area with the indenter.
68 For example, lamellar structured materials such as kyanite can deform by large-scale
69 cleavage and sliding of layers relative to one another (Boland et al., 1977; Doukhan and
70 Christie, 1982; Doukhan and Paquet, 1982; Lefebvre, 1982; Lefebvre and Menard, 1981;
71 Mikowski et al., 2007; Mikowski et al., 2008; Raleigh, 1965). When indented, the layers
72 themselves do not appreciably strain elastically or plastically. Instead, the sliding translates
73 the concentrated indenter load a distance from the indent's normal area of influence. This

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74 wholesale layer sliding will then alter the contact area with the indenter tip, and therefore,
75 the elastic pushback the material would exert when the tip is withdrawn. In other directions,
76 the applied load can cause layers to separate, essentially creating surface and disrupting the
77 transfer of load. In all of these cases, the extreme local deformation caused by the indenter
78 penetration results in inelastic damage/deformation that affected the unloading stiffness and
79 therefore, the extraction of the elastic response. There are many other geologic materials
80 possessing complex architectures, silicates for example, which will encounter similar
81 problems. The Indirect Indentation Method (IIM) can be employed to mitigate these issues
82 (Chen et al., 2018). Here, the thin metallic film is deposited serves to absorb this inelastic
83 damage while transferring the load to the material in a uniform manner. This enables the
84 unloading stiffness to reflect the materials actual elastic properties.

85 The indirect indentation method generates a film/substrate composite where both materials
86 contribute to the loading and unloading response. It uses the Zhou-Prorok thin film
87 indentation model to interpret and decouple the film/substrate composite response (Zhou and
88 Prorok, 2010a; Zhou and Prorok, 2010b). This model leverages the King modified Doerner
89 and Nix empirical function (Doerner and Nix, 1986; King, 1987), which is rearranged into
90 the basic form of the inverse rule of mixtures with elastic compliance (Reuss, 1929), see
91 Equation 2. Here, E is the elastic modulus obtained from the indenter, E_f and E_s are the
92 elastic moduli of the film and substrate respectively, ν_f and ν_s are the film and substrate
93 Poisson's ratios, t is the film thickness and h is the indent depth. The weighting factors, in
94 parentheses, are exponential terms that incorporate the Poisson's ratio of each material and
95 account for the lateral elastic interplay between the film and substrate. A principal feature of
96 IIM is that the main constants are all elastic properties of the film and substrate, which are
97 usually their bulk literature values. The model has shown to be adept at modelling the
98 composite elastic response of a penetrating indenter obtained by the Oliver and Pharr method

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99 (Oliver and Pharr, 1992) for numerous film/substrate composites (Chen et al., 2018; Liu et al.,
100 2011a; Liu et al., 2011b; Sullivan et al., 2015; Sullivan and Prorok, 2015; Xu et al., 2018;
101 Zhou and Prorok, 2010a; Zhou and Prorok, 2010b).

$$102 \quad \frac{1}{E} = \frac{1}{E_f} (1 - e^{-v_s(t/h)}) \cdot \left(\frac{E_f}{E_s}\right)^{0.1} + \frac{1}{E_s} (e^{-v_f(t/h)}) \quad (2)$$

103 Two important aspects of IMM are (1) rearranging Equation 2 to decouple the film and
104 substrate contributions and (2) taking advantage of the fact that the weighting factors in
105 Equation 2 are actually specific types of hyperbolic functions. The first is accomplished by
106 dividing both sides by the weighting factor on the film compliance $(1 - e^{-v_s(t/h)})$ as,

$$107 \quad \frac{1}{E} \left[\frac{1}{(1 - e^{-v_s(t/h)})} \right] = \frac{1}{E_f} \cdot \left(\frac{E_f}{E_s}\right)^{0.1} + \frac{1}{E_s} \left[\frac{(e^{-v_f(t/h)})}{(1 - e^{-v_s(t/h)})} \right]. \quad (3)$$

108 The second involves a convenient property of hyperbolic functions in that they approach an
109 asymptote that is easily approximated with a simple linear function. These were found to be
110 $0.5 + h/t$ for the bracket on the left side and $1/2 + v_f/v_s + (h/t)/v_s$ for the bracket on the right
111 (Batyuskov, 2001), see Equation 4. The result is a linear function with a slope of $1/(v_s E_s)$.

$$112 \quad \frac{1}{E} \frac{1}{(0.5 + h/t)} = \frac{1}{E_f} \cdot \left(\frac{E_f}{E_s}\right)^{0.1} + \frac{1}{E_s} \left(\frac{1}{2} - \frac{v_f}{v_s} + \frac{h/t}{v_s} \right). \quad (4)$$

113 The IMM procedure simply involves multiplying the composite modulus from the
114 indenter by the film-side weighting factor to obtain the reduced modulus, left-side of
115 Equation 3 or 4. As the indenter penetrates the film and approaches the film/substrate
116 interface, where $h/t = 1$, the reduced modulus approaches its linear asymptote, whose slope is
117 directly related to the substrate's elastic modulus. The film elastic modulus and Poisson's
118 ratio are contained in the constant/intercept of the linear form in Equation 4 and do not
119 influence the magnitude of the slope. Thus, by depositing a thin metallic film and confining
120 the penetration only to the film, IMM can directly measure the elastic properties of a material
121 in the absence of extreme deformation events/behavior.

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122 The development of geologic-based deformation models depends on knowledge of the
123 constituent material properties for validation and prediction accurately. IIM is an ideal
124 technique for rapid determination of elastic properties for many geologic materials, which
125 can more readily be investigated as a function of composition, structure, hydration, or other
126 physical characteristic. The aim of this work is to demonstrate its ease of use and
127 applicability to standard geologic materials as well as those whose elastic properties have
128 been difficult to ascertain. Talc for example is considered one of the softest minerals as its
129 hardness defines the lowest value on Mohs' Hardness scale (Gerberich et al., 2015; Mohs,
130 1925). However, this intrinsically weak behavior has resisted efforts to measure its true
131 elastic properties, which have scarcely been reported on in the literature. Results of IIM will
132 be presented on typical materials found in geologic settings as well as reliable measurements
133 of elastic modulus challenging materials such as talc and galena.

134

135 **Experimental Methodology**

136 **Sample Preparation**

137 Ten geologic relevant materials were chosen for study; naturally occurring kyanite,
138 feldspar orthoclase, dolomite, microcline, obsidian, galena, beryl calcite, quartz and talc
139 (obtained from Scott Resources). These materials were chosen to demonstrate the indirect
140 indentation method on minerals possessing a wide variety of material properties and
141 deformation behavior. Table 1 lists the common literature values of their elastic moduli and
142 Poisson's ratios. Here, citations were chosen that (1) represented the average of the reported
143 elastic data and (2) used methods other than indentation to determine their values. Samples of
144 each were mounted in epoxy and rough polished with sand paper and then fine polished with
145 colloidal Al₂O₃ media suspensions to produce mirror-like finishes, typically less than 5 nm
146 RMS roughness over 1 mm² area. X-ray diffraction was performed to confirm each material

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147 and determine the crystallographic orientation being tested for the single crystal samples.
 148 Chromium was chosen as the metallic film to absorb the inelastic damage from the
 149 penetrating indenter. It is ductile and has a Poisson's ratio of 0.21 (Samaonov, 1968). It is
 150 also easily deposited via sputter deposition (Sullivan and Prorok, 2015) and can wet a wide
 151 variety of materials to form a strong interface (Ohring, 2001). Films were deposited using a
 152 Denton Discovery 18 sputtering system with substrate rotation to ensure a uniform coating.
 153 Process parameters were set at a DC power of 200 W and an argon gas flow of 25 sccm
 154 (Liang and Prorok, 2007; Wang et al., 2007; Wang and Prorok, 2008). Chromium film
 155 deposition was performed on all samples simultaneously to ensure all minerals possessed
 156 films of consistent thickness and morphology. Film thickness was measured on film/mineral
 157 cross-sections with a JEOL 7000F scanning electron microscope (SEM). Indent images were
 158 also captured by the SEM using secondary electron imaging.

Table 1: Literature values of the elastic modulus and Poisson's ratios of the materials involved and their comparison with results from the indirect indentation method.

Material	Literature Values			Indirect Indentation Method
	E (GPa)	ν	[ref]	E (GPa)
Chromium	190 ± 8	0.210	(Samaonov, 1968)	---
Kyanite (100)	227 ± 30	0.290	(Mikowski et al., 2008; Whitney et al., 2007)	228.6 ± 11
Feldspar Orthoclase (002)	89 ± 7	0.240	(Christensen, 1996; Whitney et al., 2007)	77.0 ± 2
Dolomite (polycrystalline)	53-85	0.200	(Grady et al., 1976; Viktorov et al., 2014)	93.6 ± 4
Microcline(polycrystalline)	69	0.245	(Christensen, 1996; Zhou et al., 2016)	73.8 ± 3
Obsidian (amorphous)	65 ± 2	0.185	(Bass, 1995; Husien, 2010)	66.8 ± 2
Galena (002)	--	0.270	(Gercek, 2007)	58.2 ± 2
Beryl (amorphous)	212	0.039	(Yeganehhaeri and Weidner, 1989; Yoon and Newnham, 1973)	214.2 ± 7
Calcite (104)	69 ± 2	0.322	(Fiquet et al., 1994; Redfern and Angel, 1999)	66.3 ± 2
Quartz	107 ± 3	0.079	(Bass, 1995; Gercek, 2007)	97.4 ± 4
Talc	--	0.268	(Bailey and Holloway, 2000)	218.7 ± 6

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Mechanical Testing

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Data Analysis

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The mechanical response of the samples was interrogated with an MTS Nanoindenter XP with a Berkovich diamond tip operated under continuous stiffness mode (CSM). Indents were performed directly on the polished mineral samples and indirectly on the Cr/mineral composites. Here, the elastic modulus was measured as a function of penetration depth. The CSM testing frequency was set at 45Hz with a harmonic displacement target of 2nm and was conducted under a 0.05 nm/s thermal drift rate threshold. Each sample was indented in a 5 × 5 array with a 100 μm spacing between indents. The elastic modulus was determined from the unloading stiffness (Oliver and Pharr, 1992). The mean elastic modulus of the 25 indents on each sample was reported as a function of indenter depth with error bars representing one standard deviation. Micrographs of indents were acquired by the SEM.

The Indirect Indentation Method (Chen et al., 2018) was employed to determine the elastic properties of the minerals. The first step was to multiply the mean composite elastic modulus obtained from the nanoindenter by the film-side weighting factor, as per the left-side of Equation 3. Here, the mineral's literature Poisson's ratio (ν_s) from Table 1 was used. In fact, any value can be assumed with negligible effects as long as it is within the normal range of 0.0 to 0.5. This results in a plot of the reduced modulus as a function of the normalized displacement (h/t), shown later. The slope near the film/mineral interface was determined in the h/t range from 0.6 to 1.0 (Chen et al., 2018) and was equated to $1/(E_s\nu_s)$, as per the right-side of Equation 4. The elastic modulus of the mineral (E_s) was then calculated using the Poisson's ratio (ν_s) assumed when obtaining the reduced modulus. The instantaneous slope of each reduced modulus data point was then used to calculate the indirect elastic modulus as a

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185 function of h/t .

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Results and Discussion

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Direct Indentation

189 The materials kyanite (100) and feldspar orthoclase were chosen to demonstrate the
190 applicability of the indirect indentation method to geologic materials. Both materials were
191 indented to a depth of 1 μm , and the measured elastic modulus was plotted as a function of
192 displacement into the surface, see Figures 1 (a) and (b). Both material curves begin at values
193 higher than their literature values, denoted by dashed lines and listed in Table 1, and then
194 decrease as the indenter penetrates the materials. The high values at low displacements are a
195 result of the indenter geometry. Although the Berkovich tip has a three sided pyramid
196 geometry, the tip is actually spherical with a radius of 30 to 50 nm. Thus, the first 50 nm or
197 so of contact has more projected contact area than the pyramidal geometry, artificially
198 inflating the modulus values. Neither curve in Figure 1 reaches a point where the modulus
199 remains consistent enough to estimate a value. In fact, the feldspar modulus drops well
200 below its literature value for the majority of the penetration depth. The behavior of both
201 materials indicate that inelastic processes are likely consuming energy that would normally
202 be stored elastically and recovered during unloading.

203 The load-displacement curves for directly indenting both materials can help explain the
204 varying modulus results, see Figures 2 (a) and (b). As the indenter penetrates both samples,
205 each material resists and absorbs the applied load through elastic and inelastic deformation
206 mechanisms that vary based on the material and its microstructure. The kyanite curve reveals
207 three discrete events where the displacement into the surface increased rapidly, denoted by
208 arrows. These can be explained by large-scale cleavage and sliding of its lamellar structure
209 (Boland et al., 1977; Doukhan and Christie, 1982; Doukhan and Paquet, 1982; Lefebvre,

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210 1982; Lefebvre and Menard, 1981; Raleigh, 1965). An electron micrograph of a direct indent
211 made on kyanite is shown in Figure 3(a). Here, the triangular residual indent is seen with
212 lamella sliding occurring on (100) where the face of the indenter tip contacts the material and
213 cleavage along the (010) plane occurring where its edge makes contact, labelled as (1) and
214 (2) respectively. The feldspar sample did not exhibit discrete events in its load displacement
215 response, Figure 2(b). However, an electron micrograph of a direct indent, Figure 3(b),
216 reveals that the material experienced significant cracking at the edges of the indenter tip, (3),
217 that continually increased as it penetrated. The large displacement events in the kyanite and
218 the cracking in the feldspar are inelastic defects that consume energy that would normally be
219 stored elastically in their absence. Thus, their irreversible formation influences the unloading
220 stiffness, and thereby elastic modulus, through reduced elastic recovery from the sample.
221 Directly indenting these materials to determine elastic response was hindered by the presence
222 and evolution of these inelastic deformation processes.

223

224 Indirect Indentation

225 A 940 nm thick chromium film was deposited on the samples to shield the materials from
226 the inelastic deformation processes caused by the penetrating indenter. The samples were
227 indented and imaged by the SEM, Figures 3 (c) and (d), and suggest the chromium film was
228 successful in absorbing the majority of inelastic deformation for both materials. Figures 4 (a)
229 and (b) show the elastic modulus results from the nanoindenter for the Cr/mineral
230 composites. Here the results are plotted as h/t to reflect how far the indenter has penetrated
231 into the film, which is the base form of the hyperbolic determination analysis. As the film
232 thickness was 940 nm, this scale is comparable to the results in Figure 1, which was plotted
233 as h for an indent depth of 1000 nm. The results from both materials show improved
234 progression to consistent elastic modulus values with increasing h/t over the direct

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235 indentation results. The kyanite sample appears to level off after an h/t of 0.5 is reached while
236 the feldspar sample has not yet reached a consistent value. The chromium/Kyanite value is
237 very similar to its literature value of 227 GPa (Mikowski et al., 2008; Whitney et al., 2007),
238 denoted by the dashed line in Figure 4. However, this is only a convenient happenstance as
239 the chromium film possesses a very similar elastic modulus and Poisson's ratio as kyanite,
240 see Table 1. Thus, the two materials, are elastically similar.

241 In order to begin the hyperbolic analysis of the indirect indentation model, the reduced
242 modulus was determined for both materials. This was accomplished by multiplying the mean
243 composite elastic modulus obtained from the nanoindenter by the film-side weighting factor,
244 as per the left-side of Equation 3. A Poisson's ratio of 0.24 and 0.29 was assumed for kyanite
245 and feldspar respectively, see Table 1. Figures 5 (a) and (b) plot the reduced modulus as a
246 function of h/t for both materials. After penetrating an h/t of 0.4 into the film, both materials
247 exhibited a strong linear behavior, denoted by the dashed lines. The slope of these lines was
248 determined by liner regression for all points in the h/t range of 0.6 to 1.0 as per the method
249 development (Chen et al., 2018). Slopes of $0.016833 \text{ GPa}^{-1}$ and $0.044482 \text{ GPa}^{-1}$ were found
250 for the kyanite and feldspar samples respectively. Using the assumed Poisson's ratios of each
251 material, the elastic modulus of the kyanite was calculated to be $228 \pm 11 \text{ GPa}$ and $77 \pm 2 \text{ GPa}$
252 for feldspar, which match rather well with the literature values in Table 1. The indirect
253 hyperbolic analysis was applied to each individual data point through the instantaneous slope.
254 This yielded a plot of the indirect measured elastic modulus of the mineral as a function of
255 penetration depth into the film (h/t), see Figures 6 (a) and (b). When both materials reach an
256 h/t of 0.6 or higher, their measured elastic modulus reaches a consistent value numerically
257 similar to their literature value, the dashed lines. This method was repeated for the other
258 geologic relevant materials listed in Table 1 with results plotted in Figure 7 (a). Here, the
259 reduced modulus for the remaining materials is plotted in Figure 7 (a) and their indirect

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260 measured modulus in Figure 7 (b). All of the materials attained a strong linear relationship in
261 the method's h/t range of 0.6 to 1.0, enabling their elastic modulus to be determined, see
262 Table 1. The indirect indentation results for all of the materials matched well with the
263 average literature values but also varied somewhat. These differences likely reflect variations
264 in compositions, microstructure and other characteristics that play a role in mechanical
265 behavior. All materials also exhibited very stable values of indirect modulus in the same h/t
266 range. In fact, all materials, with the exception of Galina, achieved stable values around an
267 h/t value of 0.4, which is less than half the film thickness. Finally, the indirect indentation
268 method was adept at obtaining very stable elastic moduli for Galena and Talc. These two
269 materials have been historically difficult to measure due to their wide variability in
270 deformation based on impurity content, hydration, pressure and unusual deformation
271 (Stixrude, 2002). In fact, the indirect indentation method would be an ideal method to
272 investigate and discern any variation in mineral elastic behavior as a function of composition,
273 formation conditions, and other physical differences.

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Implications

276 This work has demonstrated that indirect indentation method, which employs a
277 metallic film to absorb damage from the penetrating indenter, was adept at extracting elastic
278 moduli from several geologically relevant rocks and minerals. Measured values of elastic
279 modulus matched rather well with the literature for all materials tested. Tested materials
280 included natural rocks and minerals that possessed unusual deformation behavior including
281 brittle materials and materials with complex architectures and deformation behavior. In
282 addition to absorbing the highly localized deformation from the indenter, the metallic film
283 was successful at containing the sample deformation to within the elastic regime, enabling it
284 to be directly measured with IIM. Furthermore, IIM was also successful in establishing the

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285 elastic modulus of talc and galena, whose elastic behaviors have been difficult to ascertain.
286 This new method will enable geological scientists and engineers to rapidly determine the
287 elastic behavior of most rocks or minerals in a simple manner with a robust statistical
288 response. It will also facilitate investigations of their elastic properties as a function of
289 composition, structure, hydration, or other physical characteristic. This will undoubtedly
290 impact the development of geologic-based deformation models through knowledge of the
291 constituent material properties for validation and prediction accurately.

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446 Figure Captions

447 Fig. 1. Direct indentation results of Kyanite (a) and Feldspar (b) showing the elastic modulus
448 versus displacement into the surface. The dashed line represents the literature values of each
449 material given in Table 1.

450
451 Fig. 2. Load on sample versus displacement into surface for directly indenting Kyanite (a)
452 and feldspar (b). The arrows highlight several pop-in events during penetration.

453
454 Fig 3. Scanning electron micrographs in secondary electron mode of residual indents on the
455 kyanite, direct indent (a) and indirect indent (b), and feldspar, direct indent (c) and indirect
456 indent (d).

457
458 Fig. 4. The indirect indentation results of Kyanite (a), left, and Feldspar (b) showing the
459 elastic modulus versus displacement into the surface. The dashed line represents the literature
460 value of each material from Table 1.

461
462 Fig. 5. The calculated reduced modulus results of kyanite (a) and Feldspar (b). The dashed
463 line represents the linear regression of reduced modulus from 0.6 to 1.0 h/t.

464
465 Fig. 6. The calculated indirect substrate modulus results of kyanite (a) and Feldspar (b). The
466 dashed line represents the literature value for each material given in Table 1.

467
468 Fig. 7. The calculated reduced modulus (a) and indirect substrate modulus results (b) for the
469 other geological materials tested.

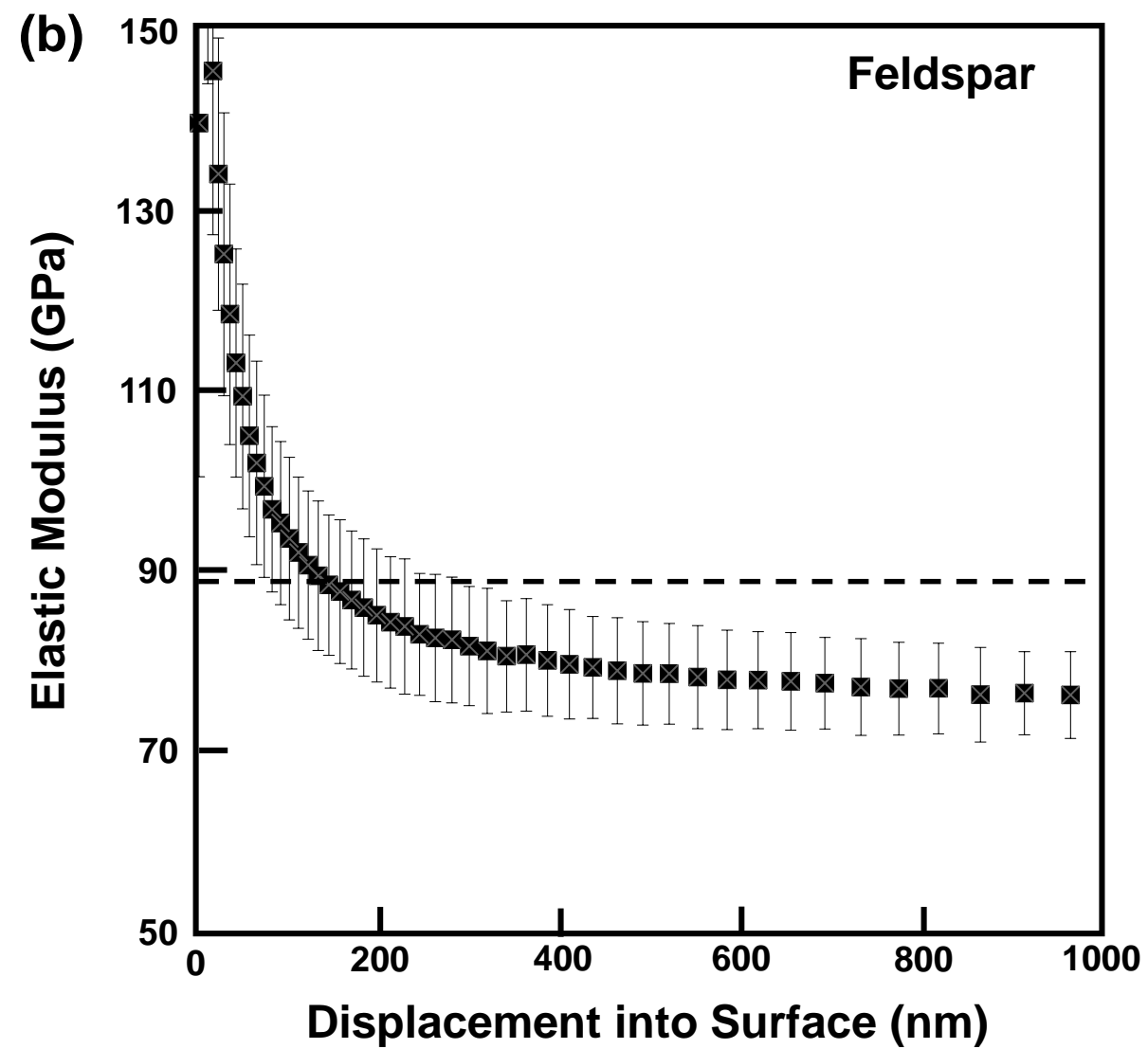
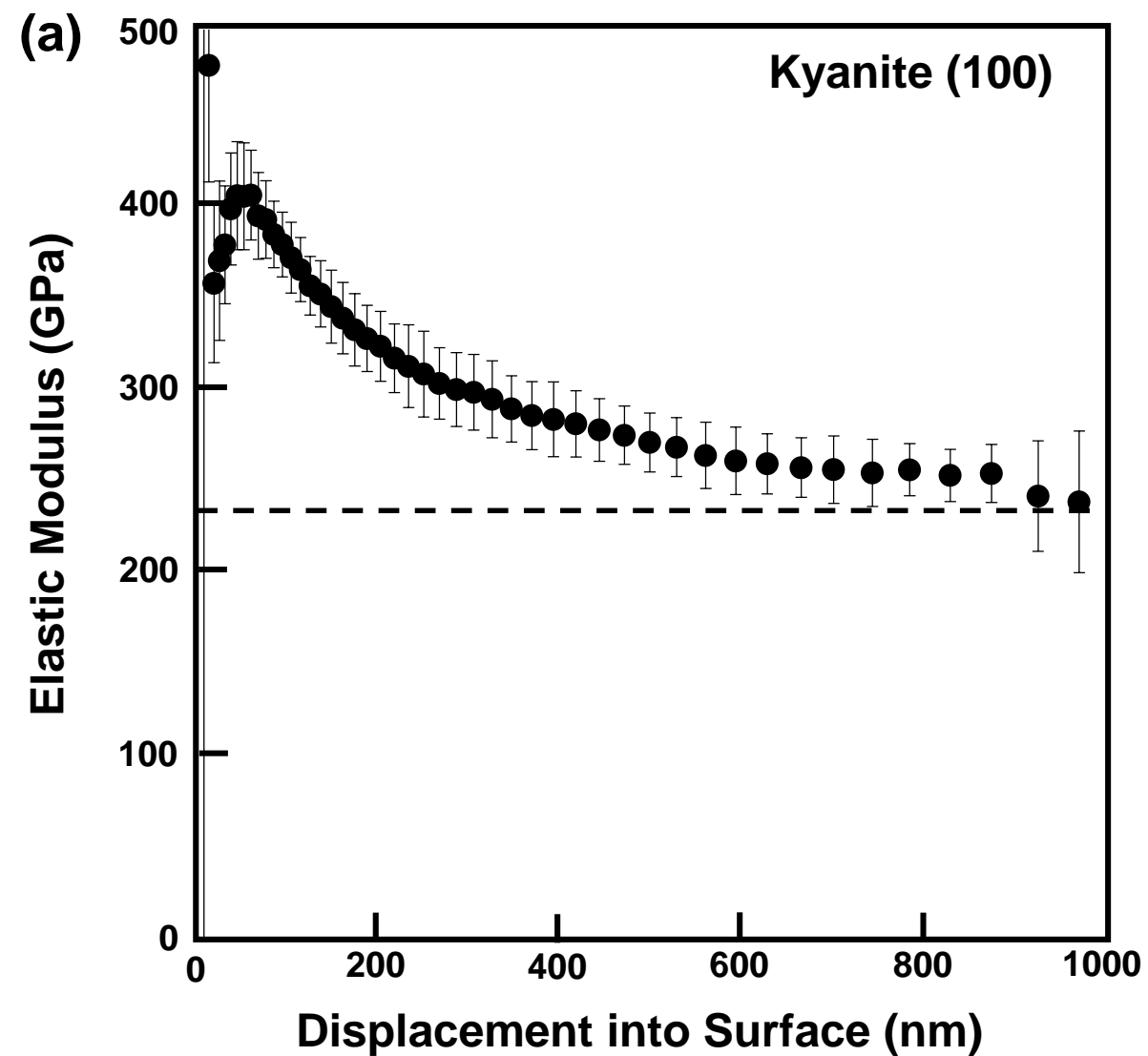


Figure 1

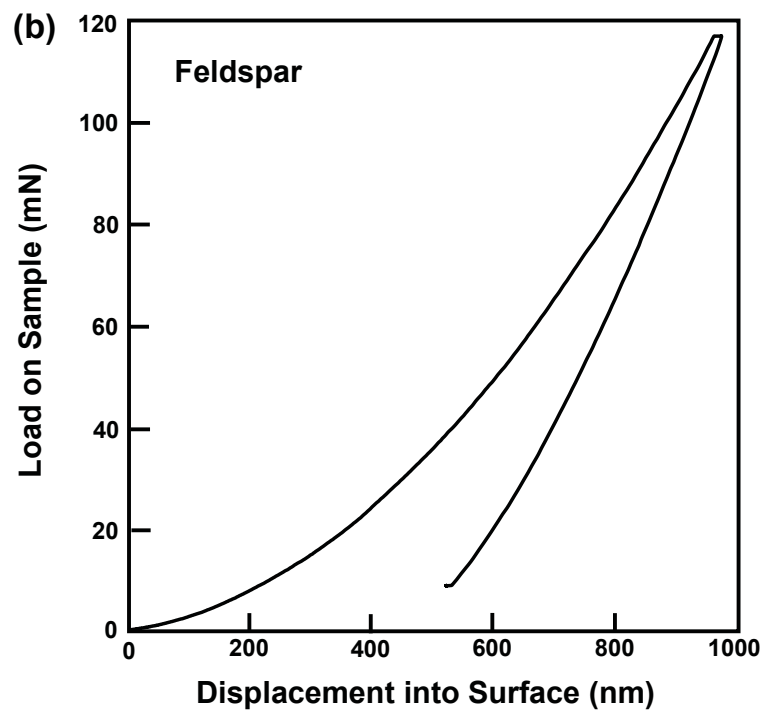
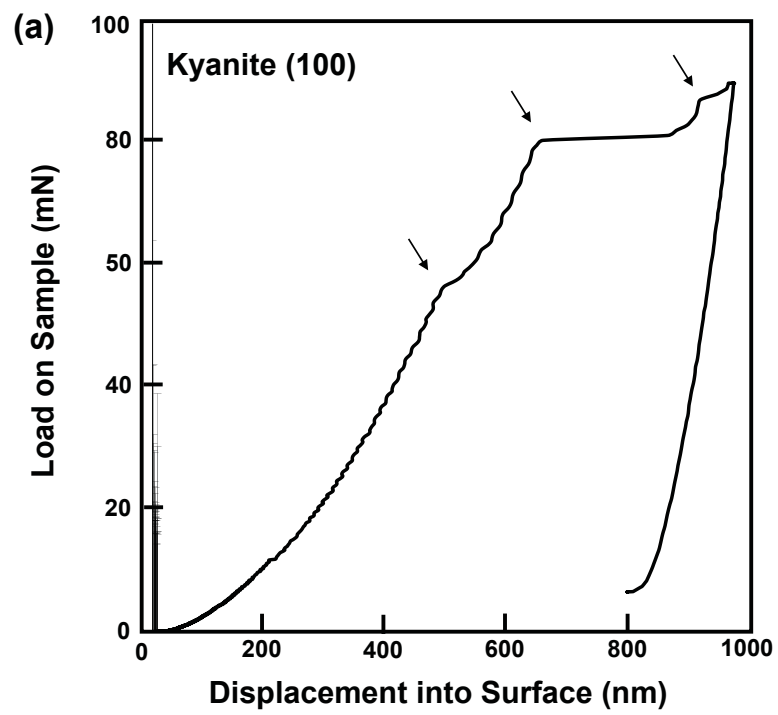


Figure 2

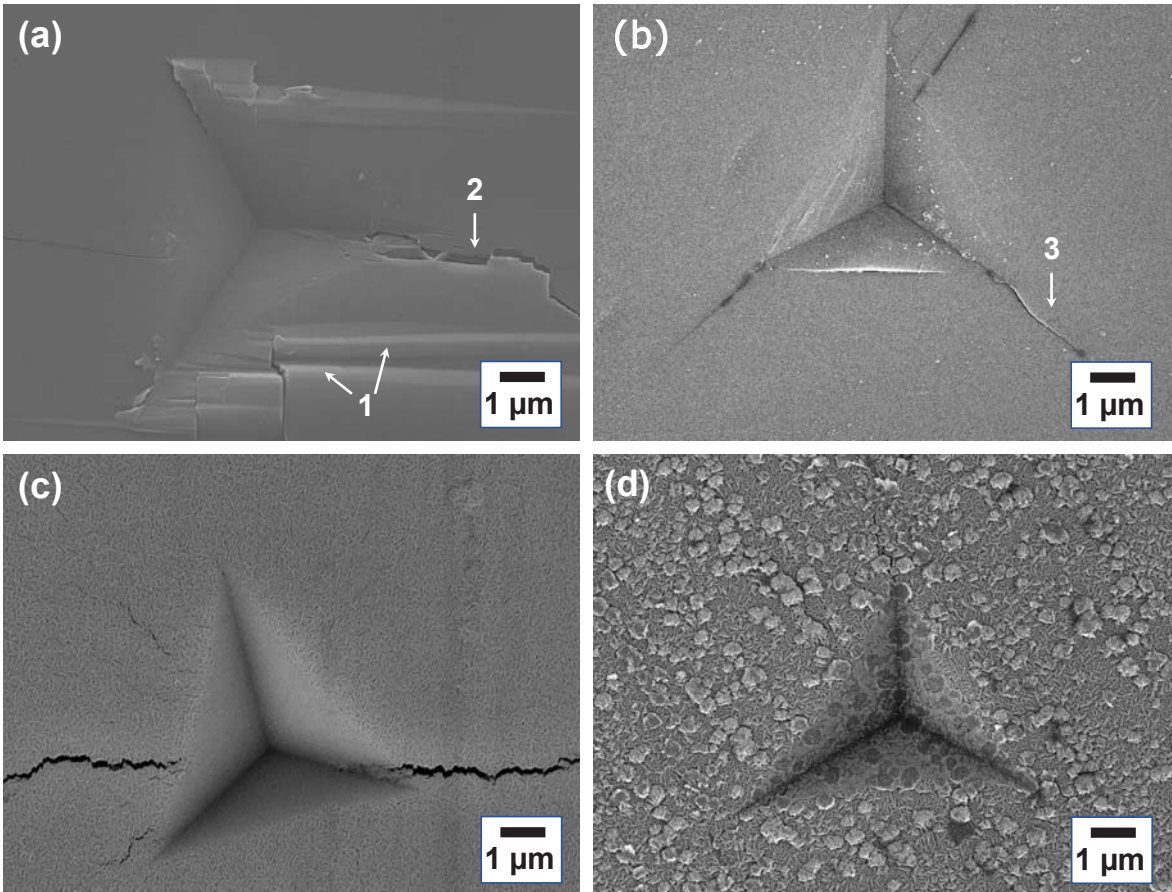


Figure 3

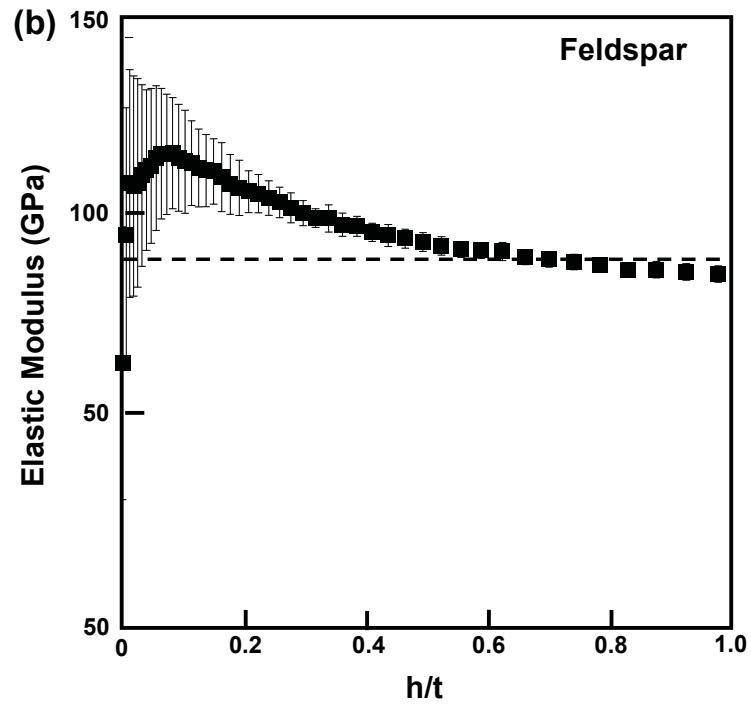
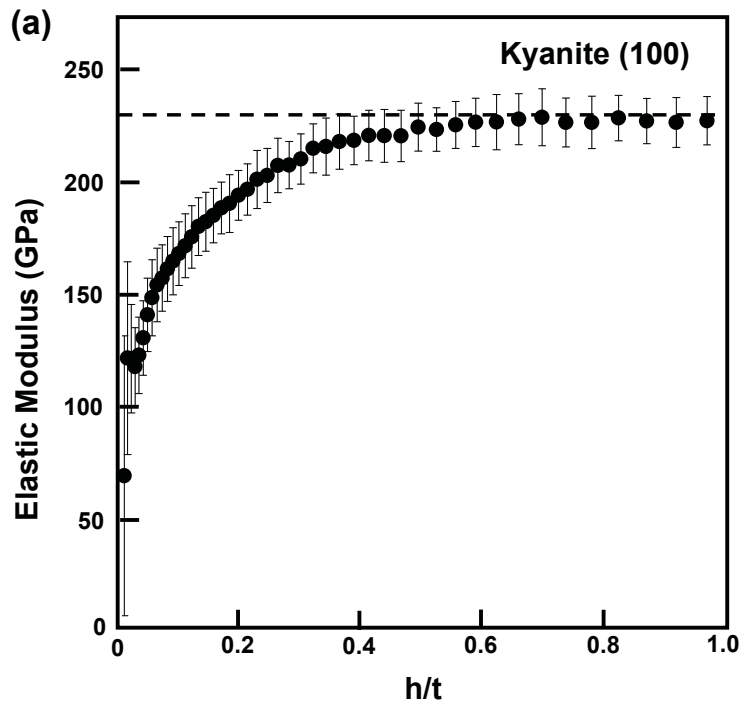


Figure 4

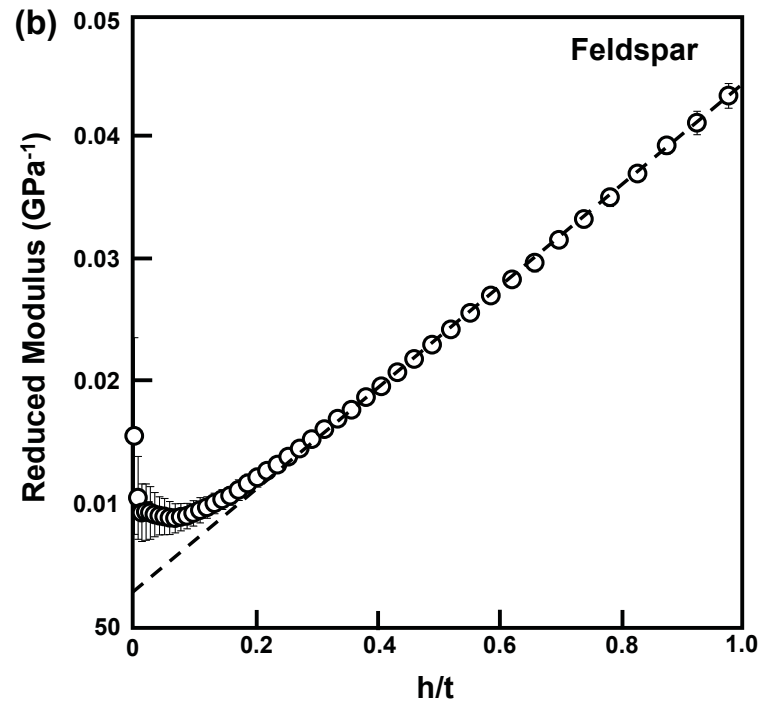
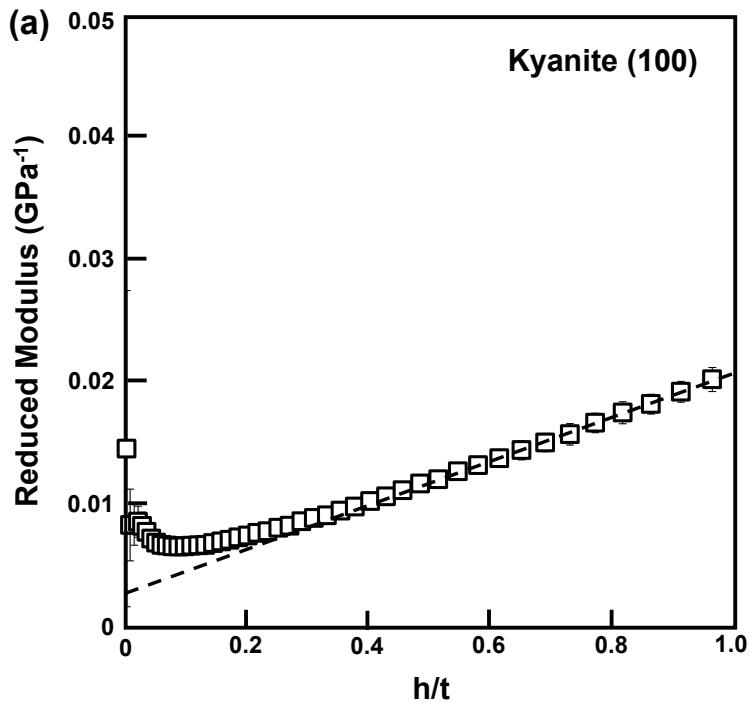


Figure 5

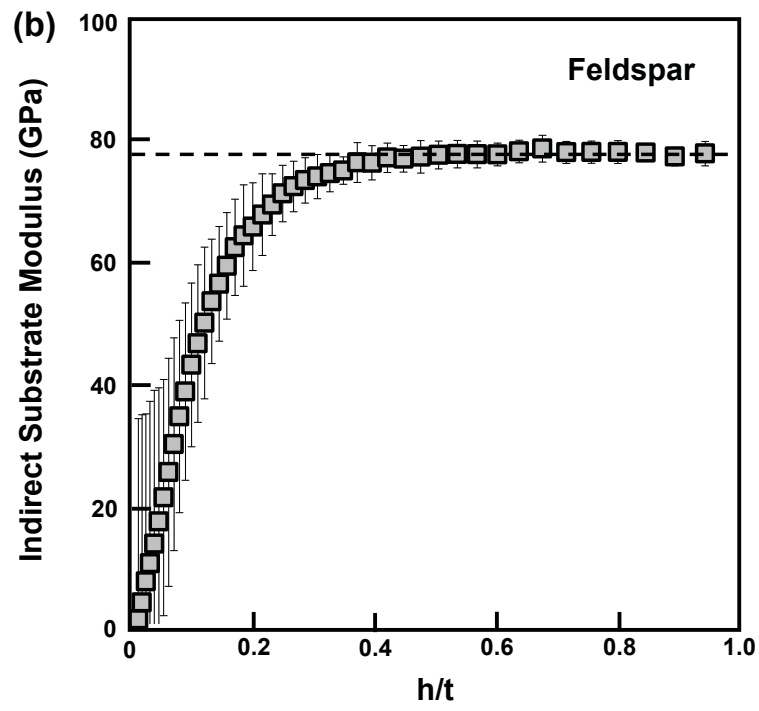
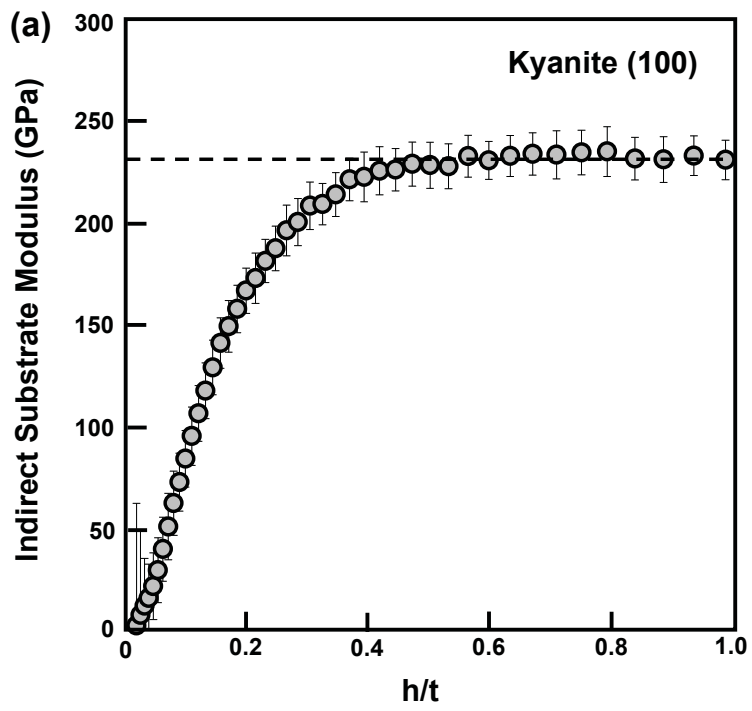


Figure 6

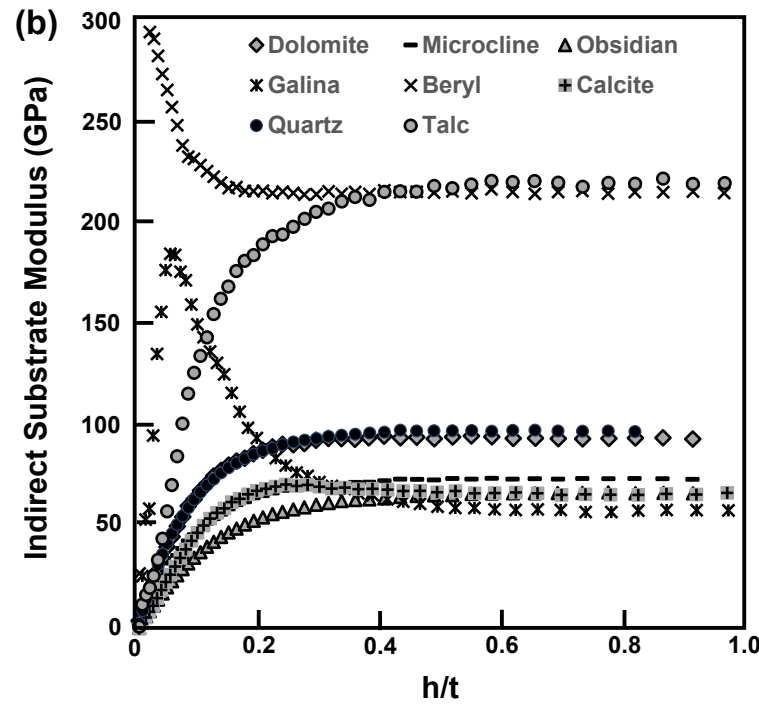
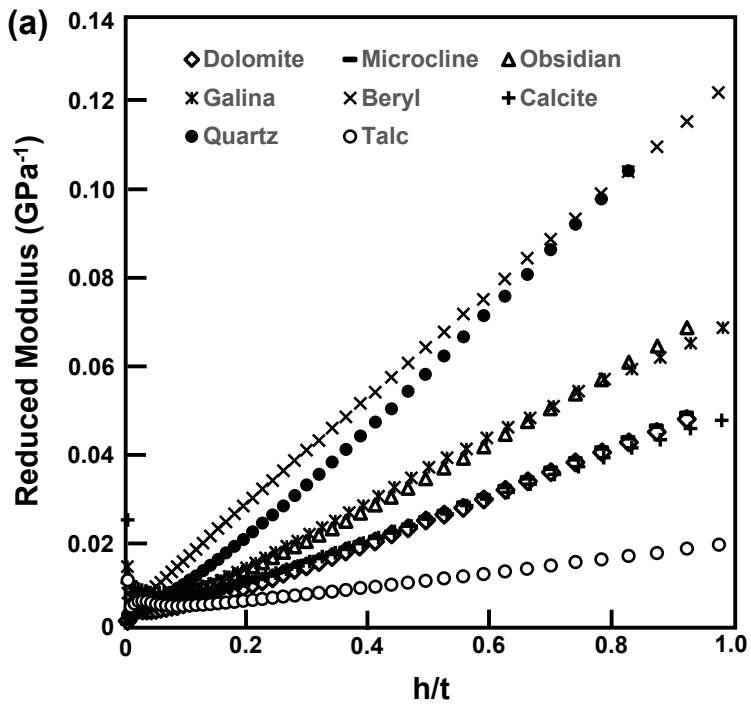


Figure 7