Influence of geochemical features on the mechanical properties of organic matter in shale

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Abstract

Organic matter is an important constituent in organic-rich shale, which influences the hydrocarbon generation, as well as the mechanical behavior, of shale reservoirs. The physical, chemical, and mechanical properties of organic matter depend on the source material and the thermal evolution process. Previous works attempted to investigate the impact of thermal maturation on the mechanical properties of organic matter. However, owing to the lack of maceral classification and the limitation of data volume during the mechanical measurement, no consistent trend has been identified. In this work, vitrinite reflectance test, scanning electron microscope observation, nanoindentation, and micro-Raman analysis were combined for geochemical and mechanical characterization. A total of 114 test areas were selected for testing, enhancing reliability of the test results. The Young's moduli of organic matter are from 3.57 GPa to 8.32 GPa. With the same thermal maturity, inertinite has the highest Young's modulus, while the modulus of bitumen is the lowest. The Young's moduli of different organic types all increase with vitrinite reflectance. When vitrinite reflectance increases from 0.62% to 1.13%, the modulus of inertinite and vitrinite is increased by 57% and 78%, respectively. In addition, with the increase of thermal maturity, the micro-Raman test results show a decrease of intensity ratio of D peak to G peak, indicating an increase of the ordered structure in organic matter. Organic type and thermal maturity reflect the diversity of the source material and chemical structure change during the thermal evolution process, and together they influence the mechanical properties of organic matter.

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Key Points:

- Young's modulus of inertinite and vitrinite is much higher than that of bitumen

- Modulus of organic matter increases with thermal maturity

- Intensity ratio of D peak to G peak of inertinite's Raman spectrum exhibits a decreasing trend with maturity

19   **Abstract**

20   Organic matter is an important constituent in organic-rich shale, which influences the

21   hydrocarbon generation, as well as the mechanical behavior, of shale reservoirs. The physical,

22   chemical, and mechanical properties of organic matter depend on the source material and the

23   thermal evolution process. Previous works attempted to investigate the impact of thermal

24   maturation on the mechanical properties of organic matter. However, owing to the lack of

25   maceral classification and the limitation of data volume during the mechanical measurement, no

26   consistent trend has been identified. In this work, vitrinite reflectance test, scanning electron

27   microscope observation, nanoindentation, and micro-Raman analysis were combined for

28   geochemical and mechanical characterization. A total of 114 test areas were selected for testing,

29   enhancing reliability of the test results. The Young's moduli of organic matter are from 3.57 GPa

30   to 8.32 GPa. With the same thermal maturity, inertinite has the highest Young's modulus, while

31   the modulus of bitumen is the lowest. The Young's moduli of different organic types all increase

32   with vitrinite reflectance. When vitrinite reflectance increases from 0.62% to 1.13%, the modulus

33   of inertinite and vitrinite is increased by 57% and 78%, respectively. The indentation creep of

34   different organic matter suggests that the stiffer organic type demonstrates less creep influence.

35   In addition, with the increase of thermal maturity, the micro-Raman test results show a decrease

36   of intensity ratio of D peak to G peak, indicating an increase of the ordered structure in organic

37   matter. From the characterization results, it was found that organic type and thermal maturity

38   reflect the diversity of the source material and the chemical structure change during the thermal

39   evolution process, and together they influence the mechanical properties of organic matter.

40

41   **Keywords:** organic type; thermal maturity; organic matter; Young's modulus; shale.

1. Introduction

Organic-rich shale is not only the source rock of conventional reservoirs, but also the reservoir rock of shale oil/gas reservoirs. Organic matter is an important constituent in organic-rich shale, which distinguishes organic-rich shale from other mud rocks with low organic content, and significantly influences the exploration and development of shale oil/gas. On the one hand, kerogen is the source material of hydrocarbon, and total organic carbon (TOC) content is an essential index for the evaluation of geological sweet spots (Cudjoe et al., 2016; Naizhen and Guoyong, 2016). On the other hand, the mechanical properties that significantly affect the fracturing effect of shale are quite different for organic matter and inorganic minerals in shale. Consequently, many indices for the evaluation of engineering sweet spots, for example, the brittleness index (Rybacki et al., 2016; Wang and Carr, 2012; Zhao et al., 2018), are also related to organic matter.

Organic matter in shale comes from deposits of the dead bodies of ancient organisms, and it can be classified on the basis of the diversity of source organisms and sedimentary environments. According to solubility, organic matter can be divided into kerogen and bitumen (Hunt, 1979). Regarding kerogen, two classification schemes exist: optical classification and chemical classification. The optical classification scheme borrows methods and concepts from coal petrology (Hutton et al., 1994; Stach et al., 1982). Through reflected light analysis, kerogen can be classified into maceral groups, such as inertinite, vitrinite, and exinite. Chemical classification focuses on the content of C, H, and O elements in kerogen. By major chemical elements analysis, kerogen can be classified as type I, type II, and type III (Tissot and Welte, 2013). Optical classification and chemical classification schemes are related to each other (Hunt, 1979). However, optical classification usually measures organic matter in certain micro regions, while

65   chemical classification refers to characterization of the whole rock. In addition to organic type,

66   thermal maturity constitutes another important geochemical property of organic matter. Thermal

67   maturity indicates the thermal evolution and hydrocarbon generation stage of the source rock.

68   Vitrinite reflectance is a key indicator of thermal maturity. Owing to the absence of higher

69   plants, rocks prior to the Devonian period do not contain vitrinite. Consequently, researchers

70   usually measure the reflectance of bitumen or graptolite as an alternative (Bertrand, 1990;

71   Bertrand and Heroux, 1987; Jacob, 1989). Organic type and thermal maturity together influence

72   the chemical structures of organic matter in shale.

73   Due to the lack of resolution, the elastic property of organic matter is difficult to characterize

74   using conventional mechanical test methods. However, micromechanical test techniques, such as

75   nanoindentation and modulus mapping based on nanoindenter and PeakForce quantitative

76   nanomechanical mapping (QNM) by atomic force microscope (AFM), offer convenience of

77   investigation at micro scale (Balooch et al., 2004; Dayal et al., 2009; Dokukin and Sokolov,

78   2012). Mechanical characterization of organic matter in shale can be divided into two types. In

79   the first type, organic matter was treated as a part of the main constituents in shale, and was

80   directly characterized through the application of micromechanical test techniques (Alstadt et al.,

81   2015; Bennett et al., 2015; Eliyahu et al., 2015; Kumar et al., 2012; Wilkinson et al., 2015;

82   Zargari et al., 2013; Zeszotarski et al., 2004). The reported modulus of organic matter was

83   generally between 0 GPa to 25 GPa, which was much lower than that of inorganic minerals.

84   These researches revealed strong micromechanical heterogeneity within different constituents in

85   organic-rich shales. In the second type, geochemical features of organic matter were taken into

86   consideration (Emmanuel et al., 2016; Li et al., 2018; Zargari et al., 2016). Kerogen and bitumen

87   with different thermal maturities were usually separated during the measurements. Previous

88   works in the second type suggested that the elastic modulus of organic matter is associated with

89   organic type and thermal maturity. Owing to the lack of classification of kerogen and the

90   limitation of data volume, however, no consistent trend has yet been discerned. Yang et al.

91   (2017) measured the mechanical properties of different macerals in shale using AFM-based

92   Nano IR. Inertinite, bitumen and tasmanite were separated in the work, but thermal maturity was

93   not considered for mechanical characterization. Similar to characterization of organic matter in

94   shale, the mechanical properties of macerals in coal and the influence of coal rank have also been

95   studied (Borodich et al., 2015; Epshtein et al., 2015; Vranjes et al., 2018; Zhang et al., 2019).

96   However, understanding of the exact influence requires further studies. In addition, molecular

97   simulation has recently been utilized to investigate the structures and mechanical properties of

98   organic matter (Bousige et al., 2016; Tong et al., 2016; Ungerer et al., 2014). The chemical

99   structure of organic matter is very complex, and to precisely construct a molecular model, and

100   predict the physical and mechanical properties, remain a challenge. Experimental works at micro

101   and nano scales can provide a useful reference and validation for simulations.

102   In this study, four shale samples with different thermal maturities from the Bonan Sub-sag

103   are prepared. A number of geochemical and micromechanical tests are combined to characterize

104   the properties of organic matter in each sample. Vitrinite reflectance is measured using a

105   photomicrometer. Inertinite, vitrinite, and bitumen are identified based on an optical

106   classification scheme. Nanoindentation is performed to characterize the mechanical properties of

107   organic matter in the selected test areas. Moreover, to elucidate the evolution of chemical

108   structures of organic matter, micro-Raman test is carried out. Micromechanical test results are

109   compared with the reported data. The influence of organic type and thermal maturity is discussed

110   on the basis of the characterization results.

111

2. Materials and methods

2.1. Sample information and preparation

Samples used in this study are shale cores of the third member of the Shahejie Formation from the Bonan Sub-sag. The Bonan Sub-sag is a third-order tectonic unit in the central part of the Zhanhua Depression in the Bohai Bay Basin with an area of 600 km^2. During the Paleogene period, the Bohai Bay Basin experienced initial, developmental, prosperous and atrophic periods of the fault depression, and successively developed the Kongdian Formation, the Shahejie Formation, and the Dongying Formation (Jiu et al., 2013; Wang et al., 2015). The Shahejie Formation can be divided into four sub-sections, and the third member of the Shahejie Formation from the Bonan Sub-sag contains a large number of shale oil formations.

Shale pieces with thicknesses from 2 mm to 5mm were cut from the shale cores. Then, mechanical polishing was performed to keep the upper and lower surface of the shale pieces parallel to each other, and to primarily decrease surface roughness. Finally, the upper surface was subjected to ion polishing to further decrease roughness. The roughness of a 20 μm × 20 μm area in the sample surface is below 100 nm.

2.2. Characterization methods

2.2.1. Vitrinite reflectance test

The vitrinite reflectance test is an effective method to characterize the thermal maturity of organic matter. The device for the vitrinite reflectance test, the photomicrometer, is a combination of an optical microscope and a spectrophotometer. The optical microscope with an oil immersion objective is utilized for observation and test area selection. The spectrophotometer is used to measure reflectance in a certain area. In addition to the vitrinite reflectance test, a

134 photomicrometer can also assist with optical classification of organic types according to image

135 contrast and reflectance data. The reflectance test and optical microscope observation, which

136 determine thermal maturity and organic type, constitute the foundation of the following tests.

137 2.2.2. Micro-Raman test

138 Raman spectrum is very sensitive to the structure and molecular arrangement of carbon

139 materials. Raman spectrum analysis is widely employed for characterization of carbon materials,

140 such as carbon nanotube and graphene (Dresselhaus et al., 2005; Ferrari et al., 2006; Pimenta et

141 al., 2007). Numerous researchers have also performed Raman spectrum tests on organic matter in

142 source rocks, and attempted to correlate the spectrum features with the results from conventional

143 vitrinite reflectance tests, to develop novel methods for the determination of thermal maturity

144 (Kelemen and Fang, 2001; Wilkins et al., 2014). Two ways of Raman spectrum analysis exist for

145 organic matter in shale. The first one needs to extract organic matter from shale rocks by

146 mechanical and chemical purification methods. However, this way necessitates destruction of the

147 shale rock during purification, which makes it unusable for mechanical tests. The second way is

148 via a micro-Raman test. Similar to the photometric test, micro-Raman combines optical

149 observation and Raman spectrum analysis (Lünsdorf, 2016; Tselev et al., 2014), which can

150 locate the micro areas and perform the measurements. Due to its advantages, the second way is

151 chosen for this study.

152 2.2.3. Nanoindentation

153 Mainstream commercial nanoindenters are developed on the basis of the method proposed by

154 Oliver and Pharr (Oliver and Pharr, 1992; Oliver and Pharr, 2004). During the nanoindentation

155 measurement, an indenter with a certain geometry is penetrated into the sample surface until the

156   targeted force or depth, and then it is pulled out. The load *P* on the sample and the displacement

157   *h* of the indenter tip are recorded in the loading, holding, and unloading process (Figure 1).
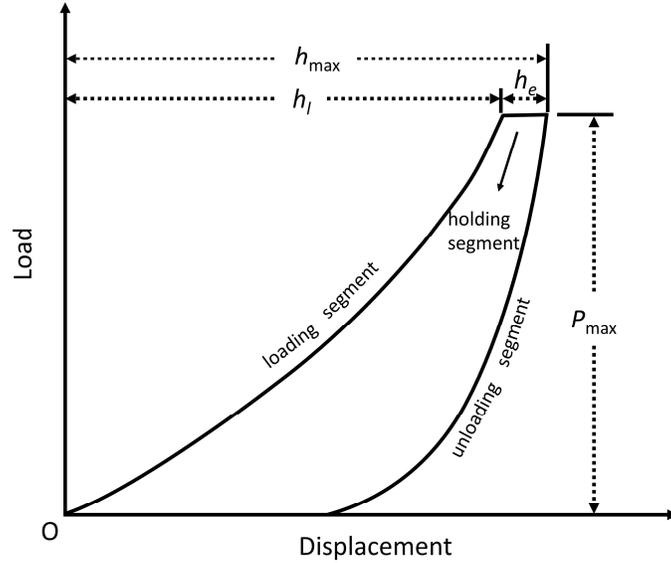

158

159   **Figure 1.** A schematic representation of load-displacement curve from nanoindentation. P_{max} is

160   the peak load; h_{max} is the maximum indenter displacement; h_l is the displacement at the end of

161   the loading segment; and h_e is the displacement increase during the holding process.

162   The unloading segment of the *P-h* curve reflects the elastic properties of the sample material.

163   According to the contact mechanics (Pharr et al., 1992), the basic model for nanoindentation is as

164   follows:

$$E_r = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A}} \tag{1}$$

165

166   where E_r is the reduced modulus; *S* is the slope of the initial unloading segment; and *A* is the

167   contact area of the indenter tip on the sample surface, which can be determined by the corrected

168   area function (Oliver and Pharr, 1992):

$$A = 24.5h_c^2 + \sum_{i=0}^{7} C_i h_c^{1/2^i} \tag{2}$$

169

170   where C_i are fitting parameters; and h_c is the contact depth of the indenter, which is related to the

171   maximum displacement h_{max} (Oliver and Pharr, 1992):

$$h_c = h_{max} - \beta \frac{P_{max}}{S} \tag{3}$$

173   where β is the parameter corresponding to the geometry of the indenter; and P_{max} is the peak

174   load. The reduced modulus is determined by both the sample and the indenter material. The

175   following equation can be used to eliminate the influence from the indenter and calculate the

176   Young's modulus E of the sample (Johnson, 1987):

$$\frac{1-\gamma^2}{E} = \frac{1}{E_r} - \frac{1-\gamma_{tip}^2}{E_{tip}} \tag{4}$$

178   where γ is the Poisson's ratio of the sample; and E_{tip} and γ_{tip} are the Young's modulus and

179   Poisson's ratio of the indenter tip, respectively.

180   2.2.4. Experimental procedures

181   Geochemical test and mechanical characterization are combined in this study (Figure 2). Ion-

182   polished samples were placed into a photomicrometer (QDI 302, CRAIC; DM4500P, Leica).

183   Test areas containing organic matters were observed and selected under an oil immersion

184   objective (Figure 2a). The spectrophotometer was used to measure the reflectance of organic

185   matter. The illumination spot size for reflectance measurement was approximately 3 μm. Organic

186   type was identified through optical image and reflectance data. Vitrinite reflectance constitutes

187   the measurement of the thermal maturity of each sample. SEM observation (Figure 2b) was then

188   carried out to observe the selected test areas at a low vacuum condition with an accelerate

189   voltage of 15 kV (Quanta 200F, FEI). For convenience of positioning in the following

190   procedures, the precise coordinate of each test area was recorded, and SEM images of organic

191   matters were captured at various magnifications. Subsequently, the elastic properties of organic

192   matter in each sample were measured using nanoindentation (Tribo Indenter 950, Hysitron).

193   Scanning probe microscopy (SPM) was performed prior to (Figure 2c) and after (Figure 2e)

194   nanoindentation, to determine the indentation position and to observe the residual indentation,

195   respectively. The indentation was usually placed at the center position of organic matter to avoid

196   the interference from surrounding minerals. A Berkovich diamond indenter with a nominal tip

197   radius of 100 nm was chosen for the test. Under load controlling mode, the maximum load was

198   set to be 1000 μN (Figure 2d), which was uniform for each indentation. The loading, holding,

199   and unloading time were set to be 10 s, 2 s and 10 s, respectively. The contact area of the

200   indentation was on the order of 10^6 nm^2. Finally, the micro-Raman test (Figure 2f) was

201   performed to elucidate the structural variation within the organic matter with different thermal

202   maturities (DXRxi, ThermoFisher). The immersion source was a 532 nm laser, and the exposure

203   time was 0.05 s. The spot size of the laser beam was about 1 μm. To decrease damage on the

204   sample surface, the laser power was set to be 0.5 mW.

205
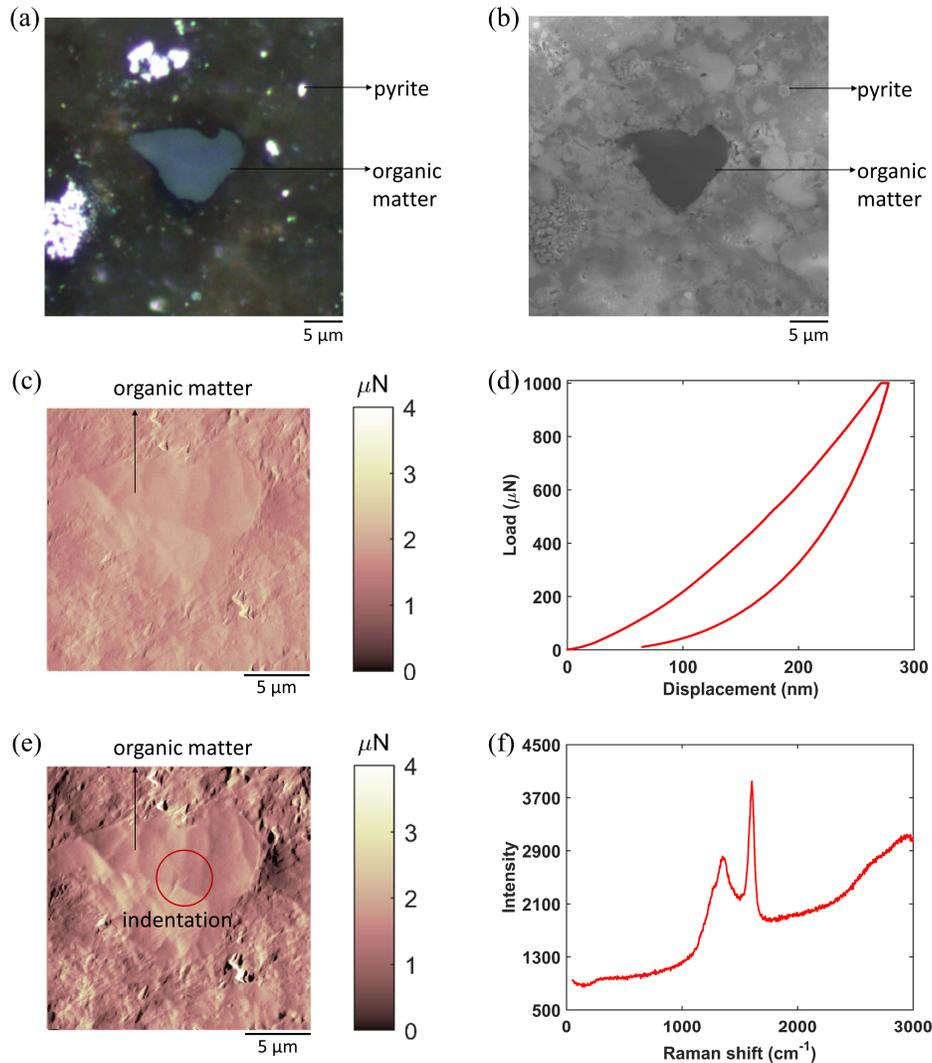
Figure 2. Experimental procedures: (a) Vitrinite reflectance test, macerals classification, and test area selection; (b) SEM observation and test area location; (c) SPM (scanning frequency = 0.2 Hz) observation prior to nanoindentation; (d) nanoindentation; (e) SPM (scanning frequency = 0.5 Hz) observation after nanoindentation; and (f) micro-Raman analysis.

3. Results and discussion

3.1. Organic type and thermal maturity

Table 1 shows the organic type classification and thermal maturity test results. The vitrinite reflectance of shale samples is from 0.62% to 1.13%, which means that all of the samples are in

214   the mature stage. According to the image features under the oil immersion objective, three

215   organic types, including vitrinite, inertinite and bitumen, are identified in the shale samples.

216   **Table 1.** Vitrinite reflectance and number of areas tested in each sample.

Sample	R_o (%)	Number of areas tested		
		Vitrinite	Inertinite	Bitumen
J1	0.62	16	11	——
J2	0.69	12	9	15
J3	0.94	10	11	6
J4	1.13	9	12	3

217

218   Figure 3 compares the optical images and SEM images of different organic types. Compared

219   with the dry objective, the oil immersion objective can greatly increase the image contrast and

220   the clarity of organic matter. Through the first column of Figure 3, one can see that the image

221   features of the typical test areas are markedly different. Specifically, the organic type in the first

222   test area is inertinite, which is cinerous and seems to protrude from the sample surface (Figure

223   3a). In contrast, vitrinite in the second test area (Figure 3c) and bitumen in the last test area

224   (Figure 3e) are dark gray and brown, respectively. However, it is challenging to differentiate the

225   organic types based only on the image contrast of SEM images (Figure 3b, d, f). In addition to

226   optical images, the reflectance test results can also assist with the classification of organic type.

227   The reflectance of vitrinite is usually higher than the reflectance of bitumen, while lower than

228   that of inertinite. In sample J2, for example, bitumen reflectance is from 0.39% to 0.52%,

229   vitrinite reflectance is from 0.46% to 1.1%, and inertinite reflectance is from 1.1% to 2.2%. A

230   total of 114 test areas that contain different types of organic matter are selected for

231   nanoindentation test (Table 1).

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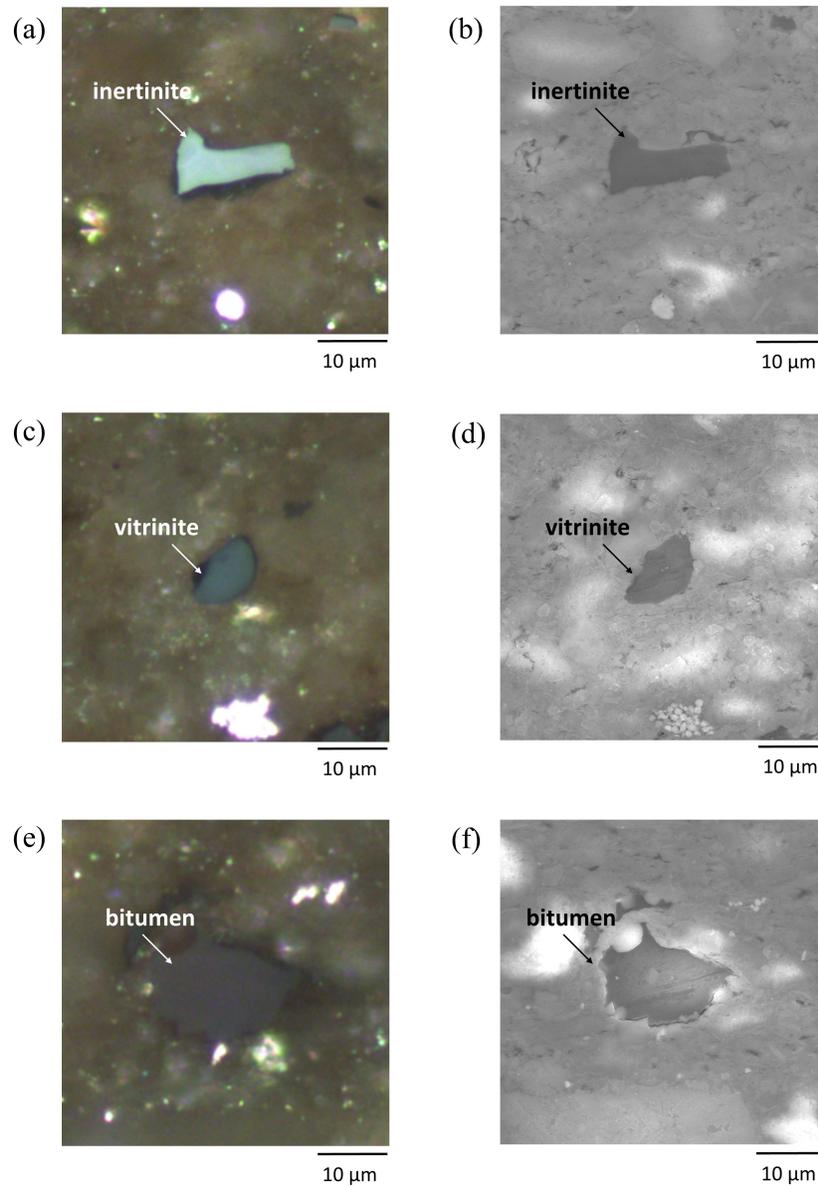
Figure 3. Different types of organic matter in sample J3: (a) Optical image and (b) SEM image

of inertinite; (c) optical image and (d) SEM image of vitrinite; and (e) optical image and (f) SEM

image of bitumen.

3.2. Nanoindentation results

The number of areas tested of each kind of organic matter in each sample is listed in Table 1.

Figure 4 assembles the *P-h* curves for different kinds of organic matter in each sample. With the

239   same maximum load setting, the maximum displacement of vitrinite is generally lower than that

240   of inertinite, while higher than that of bitumen. The sudden increase of displacement during the

241   loading process in Figure 4c, which is marked with a black circle, is termed *pop-in*. It suggests

242   the rapture of organic matter. The comparison of the *P-h* curves indicates that bitumen is much

243   softer than vitrinite and inertinite with the same thermal maturity.

244
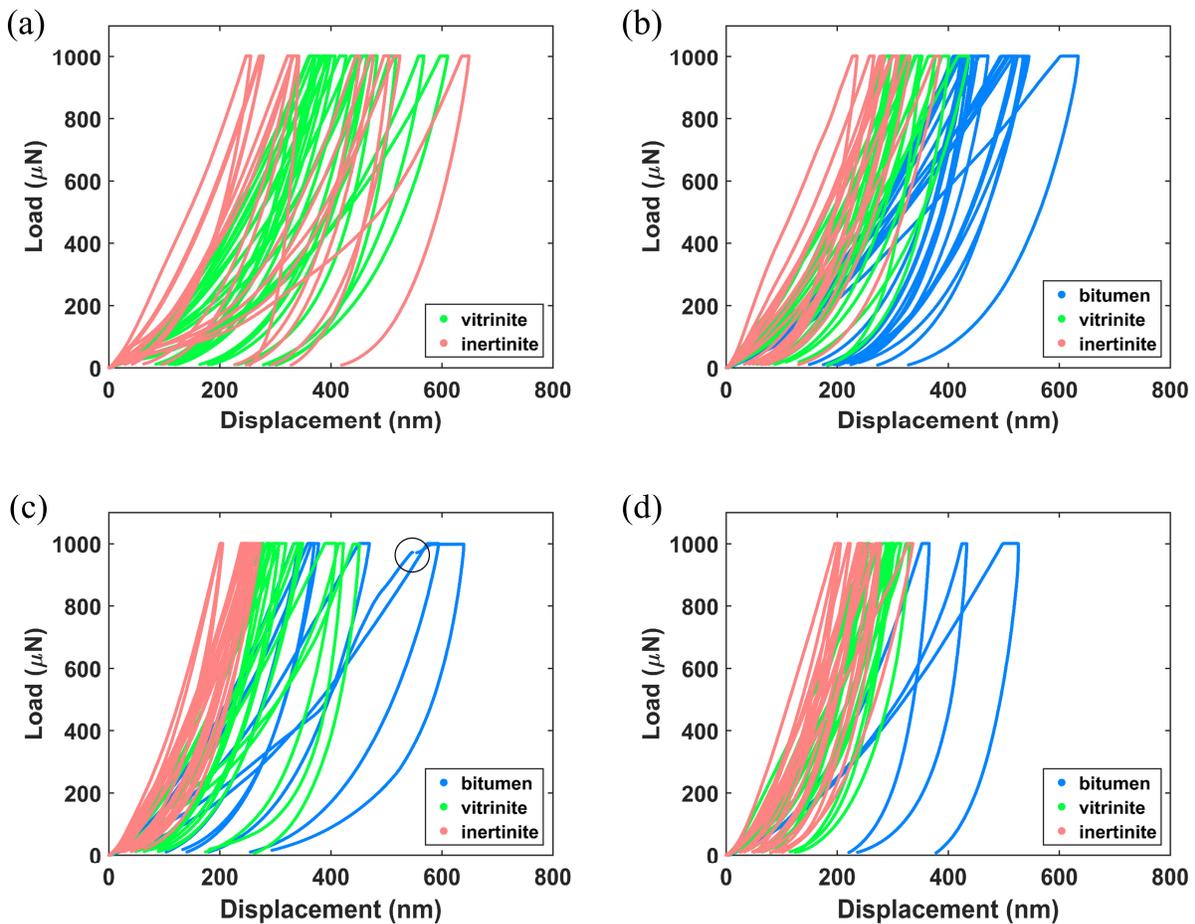
Figure 4. Assembly of representative *P-h* curves from sample J1 (a), J2 (b), J3 (c), and J4 (d).

246   The Young's modulus can be calculated using equation (1) to equation (4). The Young's

247   modulus and Poisson's ratio of the diamond indenter tip are 1141 GPa and 0.07, respectively.

248   However, the Poisson's ratio of organic matter remains unknown, and the estimation of

249   Poisson's ratio influences the calculation results. A wide range of Poisson's ratio of organic

250   matter, from 0.05 to 0.45, was used in previous literature (Ahmadov et al., 2009; Eliyahu et al.,

251   2015; Emmanuel et al., 2016; Li et al., 2018). Figure 5 illustrates the sensitivity to Poisson's

252   ratio based on the load-displacement data in this work. The calculated Young's modulus of

253   organic matter gradually increases with the selected Poisson's ratio value. In the following

254   discussions, the Poisson's ratio of organic matter is assumed to be 0.3. The maximum

255   uncertainty introduced by the assumption is approximately 12.4%.
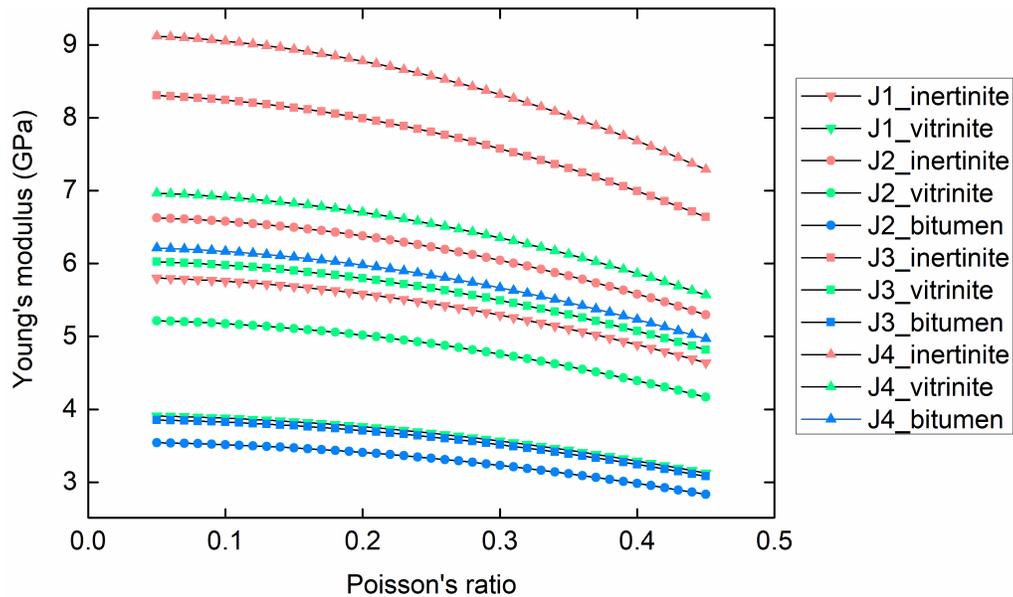
256

257   **Figure 5.** The sensitivity of calculated Young's modulus to Poisson's ratio of organic matter.

258   Table 2 summarizes Young's modulus calculation results. It is obvious that inertinite has

259   the highest Young's modulus, while the modulus of bitumen is the lowest. Moreover, the

260   Young's moduli of different organic types all increase with vitrinite reflectance. When vitrinite

261   reflectance increases from 0.62% to 1.13%, the Young's modulus of inertinite increases from

262   5.29 GPa to 8.32 GPa, by 57%, and the Young's modulus of vitrinite increases from 3.57 GPa to

263   6.35 GPa, by 78%. From Table 2, it can be concluded that both organic type and thermal

264   maturity affect the mechanical properties of organic matter. Repeated testing on a number of test

265   areas (Table 1) for different organic types in each sample provides a valuable dataset to examine

266   the variability of the mechanical properties and the reliability of the test results. Except for a

267   couple of test results (i.e., inertinite for sample J1 and bitumen for J3), the standard deviation for

268   each type of testing is moderate (Table 2), indicating either a reliable testing result or moderate

269   variability, or both.

270   **Table 2.** Young's modulus of different types of organic matter.

Sample	R_o (%)	Young's modulus (GPa) [a]		
		Vitrinite	Inertinite	Bitumen
J1	0.62	3.57±0.83	5.29±2.85	——
J2	0.69	4.76±0.85	6.05±1.63	3.23±0.80
J3	0.94	5.50±0.80	7.58±1.38	3.52±1.25
J4	1.13	6.35±0.97	8.32±1.53	5.67±0.84

271   [a]± indicates the standard deviation.

272   An increase in displacement in the holding segment (Figure 4) indicates the viscoelasticity

273   of organic matter, which was also investigated in previous works (Epshtein et al., 2015; Liu et al.,

274   2019; Zeszotarski et al., 2004). To discuss the problem, indentation creep C_{IT} is calculated by

275   using the following equation (ISO14577-1, 2002):

276
$$C_{IT} = \frac{h_e}{h_l} \times 100 \qquad (5)$$

277   where h_e and h_l are the displacement increase during the holding and loading process,

278   respectively. The C_{IT} values of different types are summarized in Table 3. With the same

279   maximum load and holding time setting, the indentation creep of bitumen is much higher than

280   that of vitrinite and inertinite. The calculation results suggest that the stiffer organic type

281   demonstrates less creep influence. However, no clear relation is identified between indentation

282   creep and thermal maturity. While this work focuses on the elastic properties of organic matter, a

283   longer holding time and more suitable test method, for example, dynamic mechanical analysis,

284 would be required for rigorous study of the creep behavior (Chagnon et al., 2013; Koch et al.,

285 2007).

286 <div align="center">Table 3. Indentation creep calculation results.</div>

Sample	R_o (%)	Indentation creep (%) [a]		
		Vitrinite	Inertinite	Bitumen
J1	0.62	2.40±0.31	2.21±0.62	——
J2	0.69	3.09±0.73	2.65±0.81	4.61±1.11
J3	0.94	2.97±1.00	2.18±0.51	4.66±2.87
J4	1.13	2.38±0.75	2.18±0.59	3.77±1.76

287 [a]± indicates the standard deviation.

288 3.3. Micro-Raman analysis

289 Because of the influence from fluorescence, the Raman spectrum of bitumen and vitrinite has a

290 poor signal-to-noise ratio, and we can only obtain reliable data about inertinite. Through

291 processing of the original spectrum, the micro-Raman test results are analyzed. Figure 6

292 illustrates a typical Raman spectrum of inertinite. There is a strong fluorescence background in

293 the original Raman data (Figure 6a). Similar to the carbon materials, two characteristic peaks in

294 the spectrum are observed. To remove the background, the baseline is calculated by using the

295 data points around the characteristic peaks. In Figure 6b, the peak around 1350 cm^{-1} is the defect

296 peak (D peak), which indicates the defects and the disordered structure in the material. The peak

297 around 1600 cm^{-1} is the graphene peak (G peak), which originates from the plane structure of

298 carbon materials. The intensity ratio of D peak to G peak (IR (D/G)) can be the indicator of the

299 degree of graphitization. A higher IR (D/G) indicates fewer defects, a smaller disordered

300 structure, and a higher degree of graphitization.

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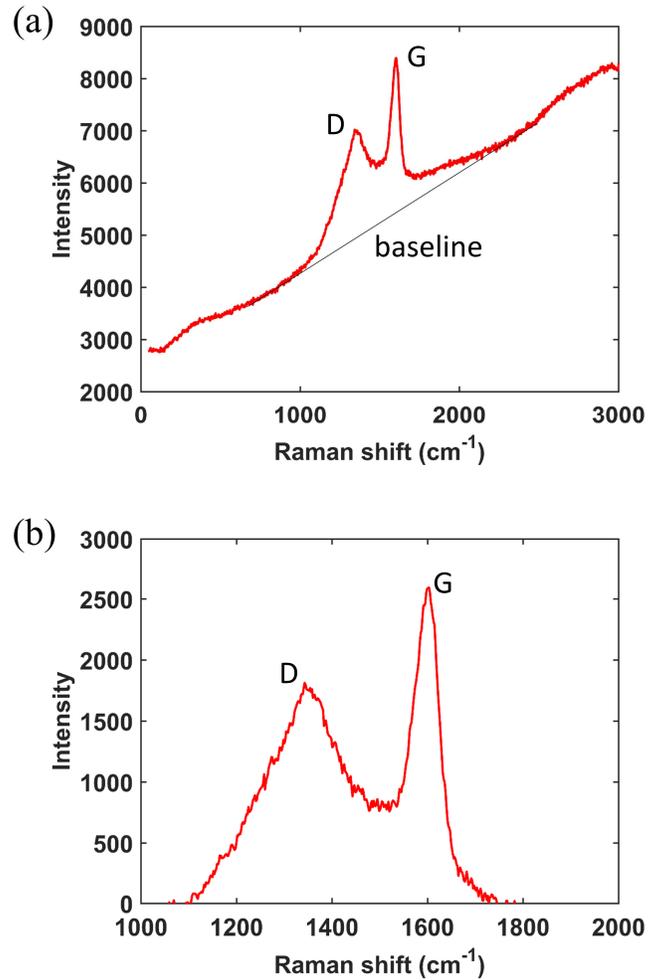
Figure 6. Data processing of Raman spectrum: (a) Original Raman spectrum of inertinite with

strong fluorescence background; and (b) processed spectrum in the region of interest. D and G

indicate characteristic peaks in the Raman spectrum of carbon material. The baseline is

calculated by using the data points around the characteristic peaks. The intensity ratio of D peak

to G peak is calculated through the division of maximum intensity value of the D band to that of

the G band.

308

Figure 7 compares two representative Raman spectrums from sample J1 and J4,

respectively. The IR (D/G) of the inertinite in sample J1 is higher than that of the inertinite in

311   sample J4, which suggests that the organic matter in sample J4 has a higher degree of

312   graphitization. The IR (D/G) of each sample is averaged (Table 4) in consideration of the

313   heterogeneity within different inertinites in a certain shale sample. When vitrinite reflectance

314   increases, the IR (D/G) exhibits a general decreasing trend. The chemical structure change

315   results from thermal evolution. With the increase of thermal maturity, the aliphatic structure is

316   gradually decomposed, and the aromaticity of kerogen increases (Craddock et al., 2018; Duan et

317   al., 2018; Tissot and Welte, 2013). Therefore, the calculation results reveal that the organic

318   matter with a higher thermal maturity may contain fewer defects and a more ordered structure.
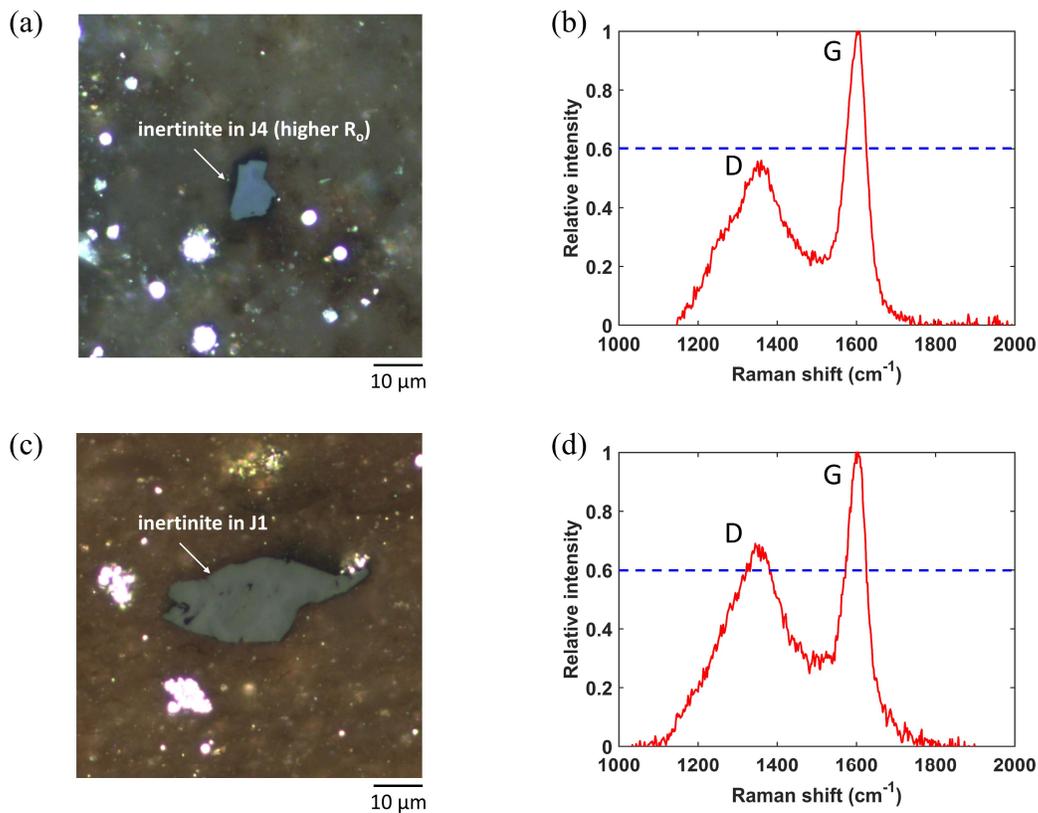
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320   **Figure 7.** Representative inertinite area and corresponding Raman spectrum from sample J4 (a,

321   b) and J1 (c, d).

322

323

324 **Table 4.** Intensity ratio of D peak to G peak of different samples.

Sample	R_o (%)	IR (D/G) [a]	Number of areas tested
J1	0.62	0.648±0.024	11
J2	0.69	0.667±0.022	9
J3	0.94	0.620±0.038	11
J4	1.13	0.600±0.034	12

325 [a]± indicates the standard deviation.

326 3.4. Comparison with extant literature

327 Table 5 presents a comparison of the test results in this study and the reported data. In previous

328 works, utilization of dynamic test techniques is preferred for mechanical characterization,

329 including modulus mapping by nanoindentation system and QNM by AFM. Kerogen and

330 bitumen are distinguished in previous research, but the macerals are not classified. Although

331 Zargari et al. (2016) and Li et al. (2018) both investigate organic matter in Bakken shale, the test

332 results show different trends. Zargari et al. (2016) find the modulus reduction of kerogen from

333 immature to mature stage, while Li et al. (2018) observe that organic matter becomes stiffer as

334 thermal maturity increases. The different findings may be owing to the lack of classification of

335 organic type and the limited data volume. The test results provided by Zargari et al. (2016) are

336 storage moduli, which are measured by using modulus mapping with a diamond indenter.

337 Storage modulus is comparable to reduced modulus, and is also influenced by both the sample

338 and the indenter tip (Zhao et al., 2018). The relation between storage modulus and Young's

339 modulus is in the same form as equation (4) (Syed Asif et al., 2001), and the Young's modulus

340 of diamond is much higher than that of organic matter. Thus, storage modulus and converted

341 Young's modulus should have the same trend from immature to mature stage. The conclusion by

342 Emmanuel et al. (2016) is that the modulus of kerogen significantly increases when vitrinite

343   reflectance increases from 0.40% to 0.82%, while the modulus hardly changes when vitrinite

344   reflectance increases from 0.82% to 1.25%. However, in this paper, we find that the elastic

345   modulus of vitrinite, inertinite, and bitumen all increase with thermal maturity.

346   Different from the previous investigations listed in Table 5, this study considers the

347   influence of not only thermal maturity, but also organic type. Vitrinite and inertinite are

348   identified based on optical microscope images and reflectance data. The difference of elastic

349   properties between vitrinite and inertinite are found by nanoindentation measurements.

350   Consequently, without consideration of maceral classification, conclusions about the influence

351   from thermal maturity may be unreliable, especially when the data volume is limited. In addition,

352   most of the previous investigations divide the shale samples into two parts. One is for

353   geochemical characterization, while the other one is for the mechanical test. Sample-to-sample

354   variation is also not adequately considered. In this work, a set of geochemical-mechanical

355   experiments is performed on the same selected test areas in a certain sample. Sample-to-sample

356   variation no longer presents a problem, and the experimental data of repeated tests on the same

357   type of test areas provide meaningful statistics. Finally, micromechanical tests on shale are very

358   time consuming, due to the complexity of sample preparation and locating the test area. The

359   number of areas tested in previous works is also relatively small. Here, the data volume is

360   significantly increased, which makes the test results in this work more reliable.

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Table 5. Comparison with the test results in previous works.

| Reference | R$_o$ (%) | Modulus (GPa) | | | Test technique | Number of areas tested |
| | | Kerogen | | Bitumen | | |
		Vitrinite	Inertinite			
Zargari et al. (2016)[a]	Immature	15~20		——	Modulus mapping	12
	Mature	7~10		——		
	Mature	7~10		——		
	Overmature	7~12		——		
Emmanuel et al. (2016)[b]	0.40	6.10		——	AFM	26
	0.82	16.0		7.50		
	1.25	15.80		8.50		
Li et al. (2018)[c]	0.35	2.91		——	AFM	3
	0.64	——		3.33		
	1.04	——		11.77		
This work[d]	0.62	3.57	5.29	——	Nano-indentation	114
	0.69	4.76	6.05	3.23		
	0.94	5.50	7.58	3.52		
	1.13	6.35	8.32	5.67		

368   [a]Modulus in this literature is storage modulus;
369   [b]Modulus in this literature is Young's modulus;
370   [c]Modulus in this literature is Young's modulus;
371   [d]Modulus in this work is Young's modulus.

372

373 **4. Conclusions**

374 In the present paper, the influence of geochemical features on the mechanical properties of

375 organic matter is investigated by combining various geochemical and micromechanical

376 characterization methods. The relations between the mechanical properties of different organic

377 types and thermal maturities are summarized in Figure 8. The main findings are as follows:

378   (1) Inertinite, vitrinite, and bitumen show different image features under the oil immersion

379   objective and have a different reflectance range. It is difficult to distinguish organic type only

380   according to the image contrast of SEM images.

381   (2) The vitrinite reflectance of the four shale samples is from 0.62% to 1.13%. With the same

382   thermal maturity, mechanical heterogeneity exists within different types of organic matter.

383   Inertinite has the highest Young's modulus, while the modulus of bitumen is the lowest. Taking

384   sample J3 as an example, the Young's modulus of inertinite is more than two times that of the

385   modulus of bitumen.

386   (3) The Young's moduli of all types of organic matter increase with thermal maturity. When

387   vitrinite reflectance increases from 0.62% to 1.13%, the Young's modulus of inertinite increases

388   from 5.29 GPa to 8.32 GPa, by 57%, and the Young's modulus of vitrinite increases from 3.57

389   GPa to 6.35 GPa, by 78%. The indentation creep calculation results suggest that the stiffer

390   organic type indicates less creep influence.

391   (4) Characteristic peaks of carbon material can be found in the Raman spectrum of inertinite. The

392   intensity ratio of D peak to G peak exhibits a decreasing trend with thermal maturity, indicating

393   the increase of graphitization degree of organic matter.

394   (5) The geochemical and micromechanical characterization results reveal that the mechanical

395   properties of organic matter are influenced by both organic type and thermal maturity. Compared

396   with bitumen, vitrinite and inertinite have more polycyclic aromatic hydrocarbons, fewer

397   saturated aliphatic hydrocarbons, and higher carbon content. Therefore, with the same thermal

398   maturity, the Young's modulus of vitrinite and inertinite is higher than that of bitumen. With the

399   increase of thermal maturity, the aliphatic structure is gradually decomposed and consumed, and

400 the aromaticity of kerogen increases. Consequently, IR (D/G) decreases, and the Young's

401 modulus increases.

402 (6) The mechanical behavior of organic matter plays an important role in the predictions of

403 macroscopic mechanical properties at the core scale and even the well scale, especially for

404 organic-rich shales. This can be done based on upscaling methods, such as the Mori-Tanaka

405 model and the self-consistence method, with the mechanical characterization results of the main

406 constituents in shale (Abedi et al., 2016; Goodarzi et al., 2017; Zhao et al., 2018). The

407 characterization in this work can serve as a supplement to micro constituent input databases for

408 more reliable overall calculation results. At micro scale, the experimental research can also

409 provide validation for related molecular simulations (Bousige et al., 2016; Wu and Firoozabadi,

410 2020). In addition, mechanical variation within organic matters has the potential to inversely

411 assist with the identification of organic type and the determination of thermal maturity.
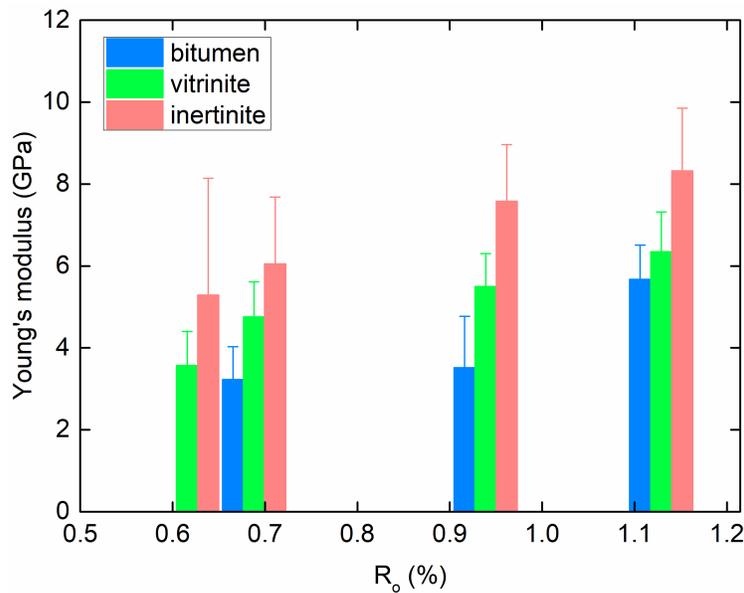
412

413 **Figure 8.** Young's modulus of organic matter with different vitrinite reflectance.

414 Considering sample-to-sample variation, this work proposes a workflow focusing on fixed

415 micro areas for different characterization. The data volume is also significantly increased

416 compared with that in the extant literature. However, the range of vitrinite reflectance in this

417 study is limited, and all of the four samples are in the mature stage. In the future, shale samples

418 in other thermal maturation stages need to be investigated.

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575

Figure 1.

Figure 2.

(a) pyrite
organic matter
5 µm

(b) pyrite
organic matter
5 µm

(c) organic matter
µN
5 µm

(d)

(e) organic matter
indentation
µN
5 µm

(f)

Figure 3.

(a) inertinite

(b) inertinite

(c) vitrinite

(d) vitrinite

(e) bitumen

(f) bitumen
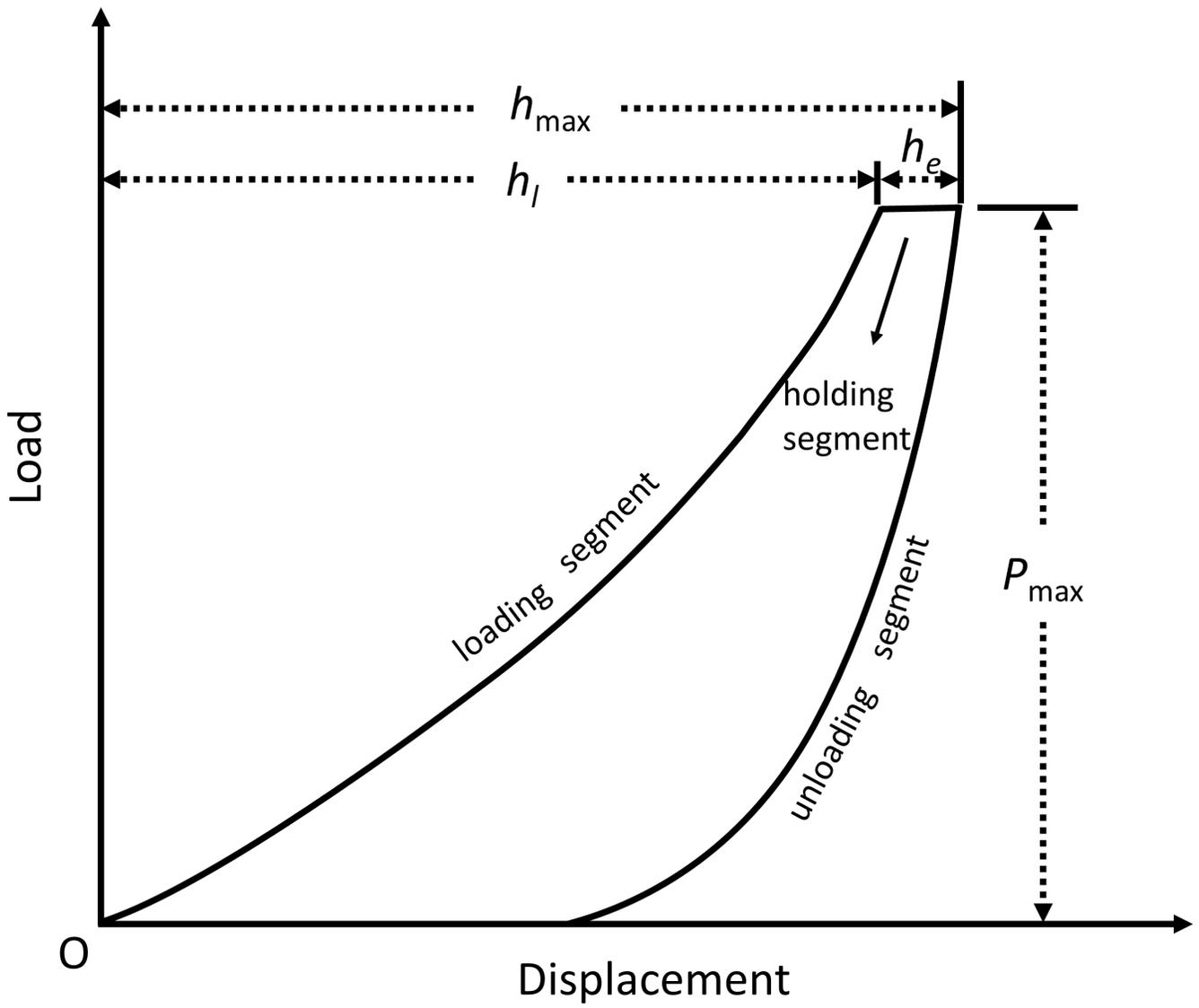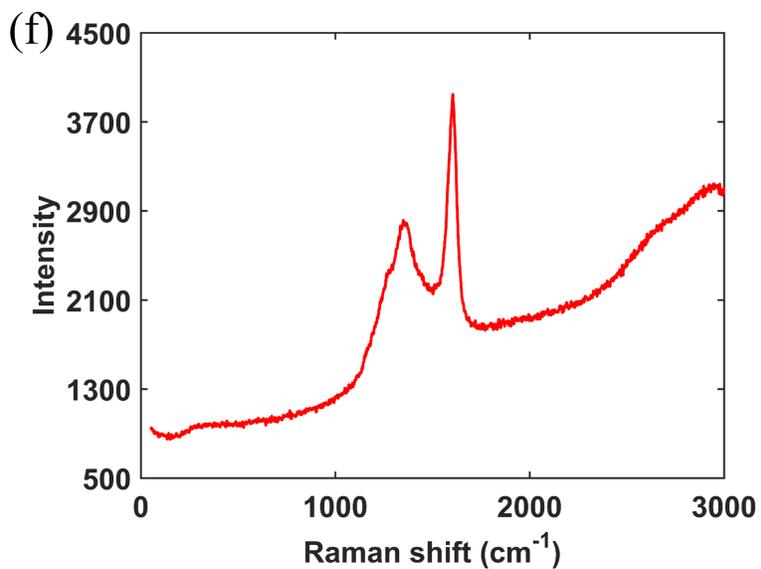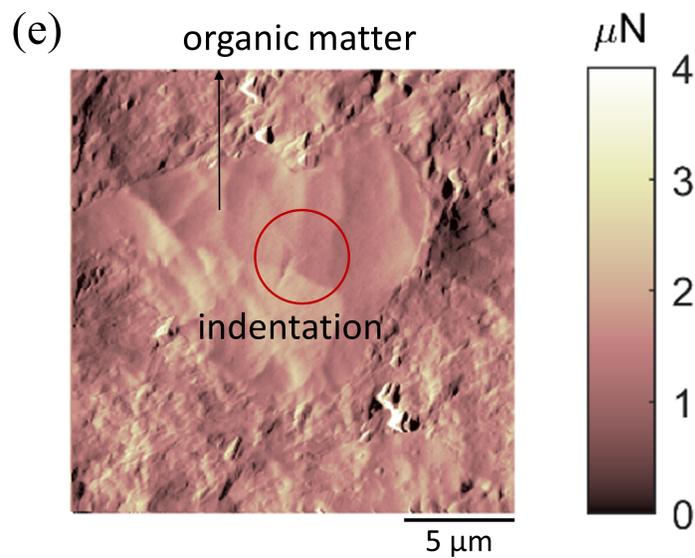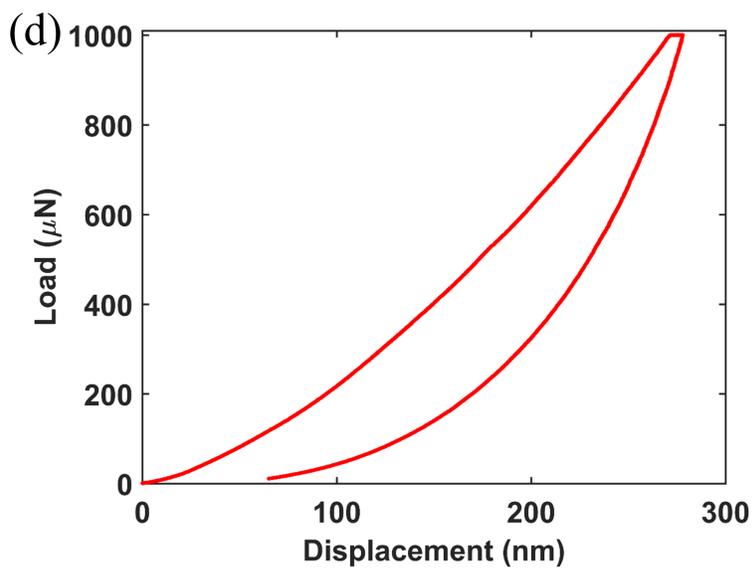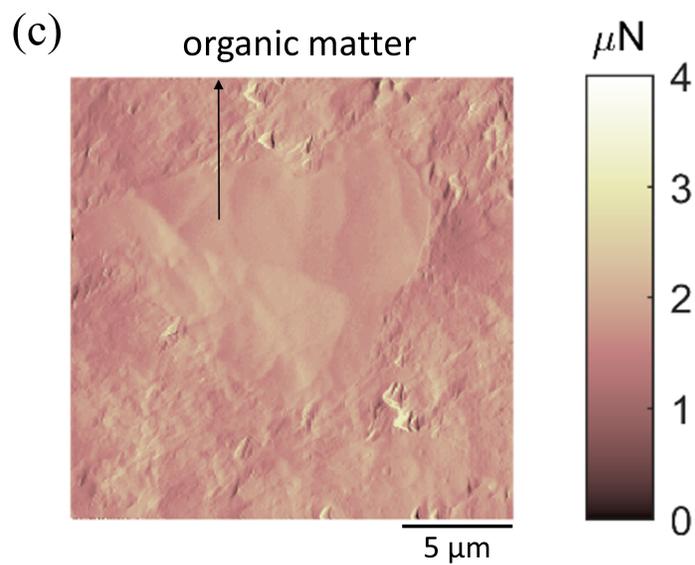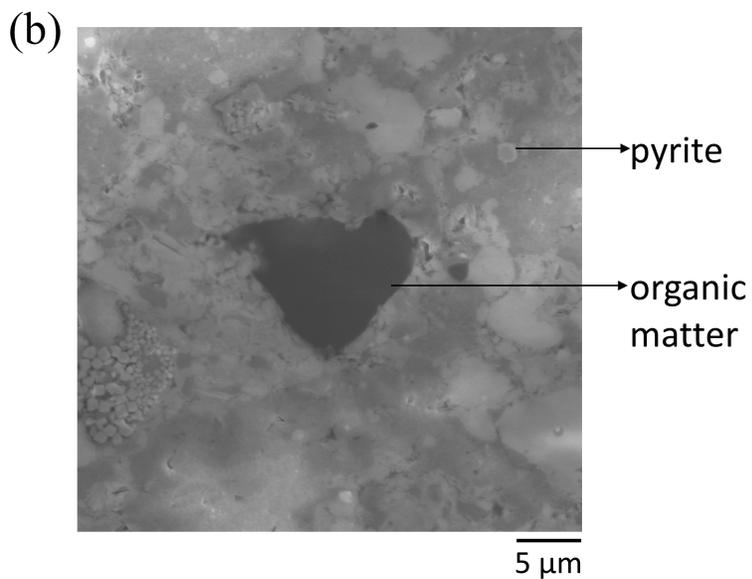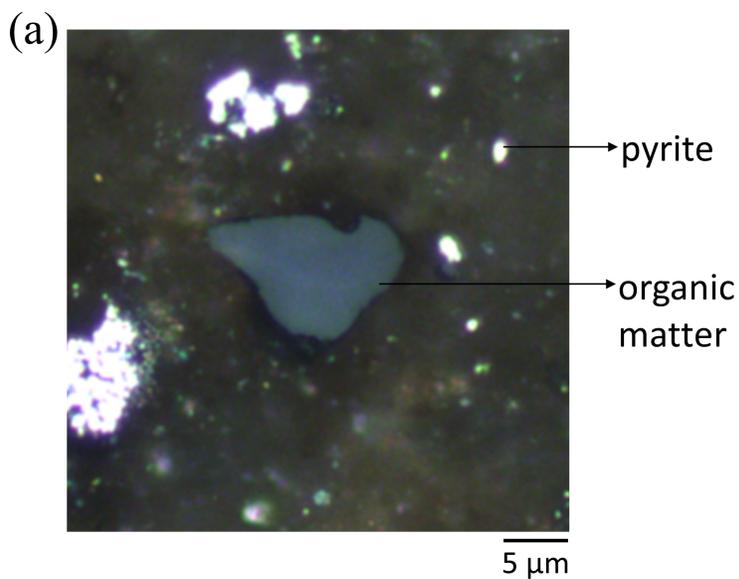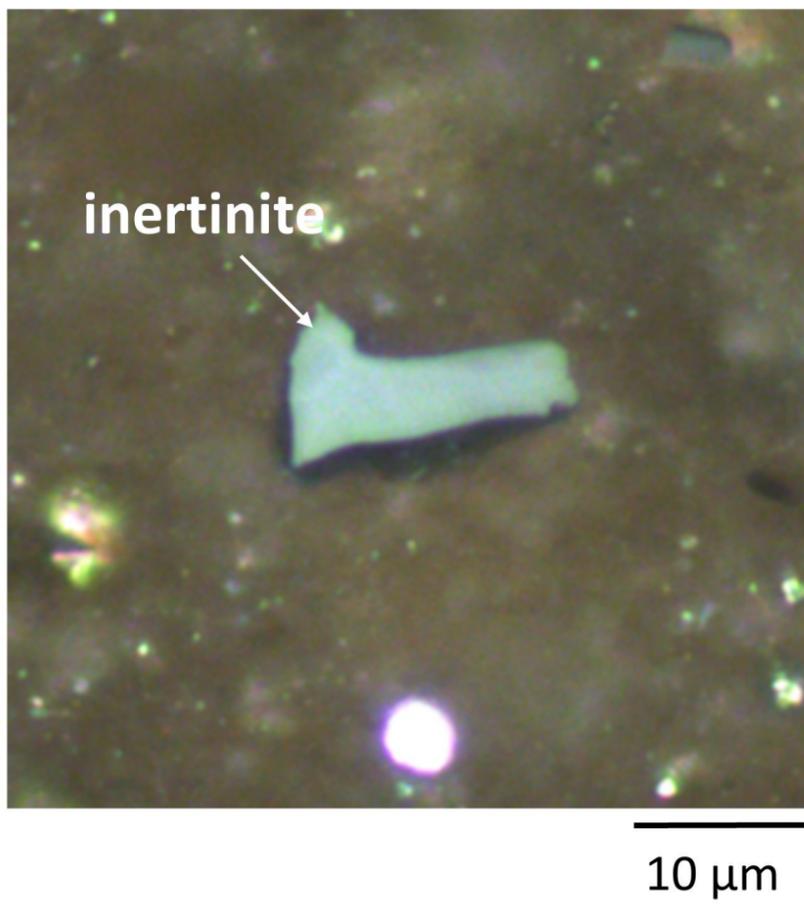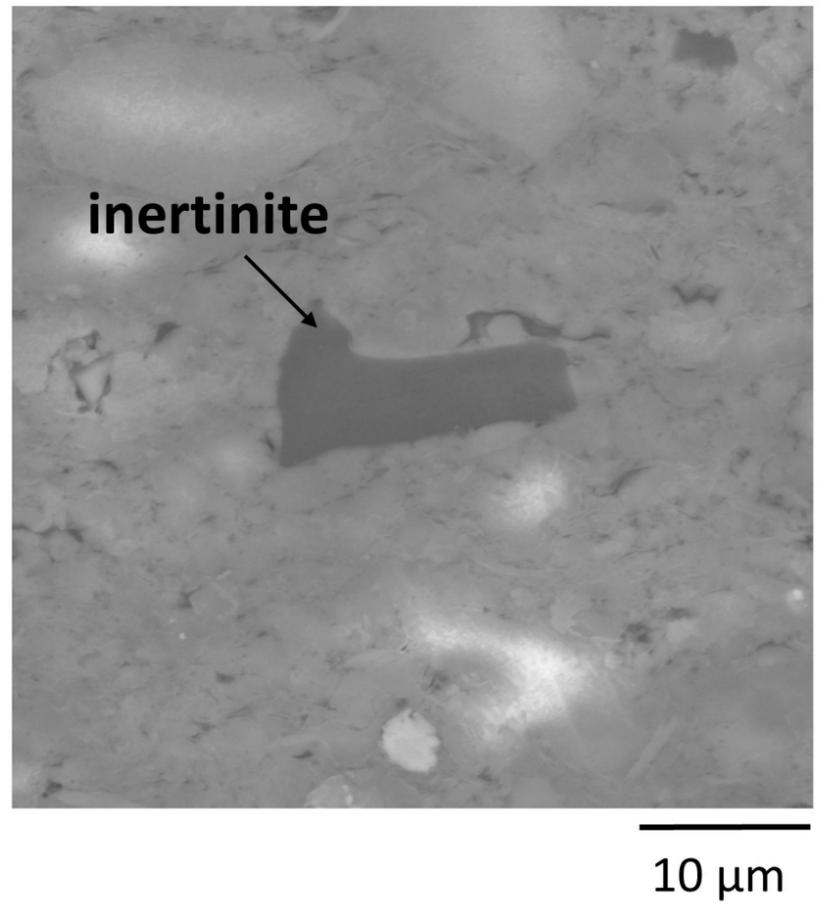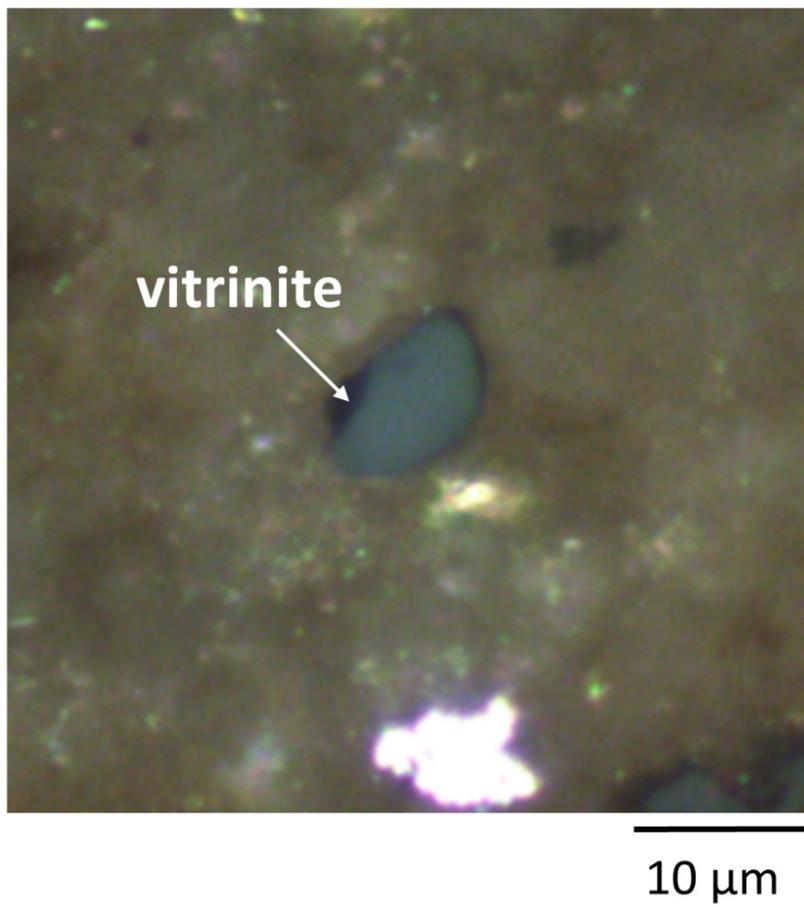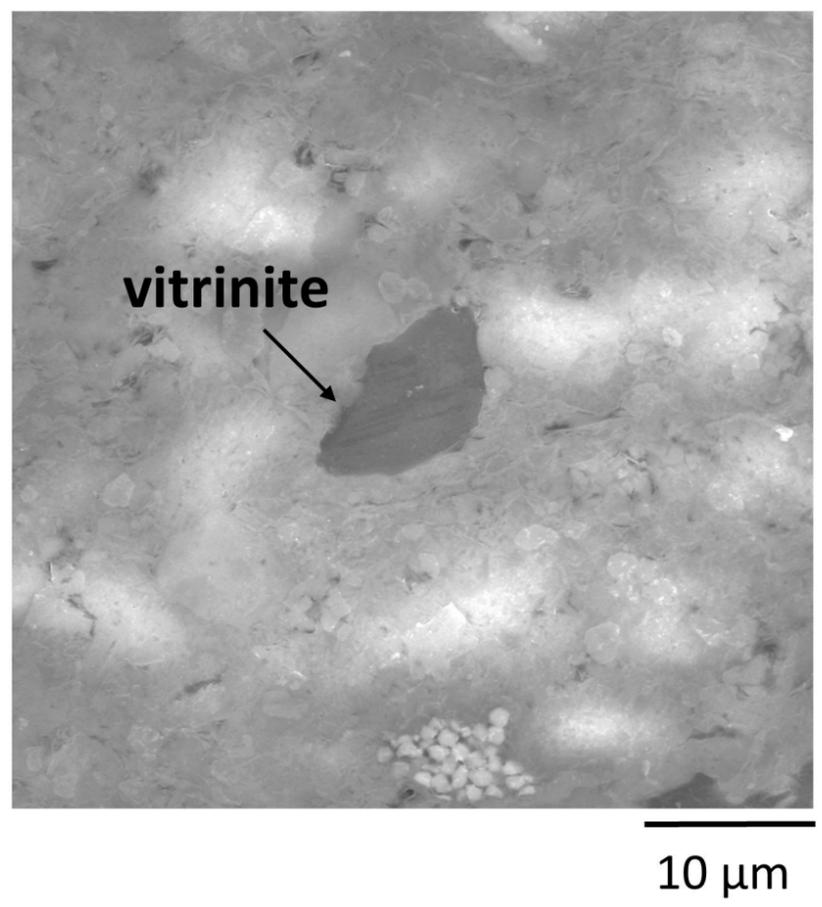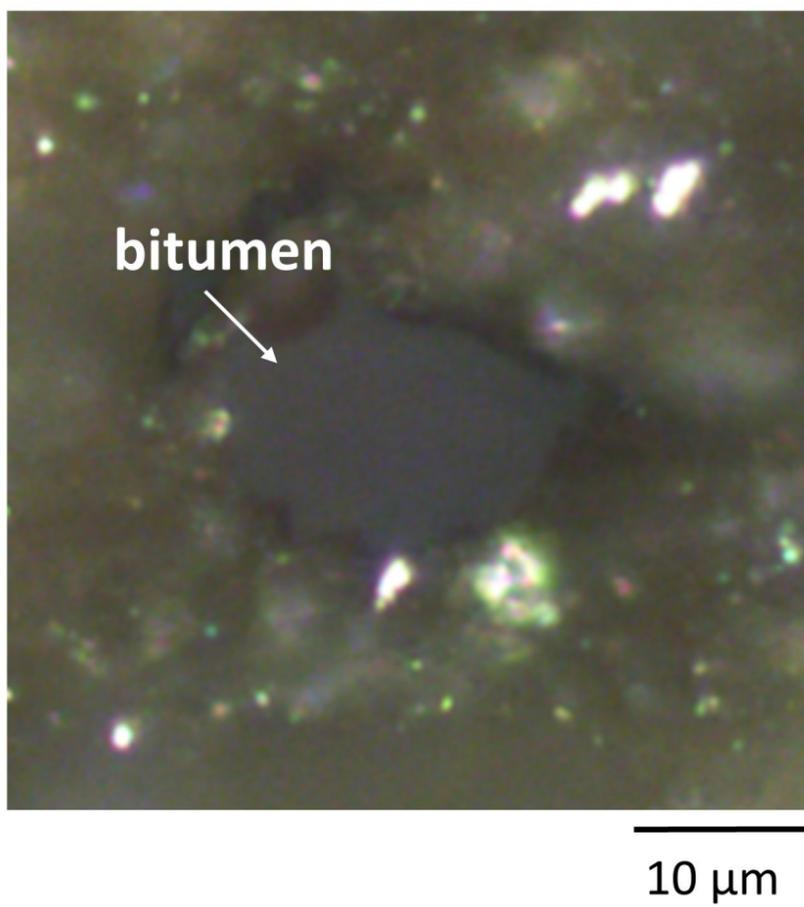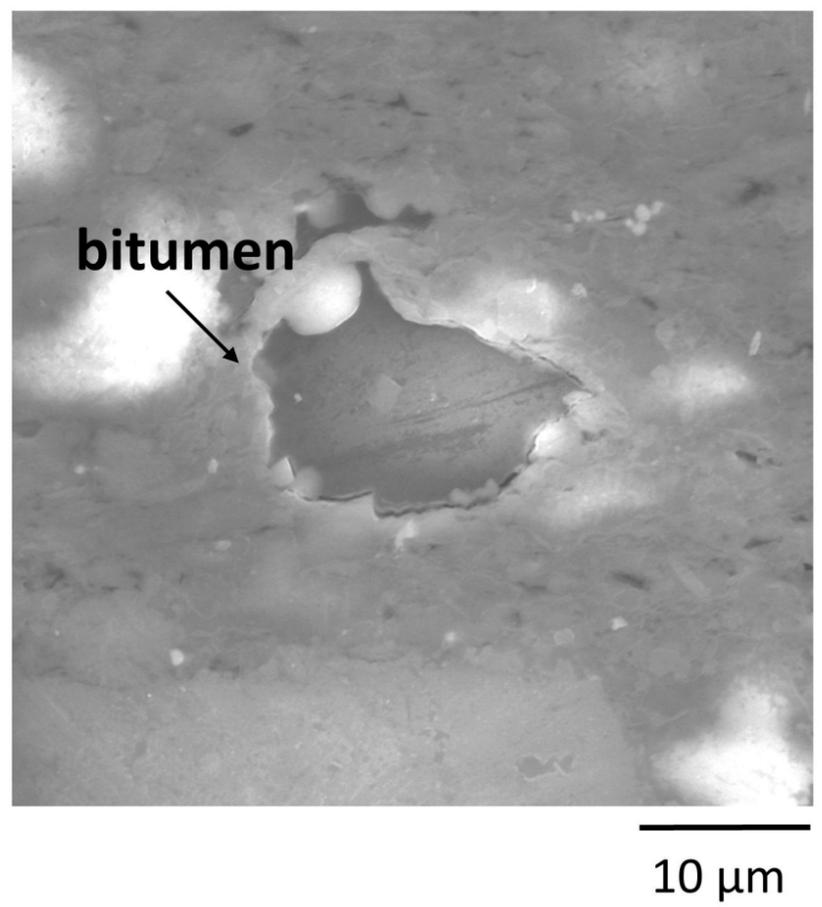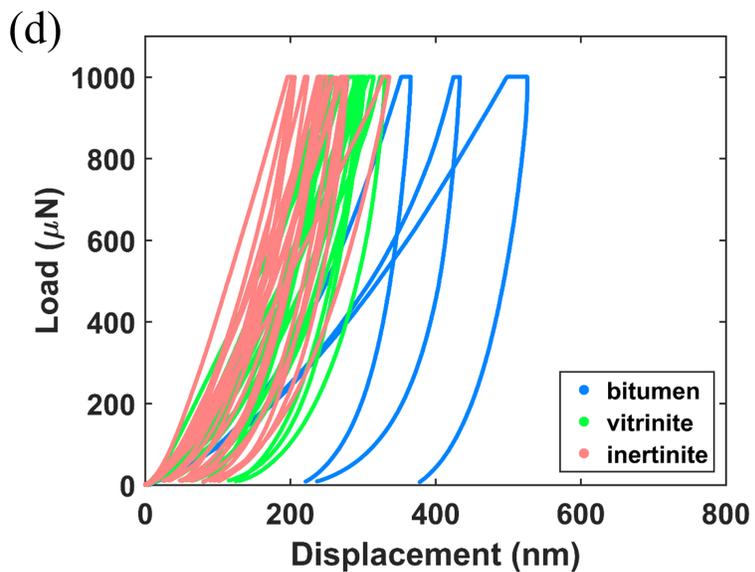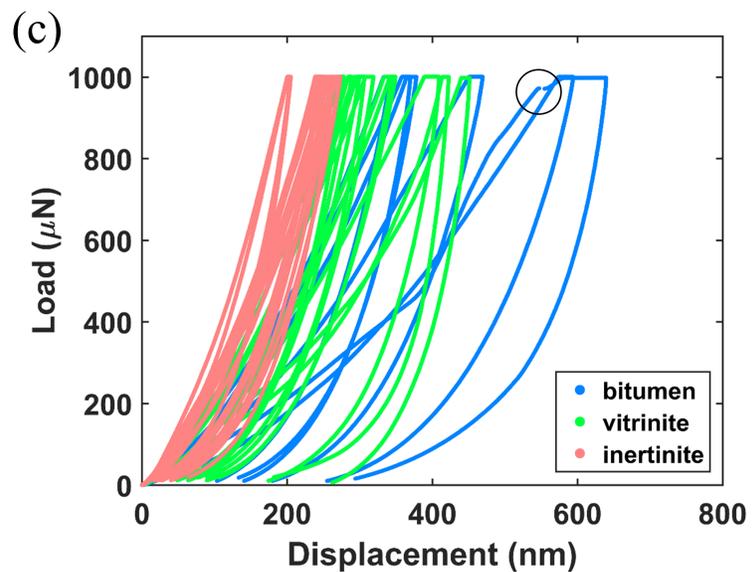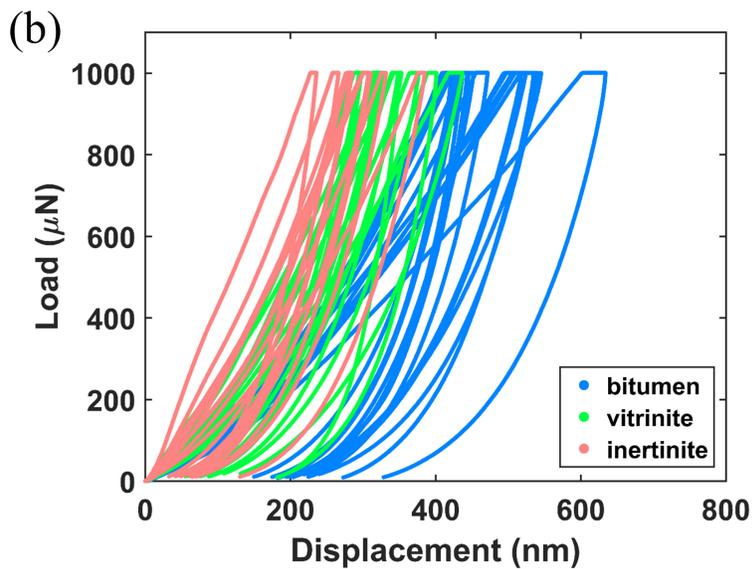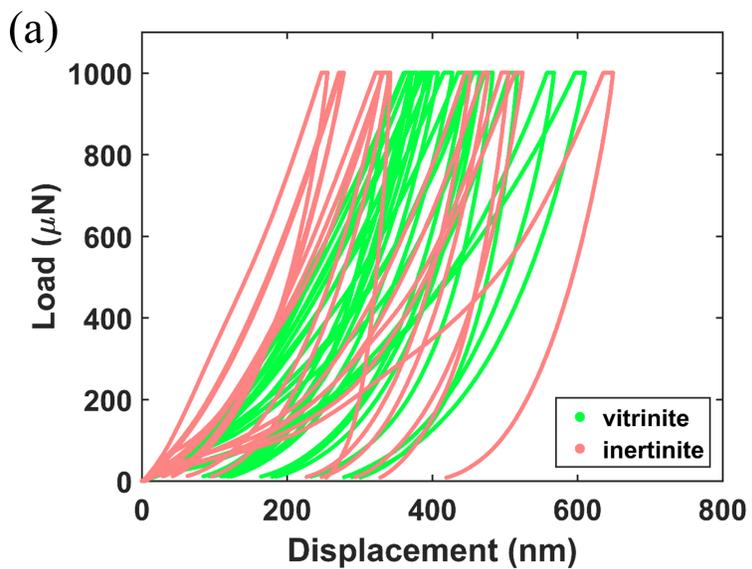
Figure 4.

Figure 5.

Figure 6.

Figure 7.

(a) inertinite in J4 (higher R_o)

10 μm

(b)

(c) inertinite in J1

10 μm

(d)

Figure 8.