Implications of the 3D micro scale coal characteristics along with Raman stress mapping of the scratch tracks

G.L. Manjunath, Rajesh R. Nair *

Department of Ocean Engineering, IIT Madras, Chennai, India

A R T I C L E   I N F O

Article history:
Received 21 February 2015
Received in revised form 24 February 2015
Accepted 24 February 2015
Available online 4 March 2015

Keywords:
Fracture toughness
Micro indentation
Micro scratch test
Raman stress mapping
Maceral
Petrography

A B S T R A C T

The microscale interfacial characteristics of coal are studied based on the analysis of mechanical behavior of individual elements contributing to coal heterogeneity with respect to high volatile bituminous A of coal, from Singrauli coal field, Madhya Pradesh, India. Micro-scale delineation of elastic plastic properties of coal by grid micro-indentation test and fracture toughness was accomplished by micro-scratch test on colliotene maceral. Micro-scratch test was performed in different directions on and critical points of failure were diagnosed by measuring acoustic emissions and tangential force. The 3D heterogeneity of coal is studied based on the damages at certain point of loads known as critical points with respect to distribution of macerals derived from coal petrography. Damages were observed at higher critical loads on samples collected from 329.7–330.9 m depth. Raman stress mapping at critical points of scratch track revealed a spectral shift due to stress inversion. Shift from 1576–1593 cm\(^{-1}\) for graphite band (G), and from 1344–1357 cm\(^{-1}\) for disordered Carbon (D) band indicate the nature of stress and deformation occurred for coal bulk sample. Raman spectra variation for maceral colliotene is studied and compared with coal bulk samples behaviour a shift from 1574–1597 cm\(^{-1}\) for G, and from 1346–1356 cm\(^{-1}\) for D.

© 2015 Elsevier B.V. All rights reserved.

1. Introduction

The heterogeneous complex structural property of coal affects its behavior and chemical reactivity. Coal characteristics based on thermochemical-mechanical variations must be analyzed thoroughly. Broader perspective of coal heterogeneity is an important requirement for the exploration of deep coal by means of Underground Coal Gasification (UCG) (Shahidzadeh-Bonn et al., 2005). Fundamentally coal heterogeneity depends on several aspects such as layering and the litho type microlithotype occurrences, and the stress fields that along with mineral matter cleats orientation, macro-to-nano-porosity and chemical mineralogy compositions.

Exploration of deep coal by UCG requires higher level of cognizance in coal heterogeneity in order to better implement geomechanical designs (Kanitpanyacharoen et al., 2011). A fundamental micro scale analysis of geomaterials, their nature and response to loading and stress distribution leading to failure is significant (Mahabadi et al., 2012). In the case of ceramics, grid micro-indentation test and micro-scratch test play a significant role in evaluating their mechanical properties (Akono and Ulm, 2011; Akono et al., 2011). These methods are quite effective in providing an overall idea of the global mechanical response of materials based on statistical distribution of the measured parameters. Material characterization techniques such as scanning electron microscopy, transmission electron microscopy and other non-destructive testing methods probe and map the surface and sub-surface structure of a material, thus revealing their chemical composition, composition variation in degree of ordering structure and ultrasonic studies. Rationalizing coal heterogeneity requires measuring the mechanical behavior of individual elements. Such studies lead to improved measurement of stress mapping and its nature along with the causes of failure. The texture of coal and its heterogeneity can be elucidated by advanced techniques such as Raman analysis (Guedes et al., 2010; Guo and Bustin, 1998; Marques et al., 2009).

With recent advances in image analysis, confocal micro-Raman spectroscopy has been extensively used for wide range of studies. Raman spectroscopy is used in many varied fields and is useful where non-destructive, microscopic, chemical analysis and imaging is required such as characterizing the chemical composition, internal structure, grain orientations, texture and phase distribution. It is also useful for mapping stress and strain and quantifying residual stress and fracture strength in 3 dimensions (Becker et al., 2007; Beyssac et al., 2003a,b; Dumpala,...
Raman spectroscopy was coupled with Scanning Microwave Microscopy a chip placed inside the coal this combination to achieve 100nm spatial resolutions for coal structure. Along with the surface morphology and chemical variations of coal and to overcome the shortcomings of Transmission Electron Microscopy (Potgieter-Vermaak et al., 2011; Tselev et al., 2014). Similarly, implementing micro-Raman imaging analysis along with X-ray diffraction (XRD) and Fourier Transfer Infrared spectroscopy (FTIR) to scrutinize the crystalline structure helps in determining the coal rank (Mastalerz and Bustin, 1993; Sonibare et al., 2010) and also in measuring the properties of partially ordered materials (Beyssac et al., 2003a,b; Ferrari and Basko, 2013).

This paper investigates 3D heterogeneity of coal at micro scale level based on grid micro-indentation test and micro-scratch test carried out in three different directions on coal sample. The grid indentation techniques for yielding accurate mechanical properties of Young’s modulus and hardness of the heterogeneity based on low load applications is performed on coal (Chen and Huang, 1963; Das, 1972; Mukherjee et al., 1989; Nandi et al., 1977; Tiryaki, 2005). Statistical analysis was performed for analyzing indentation properties based on histogram in obtaining mean values of the heterogeneous media.

The specimens were retrieved from two different depth ranges of 329.7 – 330.9 m and 332.9 – 335.6 m. At the critical points of failures, Raman analysis was performed to acknowledge the nature of stress inversion. The Raman spectrum, along with micro imaging of the critical scratch track provides the nature of stress, to which it is subjected, based on the mechanical response of coal. The general physical characteristics of coal of Singrauli coal field shows basically banded nature. Based on bright and dull bands it is inferred that vitrinite and internite are the dominant maceral groups with minor concentration of liptinite group with rank of coal vary from A-F. Chemical properties of formation contains ash (wt%) : 22.33, volatile matter (wt%) : 44.64, fixed carbon (wt%) : 55.36. Petrographic composition of coals: colliotelite (vol%) : 23 to 33; Macrinite (vol%) : 1 – 2.66; Fusinite (vol%) : 18 to 23.

The study helps in a thorough understanding of coal 3D heterogeneity relating to Raman stress mapping and also evaluation of the mechanical properties. The outcome of these experiments acts

<table>
<thead>
<tr>
<th>Formation</th>
<th>Lithology</th>
<th>Formation thickness in mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raniganj</td>
<td>Sandstone and shale</td>
<td>150</td>
</tr>
<tr>
<td>Jhingurdah top seam</td>
<td>131–138</td>
<td></td>
</tr>
<tr>
<td>Sandstone and shale</td>
<td>39–58</td>
<td></td>
</tr>
<tr>
<td>Jhingurdah bottom seam</td>
<td>10–15</td>
<td></td>
</tr>
<tr>
<td>Sandstone, shale with coal string</td>
<td>60</td>
<td></td>
</tr>
<tr>
<td>Barren measure</td>
<td>Ferruginous clayey sandstone</td>
<td>125</td>
</tr>
<tr>
<td>Barakar</td>
<td>Sandstone and thin coal beds</td>
<td>45–70</td>
</tr>
<tr>
<td>Pumhari seam (local)</td>
<td>1–2</td>
<td></td>
</tr>
<tr>
<td>Sandstone and shale</td>
<td>110–125</td>
<td></td>
</tr>
<tr>
<td>Khaolai seam (local)</td>
<td>1–2</td>
<td></td>
</tr>
<tr>
<td>Sandstone and shale</td>
<td>30–40</td>
<td></td>
</tr>
<tr>
<td>Purewa top seam</td>
<td>8–12</td>
<td></td>
</tr>
<tr>
<td>Fine to coarse grained sandstone</td>
<td>0–60</td>
<td></td>
</tr>
<tr>
<td>Purewa bottom seam</td>
<td>10–40</td>
<td></td>
</tr>
<tr>
<td>Sandstone and shale</td>
<td>45–75</td>
<td></td>
</tr>
<tr>
<td>Turra seam</td>
<td>14–23</td>
<td></td>
</tr>
<tr>
<td>Sandstone and shale</td>
<td>45–90</td>
<td></td>
</tr>
<tr>
<td>Kota seam</td>
<td>1–3</td>
<td></td>
</tr>
<tr>
<td>Sandstone and shale</td>
<td>150–250</td>
<td></td>
</tr>
<tr>
<td>Bijawar</td>
<td>Phyllites and quartzites</td>
<td></td>
</tr>
</tbody>
</table>
as input in analysing stress percolation in global finite element geomechanical modeling (Burnley, 2013). Mahabadi et al. (2014) used inputs from such micro indentation and scratch tests in accurately predicting the tensile strength and failure behaviour of rock sample.

2. Materials and sample preparation procedures

2.1. Materials

The samples belong to depths ranging from 329.7 to 330.9 m and from 332.9 to 335.6 m. These specimens were collected from Gondhbehra (Ujheni), Singrauli in Madhya Pradesh. The present studied coal field, Singrauli is covered by Raniganj formation. This formation is underlined by Barren measures, Barakar and Talchir formations shown in Fig. 1 and samples are collected from Jhingurdah bottom seam Table 1. An East West trending boundary fault (an offshoot of son Narmanda linement). Covers the northern limit of the study area and four other faults have been deciphered within the main block. The seven regions of coal seams that occur in Barakar formation is comprised within depth range 244.23–606.75 m (Misra and Singh, 1990) ranging from A–F (Singh et al., 2014). Structurally this coal block strike:

<table>
<thead>
<tr>
<th>Compound</th>
<th>Chemical formula</th>
<th>hkl</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz</td>
<td>SiO₂</td>
<td>011</td>
</tr>
<tr>
<td>Graphite</td>
<td>C</td>
<td>002</td>
</tr>
<tr>
<td>Carbon</td>
<td>C</td>
<td>005</td>
</tr>
<tr>
<td>Kaolinite</td>
<td>Al₂(Si₂O₅)(OH)₄</td>
<td>001</td>
</tr>
<tr>
<td>Silicon oxide</td>
<td>SiO₂</td>
<td>011</td>
</tr>
<tr>
<td>Lead Arsenate</td>
<td>Pb(As₂O₅)</td>
<td>001</td>
</tr>
<tr>
<td>Chlorine oxide</td>
<td>ClO₂</td>
<td>211</td>
</tr>
<tr>
<td>Boron Carbon nitride</td>
<td>B0.47C0.23N0.30</td>
<td>110</td>
</tr>
<tr>
<td>Boron nitride</td>
<td>BN</td>
<td>002</td>
</tr>
<tr>
<td>Cesium</td>
<td>Cs</td>
<td>111</td>
</tr>
</tbody>
</table>

Table 2
Range of minerals identified by XRD.

Fig. 2. (a) Surface of the coal sample showing grid indentation by Berkovich indenter. (b) Load vs. displacement for coal takes from two different depths.; Petrography study of coal indented area in white light (c) & fluorescence light (d); (e) Schematic diagram of the conical scratch test set up; (f) & (g) Scanning electron microscopy image of scratched area of coal extracted from depths 329.7–330.9 m and 332.9–335.6 m; (h) Optical image of the sample selected for Raman imaging; Petrography study of coal indented area in white light (i) and fluorescence light (j); (k) Raman micro-imaging of the selected area: purple indicates carbon, red indicates silicon, blue indicates sulfur, green indicates titanium, sky blue indicates molybdenum, and black indicates the presence of fluorescence.
NE-SW with minor swings low dip of 3–5° towards SE. Throw of major fault is ranging from 100 to 120 m trending NE-SW with throw towards west. The other three faults, the throw is ranging from 15–30 m.

2.2. Sample preparation

Cutting, sizing and polishing are the major operations involved. The sample was cut to the required shape using a diamond saw cutter and the sizing operation was performed using aluminium oxide grits extending from coarse 240 to fine 3000 to achieve a high polish. The orientation of the core sample is perpendicular to the bedding plane. The faces of the sample were polished enough to parallelize them to a dimension of 10 mm × 10 mm × 3 mm. Diamond polishing was done to achieve high degree of surface finish required for the experiments.

3. Experimental study details

3.1. X-ray diffraction methods

XRD analysis was performed to determine the degree of ordering the carbon stacking structure in coal, fraction of amorphous carbon, interlayer spacing of crystalline structure and its sizes. The existence of the peaks confirms the presence of crystalline structures in coal and other background intensity of diffractions associated with highly disordered structures. The mineralogy details based on the XRD studies along with Miller indices are shown in Table 1. Powdered coal samples were placed on the glass slide and X-ray was passed...
studies based on white and applied load of 630 mN and frame stiffness of 7.08 × 10^6 N/m, strain rate of the indenter. A grid indentation array of 7 × 7 with spacing of 150 μm, experiments were performed with Agilent instrument using Berkovich indenter. The sample shown in Fig. 2(g) (332.90 m lower depth range) offers more resistance to scratch compared to the sample shown in Fig. 2(f) (329.70–330.90 m lower depth range), which highlights the direct dependency of compactness and scratch resistance on depth.

3.2. Nano-indentation testing

Coal heterogeneity depends on the presence of nano and micro pores and the macerals and individual macerals in that group and nano-indentation useful approach to determine elastoplastic nature of coal. Understanding the complex nature of coal geology by grid indentation techniques and measuring the Young’s modulus and hardness. Experiments were performed with Agilent instrument using Berkovich indenter. A grid indentation array of 7 × 7 with spacing of 150 μm, applied load of 630 mN and frame stiffness of 7.08 × 10^6 N/m, strain rate of 0.05/s for a frequency of 45Hz, 5s pause and depth range of 2750–3250 nm. Fig. 2(a) discloses the nano-indentation images and Fig. 2(b) shows the load vs. displacement behavior of the samples. Petrography analysis of indented area is shown in Fig. 2(c) and (d), the reflectance of which is more due to due to the indentation. Petrography studies based on white and fluorescence’s light is studied. Fig. 2(c) indicates the mineral matter in variegated colour and darkest gray indicates the various grades of vitrinite (colliotenite) reflectance. In Fig. 2(d), black colour indicates the organic matter.

3.3. Micro-scratch testing

Micro-scratch testing was performed with a CSM instrument using a Rockwell diamond conical probe with a half apex angle of θ = 60°, hemispherical tip radius of R = 200 μm and transition from sphere to cone d/R = 0.132. Fig. 2(e) is the schematic representation of the experimental set up along with the dimension of the probe. A vertical load of FV was applied and the resultant scratch depth, acoustic emission, groove size and tangential load FT were recorded by the software. Progressive load of 1–50 N was applied with a loading rate of 49 N/min and with a speed of 3 mm/min up to a length of 3 mm. The fracture toughness Kc is measured using a conical scratch test based on the frictional force in tangential direction Fg, diameter of the cone d, and half-apex angle θ as shown in Eq. (1).

\[
\frac{F_g}{K_c d^{1/2}} = 2 \sqrt{\frac{\tan \theta}{\cos \theta}}
\]

Fig. 2(f) & (g) show SEM images of scratch tracks in coal at different depths. The sample shown in Fig. 2(g) (332.90–335.60 m higher depth range) offers more resistance to scratch compared to the sample shown in Fig. 2(f) (329.70–330.90 m lower depth range), which highlights the direct dependency of compactness and scratch resistance on depth.

3.4. Raman analysis

Raman measurements were performed with Witec machine using the Confocal Raman Microscope alpha-300 system comprising an ultra-high throughput system 300 spectrograph coupled with Peltier cooled charge coupled detector. An Nd: YAG 532nm laser was used for excitation and a 50 × objective lens was used for laser focus. Backscatter light from the sample was guided to the spectrometer through a 50 μm multimode optical fiber. The Raman image along with the wavelength data for the defined area was obtained as shown in Fig. 2(k). The petrography studies for the selected areas of coal are studied by Raman analysis and the coal macerals are identified to be mainly colloliteline. Fig. 2(i) and (j). A defined area was selected over the optical image, as indicated in Fig. 2(h) and a total of 14,400 Raman spectra were collected over this area to obtain the micro image, as shown in Fig. 2(k).

4. Results and discussions

4.1. Nano-indentation

The nano-indentation method was preferred due to the presence of micro and nano sized pores in the sample same as used by (Tselev et al., 2014), which demands accurate determination of heterogeneity. The nano elasto-plastic behavior of the coal sample, based on statistical analysis, is illustrated in Fig. 3(a) and (b). In order to estimate the hardness and Young’s modulus precisely, the indenter should touch the pore valley. The histogram shows the decreased Young’s modulus and increased hardness and Fig. 3(a) and (b) shows the effect of maceral composition from petrography studies Fig. 2(c) and (d) along with heterogeneity. Table 3 summarizes the mechanical properties of the coal based on the nano-indentation test based on a single run of 7 × 7 arrays for each sample. Fig. 3(a) shows indentation modulus values of the sample at a lower depth range (332.9–335.6 m) and a higher depth range (329.7–330.9 m). Due to high compaction macerals are more intact, the samples from greater depth are more ductile. Fig. 3(b) represents the hardness modulus of the samples and the

![Fig. 3. (a) Indentation modulus histogram; (b) hardness modulus histogram.](image-url)
Fig. 4. (a) Tangential force and acoustic emission plots obtained against normal loading in X11 direction. (b) Tangential force and acoustic emission plots obtained against normal loading in X12 direction. (c) Tangential force and acoustic emission plots obtained against normal loading in X13 direction. (d) Histogram indicating fracture behavior modulus in different layers. (e) SEM images of scratch length at × 1mm. (f) SEM images of scratch length at × 500μm. Petrographic image of scratched area in white (g) and fluorescence light (h).
Fig. 5. (a) Tangential force and acoustic emission plots obtained against normal loading in X11 direction. (b) Tangential force and acoustic emission plots obtained against normal loading in X12 direction. (c) Tangential force and acoustic emission plots obtained against normal loading in X13 direction. (d) Histogram indicating fracture behavior modulus in different layers. (e) SEM images of scratch length at ×1mm. (f) SEM images of scratch length at ×500μm. Petrographic image of scratched area in white (g) and fluorescence light (h).
samples obtained from greater depth was found to be harder. The presence of lesser cleats is responsible for the increased hardness of the sample at greater depth along with macerals and microlithotype.

4.2. Micro-scratch test

Figs. 4 to 5 show the results of micro-scratch test on samples collected from two different depths, where acoustic emission and tangential force are plotted against progressive normal loads. SEM images of the scratched area are analyzed after the experimentation and a series of failure events were identified by acoustic emission peaks, marked as critical loads, along with tangential loadings due to change in frictional contact as shown in Fig. 4(a–c) obtained from a depth of 329.70–330.90 m and 5(a–c) from a depth of 332.90–335.60 m. Observation reveals that at critical loads of 4, 15 and 38 N, the series of failures occur in X11-direction, as shown in Fig. 4(a); at critical loads of 3, 38 and 48 N, the series of failures occur in X12-direction, as shown in Fig. 4(b); and at critical loads of 18, 29 and 38 N, the series of failures occur in X13-direction, as shown in Fig. 4(c) owing to the release of elastic energy. Fig. 4(d) is a histogram that indicates the fracture behavior of the samples along different directions (X11, X12 and X13) and Table 4 exposes the fracture toughness values based on Eq. (1) corresponding to different directions shown in Fig. 2c. The SEM image shows the scratch track; initially, no failure occurs and after certain distances a transition in stress inversion from compressive to tensile, leading to shearing as observed in Fig. 2c.

Table 4 Fracture toughness in different directions.

<table>
<thead>
<tr>
<th>Directions</th>
<th>Fracture toughness MPa√m initial sample depth (329.7–330.9 m)</th>
<th>Fracture toughness MPa√m deeper sample depth (332.9–335.6 m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>X11</td>
<td>0.0749 ± 0.0157</td>
<td>0.0768 ± 0.0146</td>
</tr>
<tr>
<td>X12</td>
<td>0.0804 ± 0.0143</td>
<td>0.0777 ± 0.0135</td>
</tr>
<tr>
<td>X13</td>
<td>0.0656 ± 0.0155</td>
<td>0.07044 ± 0.0145</td>
</tr>
</tbody>
</table>

The experiment was repeated with samples from a different depth and the results are disclosed in Fig. 5. Observation uncovers a series of failure events at critical loads of 8, 22 and, 25 N along X11-direction, as in Fig. 5(a); 46 N along X12-direction, as shown in Fig. 5(b) and 31 and 45 N along X13-direction, as in Fig. 5(c), on account of the release of elastic energy. SEM analysis performed on scratch, shown in Fig. 5(a), indicates damages at lengths 0.97 mm, 1.58 mm and 2.1 mm which are represented in Fig. 5(e) and (f). The critically damaged zone is selected for petrography from Fig. 4(f) shown in studied in Fig. 4(g) and (h). Light Gray is vitrinite (colotelinite) and mineral ground mass variegated in colour Fig. 4(g) and degraded bitumen showing yellow fluorescence in Fig. 4(h).

The experiment was repeated with samples from a different depth and the results are disclosed in Fig. 5. Observation uncovers a series of failure events at critical loads of 8, 22 and, 25 N along X11-direction, as in Fig. 5(a); 46 N along X12-direction, as shown in Fig. 5(b) and 31 and 45 N along X13-direction, as in Fig. 5(c), on account of the release of elastic energy. SEM analysis performed on scratch, shown in Fig. 5(a), indicates damages at lengths 0.97 mm, 1.58 mm and 2.1 mm which are represented in Fig. 5(e) and (f). Fig. 5(g) shows the fracture behavior of samples in X11, X12 and X13 directions. Similarly critically damaged zone is selected for petrography from Fig. 5(g) shown in studied in Fig. 5(g) and (h). Light Gray is vitrinite (colotelinite) and mineral ground mass variegated in colour Fig. 5(g) and degraded bitumen showing yellow fluorescence in Fig. 5(h). Compared to Fig. 5(g) in Fig. 4(g) the maceral colotelinite is highly damaged.

Micro-scratch test results confirm more fracture toughness along X13 direction, compared to other directions, for samples from lower depth range (329.7–330.9 m). According to Fig. 5(d), for samples belonging to greater depth range (332.9–335.6 m), fracture toughness was found more along the X12 direction, in comparison with other directions. The acoustic emission and tangential force curves indicate the characteristics of the coal samples from different depths. In this regard, the sample obtained from a greater depth offered resistance to scratch due to increased hardness and compaction based on macerals and microlithotype characteristics. Samples exhibited quasi-brittle failures, as a result of shearing at the surface due to high compaction, around the indented area. The scratch test based on number of acoustic emission peaks indicates weakest spots and the level of heterogeneity.

4.3. Raman stress mapping analysis

Since the micro-scratch test is destructive in nature, the applied progressive normal load reaches yield limit and causes failures. The critically damaged zone of colotelinite from petrography studies shown in Fig. 4(g) and (h) is considered for Raman analysis Fig. 6(a) and (b). Raman spectroscopy analysis was performed on the scratch tracks by K-means cluster analysis method and critically damaged regions around the failure points were analyzed, as in Fig. 6(c) and (d). A critically scratched area at a lower depth was selected for Raman analysis as it is more susceptible to damages. The characteristic Raman peak for uncompressed and stressed regions of coal bulk sample was studied. Normally, for an unstressed region of coal the Raman peak is noticed at 1593 cm\(^{-1}\), known as graphite (G) band and at 1345 cm\(^{-1}\), referred to as disordered (D) band of carbon. For stressed region, a spectral shift from 1576 to 1593 cm\(^{-1}\) is observed for G band and a spectral shift from 1344 to 1357 cm\(^{-1}\) is observed for D band. The behavior of peak shifting from a higher to lower side indicates the presence of compressive and tensile stresses inside and outside the scratch track, as shown in Fig. 6(e).

A band of spectrum ranging from 1576 to 1593 cm\(^{-1}\) is selected to obtain the Raman images and to perform cluster analysis for obtaining different color intensity contrasts Fig. 6(e). The degree of graphitization was measured based on G and D peak intensities, which indicates the disorderliness in the carbon structure. The increase in G band indicates higher graphite content and the decrease in D band indicates the disorder in carbon structures. Normally, the ratio of intensity of G to intensity of D indicates the structural properties of coal. A decrease in the value of this ratio indicates the destroyed tensile mode, and an increase in its value indicates the compressive mode of coal during the scratch-test (Zhao et al., 2014).

A lower peak value in Fig. 6(e) indicates the tensile state of stress that makes it less compact, while a higher peak value indicates the compressive nature of stress, which makes it more compact. Stress mapping at 1593 cm\(^{-1}\) Fig. 6(e) X graph indicates the presence of compressive stress and at 1576 cm\(^{-1}\) Fig. 6(e) Z graph indicates the presence of tensile stress field. Transition from compressive to tensile indicates the severity in deformation and cause of failure events. Tests were repeated focusing on collotelinite and compared with coal bulk results shown in Fig. 6(f). The Raman analysis of the individual maceral colotelinite indicates 1354–1592 cm\(^{-1}\) for X, 1346–1597 cm\(^{-1}\) Y and 1356–1574 cm\(^{-1}\) for Z, no much variation in G band is noticed compared to D band shows slight variations.

While comparing the images of scratch at different depths of coal, it was found that the failure mechanisms were similar and gradual damage was observed in a brittle mode at certain lengths. The regions away from the scratch tracks experienced the impact of failures due to compressive shearing by the indenter. Decohesion occurred due to compressive stresses induced by the indenter and developed a stress inversion from compressive to tensile, leading to shearing and failures. Coal from a greater depth exhibited increased hardness and increased resistance to deformation due to increased compaction.

5. Conclusions

Scratch characteristics of coal at different depths were selected for experimental purposes by applying progressive normal load within the range of 1–50 N and nano-indentation test was successfully conducted to measure the mechanical properties. Scratch tracks were analyzed step-by-step using SEM and Raman spectroscopy. Investigation reveals that events of failure occurred at critical loads of 4, 15, 38 N in X11-direction and critical loads of 3, 38, 48 N in X12-direction; at critical loads of 18, 29, 38 N, the series of failures occur in X13-direction for

\[ \text{Fracture toughness in different directions.} \]

\[ \begin{array}{l|l|l|l}
\text{Directions} & \text{Fracture toughness MPa√m} & \text{Fracture toughness MPa√m} \\
\hline
\text{initial sample depth} & \text{deeper sample depth} & \\
(329.7–330.9 m) & (332.9–335.6 m) & \\
\hline
X11 & 0.0749 ± 0.0157 & 0.0768 ± 0.0146 \\
X12 & 0.0804 ± 0.0143 & 0.0777 ± 0.0135 \\
X13 & 0.0656 ± 0.0155 & 0.07044 ± 0.0145 \\
\end{array} \]
samples from 329.7–330.9 m depth. The failure events occur at critical loads of 8, 22, 25 N in X11-direction, 46 N in X12-direction and 31 N, 45 N in X13-direction for samples at 332.9–335.6 m depths. The macerals are more intact at higher depth coal samples based on petrography analysis. Specific conclusions can be made using Raman stress mapping for measuring strain associated with the stress inversion, from compressive to tensile stresses, leading to failures of brittle nature. Under gradual loading condition, at certain points the gradually damaged zones are detected by progressive loadings. The current study is also beneficial for calculating the stress orientation at different depths.

Fig. 6. Petrographic image of scratched area in white (a) and fluorescence light (b). (c) Raman intensity imaging on the selected optical area. (d) Cluster analysis for Raman stress mapping 1, 2, 3. (e) Raman spectra for 1, 2, 3 corresponding color images along with wave numbers X, Y & Z graphs of coal bulk. (f) Raman spectra of for 1, 2, 3 corresponding color images along with wave numbers X, Y & Z graphs of collotelinite.
by providing an understanding of fundamentals of geomechanics, as applied to coal, of selected reservoirs.

Acknowledgement

The authors would like to thank Central Mine Planning & Design Institute Limited (CMPDI), Ranchi, Jharkhand, India, for providing the coal sample for carrying out the research.

References


