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

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ABSTRACT

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 collotenite 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 332.9–335.6 m depth compared to 329.7–330.9 m depth samples owing to less heterogeneity and increased compaction. 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 the deformation occurred for coal bulk sample. Raman spectra variation for maceral collotelinite 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.

1. Introduction

The heterogeneous complex structural property of coal affects its behavior and chemical reactivity. Coal characteristics based on thermo-chemico-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

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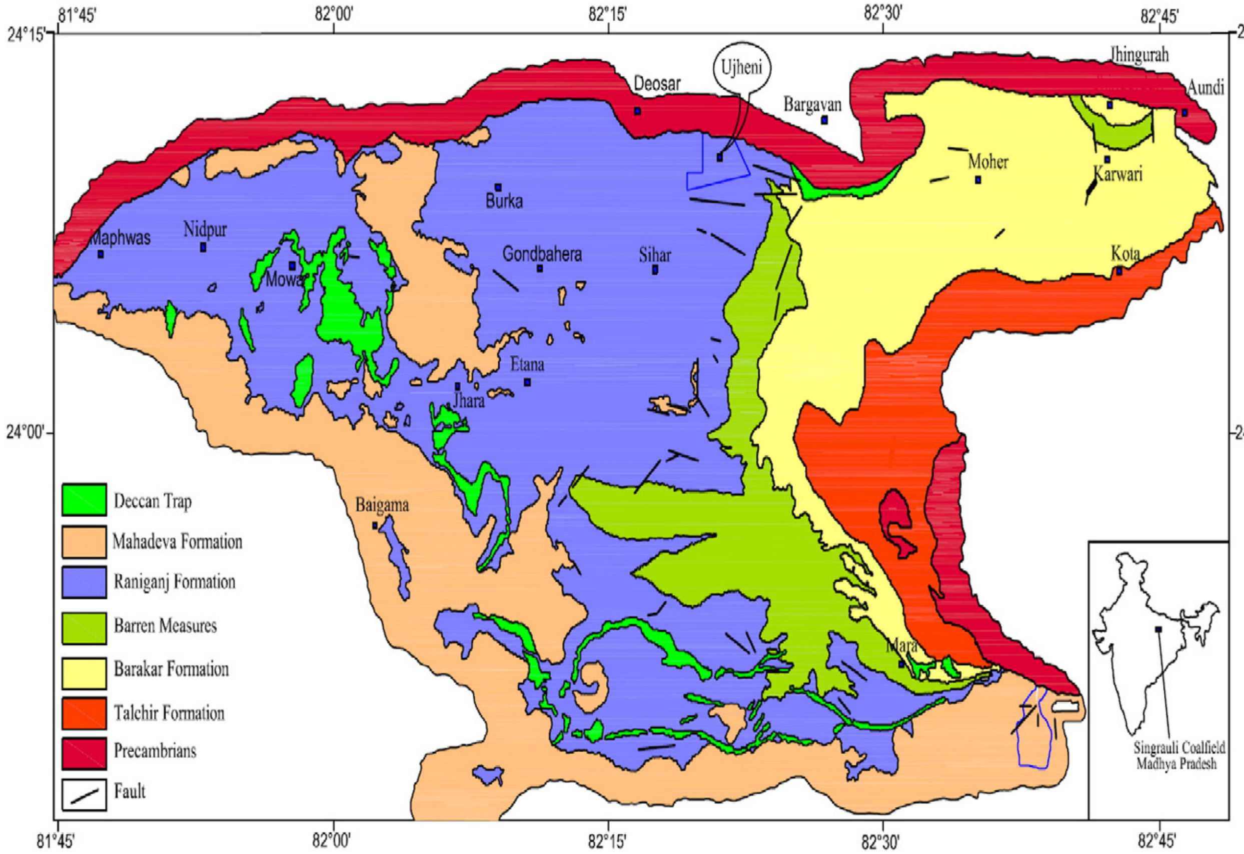

Fig. 1. Geological map of the Singrauli coal field showing the area of the study (Geological survey of India).

et al., 2014; Ghosh et al., 2008; Kang et al., 2005; López-Honorato et al., 2010; Puech et al., 2000; Ryu et al., 2012; Sadezky et al., 2005).

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, voltaile matter (wt%) : 44.64, fixed carbon (wt%): 55.36. Petrographic composition of coals: collotelinite (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 1
Stratigraphic formation sequence of Singrauli coalfield (Raja Rao, 1983; Singh et al., 2014).

Formation	Lithology	Formation thickness in mm
Raniganj	Sandstone and shale	150
	Jhingurdah top seam	131–138
	Sandstone and shale	39–58
	Jhingurdah bottom seam	10–15
	Sandstone, shale with coal string	60
Barren measure	Ferruginous clayey sandstone	125
Barakar	Sandstone and thin coal beds	45–70
	Panihari seam (local)	1–2
	Sandstone and shale	110–125
	Khadia seam (local)	1–2
	Sandstone and shale	30–40
	Purewa top seam	8–12
	Fine to coarse grained sandstone	0–60
	Purewa bottom seam	10–40
	Sandstone and shale	45–75
	Turra seam	14–23
	Sandstone and shale	45–90
	Kota seam	1–3
	Sandstone and shale	150–250
	Unconformity	
Bijawar	Phyllites and quartzites	