



# Characteristics of coal re-oxidation based on microstructural and spectral observation



Liang Yuntao <sup>a,b</sup>, Tian Fuchao <sup>a,b,\*</sup>, Luo Haizhu <sup>b</sup>, Tang Hui <sup>b</sup>

<sup>a</sup> School of Safety Engineering, China University of Mining & Technology, Xuzhou 221116, China

<sup>b</sup> State Key Laboratory of Coal Mine Safety Technology, China Coal Technology & Engineering Group of Shenyang Research Institute, Shenyang 110016, China

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## ABSTRACT

In order to investigate the characteristics of re-oxidation of residual coal in goafs in close coal seam mining, scanning electron microscope and infrared spectrometer are used to study the changes of coal microstructure and chemical reaction of functional groups of eight coal samples at different ranks. Result shows that after initial oxidation, the surface morphology of pore are different, and the porosity of coal is increased and the oxygen adsorption capacity of coal is improved. The change of coal molecular structure and presence of a large amount of active oxygen-containing functional groups lead to increasing tendency of coal to further oxidation. In addition, the higher lever of the initial oxidation is, the easier the re-oxidation occurs.

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## 1. Introduction

Due to depletion of shallow buried coal resources, deep coal seams are being increasingly extracted. As coal left in goafs and coal pillars have been originally oxidized, the characteristics of self-heating spontaneous combustion of the coal has changed. The previous oxidation is supposed to accelerate secondary oxidation procedure and may lead to open flame in cases of close coal seam group during combined mining or goaf remaining coal mining. Presence of gas in goaf increases the risk of explosion which is one major threat for mining safety [1,2]. A huge amount of studies have been conducted on the fundamental theory of coal spontaneous combustion. However, they mainly focused on the characteristics of the first oxidation procedure of the coal. Based on microstructural analysis of the coal, foreign scholars revealed the relationships of the microstructure of the coal to gas adsorption of different coal at different temperatures. However, microstructural study of secondary coal oxidation is not conducted yet [3–6]. Scholars in China often focus on the characteristics of coal oxidation procedure. For example, Zhang et al. analyzed the variation of the gas contents during coal oxidation using spectral technology [7–11]. To better understand the mechanism of secondary oxidation of the coal, Deng studied the macro parameters and micro surface characteristics

based on adsorption experiment and infrared spectrum measurement, and key parameters of the secondary oxidation of the coal samples at different oxidation stages were obtained [12]. Wen studied the generating rate of CO, consumption rate of oxygen and the maximum heat intensity if the secondary oxidation used a programmed oven. Results showed that both aforementioned generating rates increased but the generating rate of CO<sub>2</sub> decreased [13].

In addition, scholars in China also undertake studies on the secondary oxidation control of residual coal. Fire prevention measures, including the positive-pressure ventilation in working face, grouting at the upper layer of goaf, borehole gel injection behind chocks and nitrogen injection, have been innovated [14]. A systematic study on the microscope features of coal in the secondary combustion is yet scarce. This study examined the microstructure of coal sample with different ranks selected from typical mines in China using scanning electron microscope (SEM) and infrared spectrum analyzer to reveal variations of porosity and molecular composition of the coal sample in secondary oxidation. The present work provides a fundamental basis for prevention of coal secondary oxidation.

## 2. Experimental

### 2.1. Experimental observation on coal microstructure before and after initial oxidation

Coal spontaneous combustion is caused by a variety of internal and external factors and is a result of several complex physical and

\* Corresponding author at: School of Safety Engineering, China University of Mining & Technology, Xuzhou 221116, China. Tel.: +86 15998350371.

E-mail address: [tfc5@163.com](mailto:tfc5@163.com) (F. Tian).

chemical processes. From a view point of microstructure, the structure of organic molecular, activation energy and reaction heat are important factors for coal oxidation with low temperature. From chemical kinetics point view, coal is a large molecular body which consists of organic and inorganic components. Changes of the coal porosity and molecular structure are the main causes for the variation of kinetic parameters of the coal [15–17]. Investigation on the changes of pore structure and molecular structure is therefore of importance to reveal the mechanism of secondary coal oxidation.

### 2.1.1. Experimental equipment and procedures

The coal samples with different metamorphic degrees were selected from eight coal mines in China. They are lignite from Dayan Coal Mine in Mongolia autonomous region, gas coal from Fushun Coal Mine in Liaoning province, jet coal samples from Wudong Coal Mine in Sinkiang autonomous region, fat coal from Pingdingshan Coal Mine in Henan province, coking coal from Linfen Coal Mine in Shanxi province, lean coal from Qingxu Coal Mine in Shanxi province, meagre coal from Tianfu Coal Mine in Sichuan province and anthracite coal from Jincheng Coal Mine in Shanxi province. According to national standard of GB/T 482–2008 as stating in *Coal samples taken method from coal seam*, sampling was undertaken at the upper corner near the goaf, sealed in black bags, labeled appropriately and sent to laboratory within 24 h.

In order to observe the changes of coal pore caused by oxidation, KYKY-2800B type SEM (Fig. 1) and a high temperature test oven (Fig. 2) from Beijing KYKY Technology Co., LTD were used.

All coal samples were divided into several groups evenly. After simple preparation, one group was sprayed for 5 min in the ion sputtering apparatus. These coated coal samples were used to obtain microstructure images by KYKY-2800B type SEM. Coal samples of another group were sprayed in the ion sputtering apparatus and tested at the high-temperature experimental table which consisted of DH1720A-6 type, DC voltage current regulator, CKW type temperature controller and the work station (Fig. 2). The coal samples were heated to 240 °C, and the changes of microstructure before and after the initial oxidation were optically observed by SEM.

### 2.1.2. Experimental results

Eight microscopic images of original coal samples and eight images after oxidation were obtained via SEM, as shown in Figs. 3–10.

### 2.2. Infrared spectrum analysis of the coal samples before and after initial oxidation

Coal is an organic structure, and contains a variety of condensed aromatic ring structure unit. Coal is also a polymer network structure connected by methyl ethyl, ethenyl and ether bond, etc. The number, types and spatial distribution of active groups in coal molecule determine oxygen adsorption and reaction heat, and



Fig. 1. KYKY-2800B scanning electron microscope.



Fig. 2. High temperature test rig.

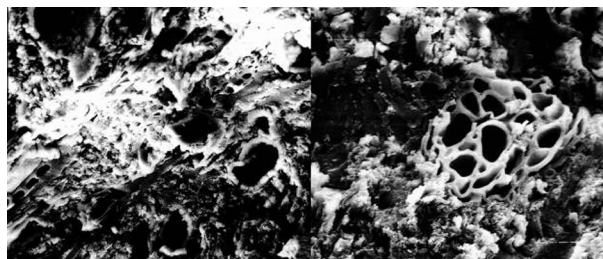


Fig. 3. Pore structure of lignite before and after oxidation.

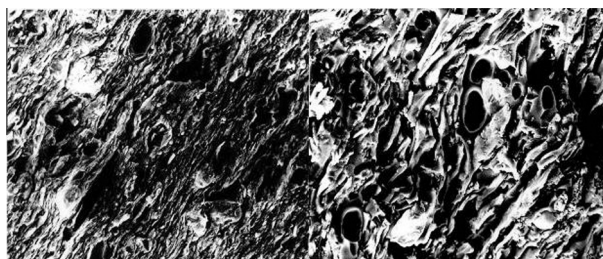


Fig. 4. Pore structure of gas coal before and after oxidation.

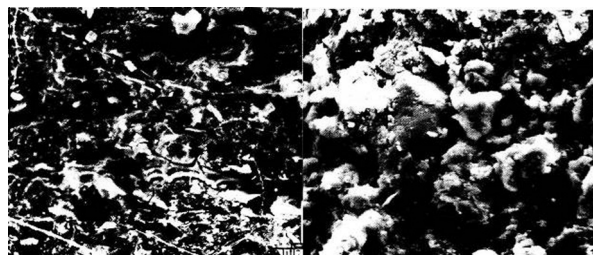


Fig. 5. Pore structure of candle coal before and after oxidation.



Fig. 6. Pore structure of metabituminous before and after oxidation.

those factors in turn determine the propensity of coal to spontaneous combustion. The functional groups with strong oxidation activity can be measured quantitatively or semi-quantitatively by

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