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Full Length Article

Estimating coke fracture toughness using acoustic emissions and changes in coefficient of friction during scratch testing

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ABSTRACT

Acoustic emission profiles generated during scratch testing of a range of metallurgical coke samples were recorded and linked to the concurrent energy release, dispersal or absorption on coke fracture or damage. Three different signatures were identified, which were based on the simultaneous measurement of acoustic and total energy release profiles, and these signatures could be correlated with both the microstructure and microtexture of the coke being traversed at the time. The acoustic emission signature for fracture or damage to the coke reactive maceral derived constituents (RMDC) was correlated to the rank of the parent coal or coal blend, with the signature number generally increasing with increasing rank. Conversely, the signature numbers did not vary with parent coal rank for fracture or damage to the inertinite maceral derived constituents (IMDC), with the majority of IMDC fractures associated with a release of mechanical energy. The incidence of the signature associated with a release of mechanical energy (type 1) became increasingly dominant from RMDC to RMDC-IMDC interfaces to IMDC. Conversely, signature types associated with a dispersal (type 2) or absorption (type 3) of mechanical energy become increasingly dominant from IMDC to RMDC-IMDC interfaces to RMDC. The findings suggest acoustic emissions recorded during scratch testing and their subsequent characterisation can be used to indicate the fracture toughness of a given coke. This study contributes towards a broader program of research to improve understanding of the factors which influence the strength of coke and its microtextural constituents and interfaces, and how this relates to the properties of the parent coals.

1. Introduction

The strength of metallurgical coke is difficult to predict, due to its heterogeneous nature. How a particular coke fractures under stress depends not only on its composition, but also its microstructure [1–4] and the properties and interactions at the interfaces [5–10] between the different forms of carbon in the coke. These forms of carbon can broadly be divided into two microtextural components. These are the reactive maceral derived components (RMDC) and the inertinite maceral derived components (IMDC). The RMDC are formed from organic coal macerals that fuse and are the major contributor to the plastic phase during cokemaking. IMDC are formed from non-fusible and semi-fusible coal macerals that are mostly structurally unaffected during cokemaking. The mechanical properties of these individual coke constituents, such as their fracture toughness and hardness, are very different and thus the amounts of these components in the coke and the nature of the interfaces between them have a major impact on coke strength and its mechanisms of degradation [5,11–13].

Recently, the study of the wear behaviour of a material, known as tribology, was carried out, to elucidate the mechanisms by which pilot oven coke samples [14,15] and blast furnace coke samples [16] are worn or damaged at room temperature. Whilst tribological testing is well-known for assessing the interfacial properties of a wide range of materials [17,18], our earlier research was the first example of applying tribological testing to metallurgical coke samples to determine the nature and strength of the interfaces between their different microtextural components [14,16]. However, the factors that influence the nature and strength of these interfaces remain incompletely understood.

In this paper, we report the results of progressive loading linear scratch tests [19,20] on a suite of metallurgical coke samples formed from coals with different properties. The aim of this research work was

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to develop an acoustic-based approach to measure the mechanical energy release during coke scratch tests, and relate this energy release to the specific wear mechanisms, e.g. the release of an IMDC due to tribological wear processes.

Broadly speaking, three types of failure can occur during scratch testing of coke; elastic wear, plastic wear and fracture. We linked the acoustic emissions to the specific mechanism of damage (or main mechanism, where appropriate), the microtexture or microtextural interface, and the change in coefficient of friction, at the acoustic emission maxima. This led to the identification of distinct acoustic emission "signatures", each of which is linked to the energy release, dispersal or absorption that occurs when damage to the coke takes place during loading. The objective was to then use the acoustic emissions recorded during scratch testing as a rapid and simple comparison of the *relative* fracture toughness of distinct microtextural constituents and their interfaces as a function of key properties of the parent coals, such as rank.

2. Experimental

2.1. Coal and coke selection

A total of sixteen coke samples were examined. These were:

1) Pilot oven cokes, the majority of which were formed from single coals, covering a range of rank, petrographic composition and Australian coal measures. The selection also included a coke produced from a 50:50 binary blend of two of the single coals, and a coke from a non-Australian coal.
2) Cokes from controlled maceral mixes which were formed on a small scale using a sole-heated oven at the University of Newcastle (UoN).

The conditions for formation of the pilot oven and sole-heated oven cokes can be found in Refs. [21–23], respectively. Whilst the acoustic emission signatures were developed using the database of results for all sixteen cokes, this paper presents examples for seven of the pilot oven cokes, six of which were prepared from Australian single coals plus one from a binary Australian coal blend. It is important to note that these cokes are not representative of commercially traded products. Key parent coal properties and coke strength indices are shown in Table 1.

N.B. An investigation of the effect of coke preparation conditions on results will be the subject of a subsequent paper [23]. To summarise, the differences between cokes as a function of preparation conditions were found to be statistically insignificant.

2.2. Sample preparation

Coke samples were mounted in an epoxy resin mixture comprising hardener and a red pigment in plastic containers. The set samples were then cut into 10–20 mm thick slices using a LECO drop down saw. Slices with maximum coke coverage were cored using a bench drill with a 40 mm diamond coring bit attached. The cores were ground down to fit inside 40 mm diameter plastic containers and remounted in resin. These remounted samples were then smeared with resin to ensure the resin filled all capillaries and pores. Using a PICO 155 Precision Saw the samples were cut to a 10 mm thickness whilst placed in a 40 mm holder to ensure the upper and lower surfaces were parallel.

A Struers TegraSystem Autopolisher was used to polish the coke samples to a < 3 μm finish. Three polished blocks of each coke were prepared, ensuring where possible that each repeat was from a different coke lump to improve representation. Where possible, one polished block from each coke was sent to Pearson Coal Petrography Inc. (Victoria, BC, Canada) for petrographic imaging and microtextural composition analysis [16,24,25]. A photograph of a typical sample prior to testing is shown in Fig. 1a.

Table 1
Key coal properties and coke strength indices. N.B. Coke strength indices were not measured for cokes LRHV, LRHV 50:50 HRLV and HRLV.

Coal/Coke	Vitrinite Content (incl minerals basis, %)	Mean Max Vitrinite Reflectance (%)	ASTM Stability	ASTM Hardness
LRHV	78.5	0.85		
LRHV 50:50 HRLV	65.8	1.12		
MRMV	60.3	1.17	64.2	69.5
MRLV	44.3	1.23	59.2	66.0
HRLV	53.0	1.39		
HRLV2	48.5	1.46	62.2	65.9
HRMV	66.0	1.51	66.6	68.7

2.3. Scratch testing

Progressive loading linear scratch tests were performed on the polished coke blocks mounted in epoxy resin using a Revetest Xpress Plus Scratch Tester (RSX) with a spheroconical (400 μm diameter) Rockwell diamond indenter. A photograph of the test setup is shown in Fig. 1b. The instrument was equipped with a 150 kHz acoustic emission sensor. Since this sensor was unable to discriminate between different acoustic frequencies, only the 150 kHz emission was recorded. A loading range of 10–100 N over a scratch length of 5 mm and loading rate of 90 N/min were determined to be the optimal testing conditions based on the properties of the indenter and its profile, and the wear behaviour of a range of coke samples under rotational tribological testing [26,27]. Progressive loading was chosen over constant loading as it allowed collection of data over a 10–100 N range, and thus a better understanding of the wear behaviour of different cokes and their microtextural constituents under increasing stresses.

A minimum of six scratch tests were performed for each sample of each coke, with the acoustic emission and frictional force data from each test captured by a data logger. The change in frictional force as a function of the loading force, i.e. the change in the coefficient of friction (COF), gave an indication of the energy absorbed, dispersed or emitted by the deformation, fracturing or other mechanism of damage to the coke sample during testing.

Following testing, samples were analysed using a combination of different analytical and imaging techniques. These included high resolution optical microscopy, 3D laser scanning microscopy and scanning electron microscopy.

2.4. High resolution optical microscopy

High resolution optical micrographs were recorded both before and after linear scratch tests using a Zeiss Axio Imager.Z1m microscope equipped with an automatic stage and both high and medium resolution cameras used to capture images using AxioVision software. Individual images (recorded at 5× magnification) were stitched together to capture a high resolution image of the entire sample using the AxioVision Mosaic software.

2.5. 3D laser scanning microscopy

A Keyence 3D laser scanning microscope (VK-X100 Series) with an automatic stage was used for 3D imaging (at 200× magnification) following scratch testing. Individual images were stitched together to form a mosaicked image of each scratch. Optical, laser and height profile images were recorded simultaneously. Analysis of the 3D images and profiles was carried out using VK Analyser software.

2.6. Scanning electron microscopy

Samples were carbon coated using an SPI carbon coating unit and mounted onto aluminium stubs using a carbon tab. Conductive