Fuel Processing Technology 155 (2017) 183–190

Contents lists available at ScienceDirect

Fuel Processing Technology

journal homepage: www.elsevier.com/locate/fuproc

Research article

Examining mechanisms of metallurgical coke fracture using micro-CT imaging and analysis

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ARTICLE INFO

Article history:
Received 15 January 2016
Received in revised form 25 May 2016
Accepted 29 May 2016
Available online 6 June 2016

Keywords:
Coke strength
Finite element analysis
Fracture
Fractography
Micro-CT

ABSTRACT

Micro-CT imaging of samples of metallurgical coke was achieved at high resolution (~10 μm), using the Imaging and Medical Beamline of the Australian Synchrotron. The coke samples were then stressed to breaking point and imaged again. The result is that we can examine, in fine detail in 3D, the locations of the fracture surfaces in the coke, and their relationship to the different aspects of the microstructure. In addition, we have performed 3D finite element linear elasticity analysis, using the microstructure of the cokes as input to the calculations. The result is a 3D map of the von Mises stress in the structure, which provides information about the likely locations of fracture. Finally, we have performed a fractographic analysis of the fracture surfaces, in order to characterise the nature of the fracture mechanisms. The imaging and analysis has been performed for both compressive and mixed-mode loading of the coke samples. The work provides the opportunity to identify the key mechanisms for fracturing of coke due to the aspects of their microstructure, and has the potential to aid in the production of stronger metallurgical coke, by means of blending coals to produce coke with improved strength properties.

1. Introduction

Strong metallurgical coke is critical for good performance of modern blast furnaces. As part of a program of investigation of the factors that affect the quality of coke, we have been investigating the link between the microstructure of coke and its mechanical properties. Coke is a porous composite material, consisting of (highly-connected) pores, inert particles, mineral matter and reactive maceral derived material, having micron sized dimensions. X-ray microtomography (micro-CT) imaging is therefore an ideal mechanism to study the microstructure of coke in full 3D.

Micro-CT is a technique similar to X-ray CT scans performed in medical fields, but with considerably higher resolution [1] and a pixel size on the order of microns. Internal reconstructions of structures can be achieved, allowing acquisition of the fine detail of the microstructure of various cokes. These reconstructions comprise a series of 2D cross sections which can be combined into a full 3D representation of the material structure. The technique has the advantage of being non-destructive, and has been used successfully in a wide variety of applications, ranging from biology [2,3] to materials science [4,5]. In particular it can elucidate detailed information on the 3D structure of porous [6] and/or composite materials [7], which would be challenging to fully characterise using more conventional laboratory-based methods of analysis.

However, the current state of the art presents a challenge in terms of interpretation of the large quantities of data produced by the technique, with respect to obtaining measures of coke quality. Previous studies [8–11] have performed micro-CT analysis, combined with finite element analysis, in order to relate the microstructure properties to mechanical strength. Here, we have extended these approaches by (a) considering a much wider range of coke samples and (b) examining in more detail the nature of the mechanisms associated with breakage of coke samples. In particular, we use image registration to compare imaged coke microstructures before and after cracking, then relate the location of the crack surfaces to high stress points identified by finite element analysis.

Finally, we apply fractographic analysis to identify the particular breakage mechanisms occurring during failure of the coke. Fractography is a technique used to investigate the breakage behaviour of a material by decoding the fracture surface topography and patterns of crack propagation, in order to locate the origin of failure as well as the regions of the microstructure at which stress is concentrated. It is a

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http://dx.doi.org/10.1016/j.fuproc.2016.05.039
0378-3820/

common method of identifying the modes of failure in metals and ceramics, and we have successfully applied the technique to identify and quantify the fracture mechanisms and microstructural features of high stress concentration in metallurgical cokes of varying properties [12–15].

In this study, we used both compression and "mixed mode" loading to examine the fracture behaviour of the different cokes. A mixed mode test is so named as it results in fracture of a sample due to a combination of compression, tensile and shear failure modes. Mixed mode tests in this study were performed as for the diametral compression test [16] in which a disc of the specimen is placed between two flat platens which apply a compressive load to the specimen. This typically results in fracture from the centre of the disc in the direction of the applied compressive load due to the development of tensile stresses perpendicular to this applied load.

In our earlier studies, a shearing [17] or compressive [18] load was applied to samples of pilot oven scale cokes formed from coals of varying properties. Using the micro-CT images as input, we conducted 3D finite element analysis in order to determine the von Mises stress distribution within the structure, caused by the applied load. In the first case, the boundary conditions used in the finite element calculation considered the application of a shearing force to one face of a cubic section of the rendered coke microstructure obtained by micro-CT imaging, to better understand the microstructural features contributing to coke abrasive strength. In the latter study, micro-CT images were recorded before and after applying a compressive load to the coke, to examine the differences between the unstressed and stressed states, and in particular to locate regions of internal damage for comparison with the stress analysis carried out. However, in this particular study, the loads applied were insufficient to cause any significant damage or fracture of the coke samples.

Under mixed mode loading, the maximum load needed to fracture a sample is generally less than under compressive loading, due to the combination of additional forces applied during the mixed mode test. The lower maximum load requirements allow fracture of the coke samples using a portable stress testing setup on location at the Australian Synchrotron. Moreover, in strength testing of metallurgical coke, it is highly applicable to test fracture behaviour under mixed mode in addition to the standard compression test, since in the blast furnace, coke lumps are subjected to varying loads and stresses, which include compressive, tensile and shear stresses.

2. Materials and methods

2.1. Coal selection

The properties of the coals used to form the cokes in this initial study are summarised in Table 1. These coals were selected because of their similar rank yet significantly different vitrinite content. Different coking behaviour and pore size distribution within the final coke microstructure were therefore expected. Coke strength indices are also shown in Table 1.

2.2. Sample preparation

The cokes were prepared in two pilot coke ovens using similar operating conditions [19] which simulated commercial coking conditions. The coal grind for both cokes was ~85% passing 3.35 mm.

Coke cores were prepared from lumps of coke using a 16.9 mm diameter core drill, and cut into discs of approximately 10 mm thickness using a diamond saw. Discs were gently ground flat using sandpaper and wrapped in transparent tape to avoid edge dislocations of the coke during the compression tests. For compression tests, samples were glued onto aluminium sample mounts, designed to fit tightly into the head of the goniometer on the beamline. For the mixed mode tests, small flats were added to the sample circumference at the points at which the platens come into contact with the sample in compression tests. These 'flats' reduce the effects of friction as the load contacts the sample and helps the fracture to occur in the direction of the applied compressive load due to the development of tensile stresses perpendicular to the applied load. The mixed-mode test samples were attached to the aluminium mounts using adhesive putty. Fig. 1 shows discs in the two different orientations.

2.3. Micro-CT imaging

Samples were imaged at the Imaging and Medical Beamline of the Australian Synchrotron, adapting a method we used in a previous study [18]. The zooming 'Ruby' x-ray imaging detector was used, set to 8.87 μm per pixel. Data collection was carried out at an x-ray energy of approximately 30 keV with 1800 images acquired as the sample was rotated over 180 degrees. In addition to images of the sample, images of the illumination without the sample and of the CCD signal in the absence of x-rays were also acquired. These were used to correct for image illumination and CCD artefacts. The image data was treated with a phase-retrieval algorithm in order to make best use of the phase contrast and improve the quality of the final images by enhancing the visibility of the edges, boundaries and small features. This and other image processing together with tomographic reconstruction steps were carried out using CSIRO's X-TRACT software running on the MASSIVE cluster. A series of vertical scans was required in order to cover the whole sample. Once these images were stitched together, the resulting image stacks consisted of the order of 2000^3 voxels of greyscale data for each sample.

2.4. Coke fractography

After the samples were imaged, they were fractured then imaged again. Fracture tests were performed using a 15 tonne pellet press and a custom-made portable stress testing device for the samples adhered to aluminium sample mounts (see Fig. 2). A mini disc load cell with a maximum capacity of 1000 kg (LPX-1000) connected to a data logger (dataTaker DT80 model) was placed under the stress testing device, to allow for capture of the maximum load required to initiate fracture of the samples. The acoustics of the fractures were also recorded for each experiment using a microphone and Audacity software, to detect the propagation of the fracture and identify the type of fracture.

3. Results and discussion

3.1. Coke microstructure and pore size distribution prior to fracture

For illustration of the different nature of the cokes, Fig. 3a shows a single slice micro-CT image of a C147 coke sample, and Fig. 3b a single slice through a C155 sample.

Table 1
Basic properties of the coals used to form the pilot oven cokes studied, and the measured M_{40} and M_{10} tumble drum indices of the cokes.

Coke	Coal properties		Source	Coke properties	
	Vitrinite content (%, incl minerals basis)	Mean maximum vitrinite reflectance (%)		M_{40} index	M_{10} index
C147	83	1.23	Non-Australian	81.2	6.6
C155	61	1.22	Australian	79.4	5.8