Fuel 226 (2018) 163–171

Contents lists available at ScienceDirect

Fuel

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

Full Length Article

Uniaxial compression of metallurgical coke samples with progressive loading

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ARTICLE INFO

Keywords:
Compressive loading
Metallurgical coke
Micro-CT

ABSTRACT

Coke samples, produced from coals having a range of rank and vitrinite content, were subjected to uniaxial loading in a universal tester. Cokes were also imaged at high resolution using micro-CT. The aim was to understand the relationship between the internal microstructure of the coke and coke strength under load. The loading was done in two separate ways, being either compression to failure or progressive loading. The results showed that the coke samples underwent a form of stiffening at low loads, potentially due to closing of fine-scale pores and/or re-alignment of graphitic layers in the RMDC. Measurements of plastic strain indicated that these changes were permanent. At higher loads, small load-bearing components of the microstructure were found to break, leading to a softening of the coke samples. Evidence of the breaking was observed using micro-CT images before and after loading of samples. The work has relevance to the understanding of the fundamentals of coke strength, as well as to issues relating to handling and preparation of coke for standard testing.

1. Introduction

A key attribute of metallurgical coke is its strength – both cold strength, which is required for handling before input into the blast furnace, as well as load bearing at the top of a blast furnace, and its hot strength, at lower regions in the blast furnace where it may have been degraded by reaction as well as mechanical factors. In practice, cold strength of metallurgical coke is principally measured by a range of standardised drum tests (ASTM, IRSID, JIS) [1] resulting in indices that are believed to reflect the effect of abrasion and bulk breakage upon the coke sample during tumbling in the drums. In a sense, these tests are *indirect*, in that they report coke size fractions arising from the degradation from the tumbling in the drum, but it is somewhat difficult to determine the mechanisms that lead to the resulting indices.

Metallurgical coke can be thought of as a porous composite material. It typically has up to 50% porosity, almost all of which is connected [2], and the solid phase consists of reactive maceral derived component (RMDC), inert maceral derived component (IMDC) and mineral matter [3]. The size, shapes and distributions of these different components within the complicated microstructure of the coke are a result of both the properties of the original coal as well as the physical and chemical transformation undergone during carbonisation [4]. Together, these components provide the building blocks of the microstructure and the factors associated with the above will determine the material properties of the composite. As a result of the complications of the structure due to its formation process, it is extremely difficult to predict *a priori* the mechanical properties of the resultant coke from its input coal (blend).

The more usual criteria for failure of a material are based on the tensile strength value, which is a material property that is difficult to determine for metallurgical coke, ceramics and similar composites [5,6]. Traditionally, three or four point bending tests have been used to measure a strength value which has not strictly determined the tensile strength of the material because it has not been subjected to a uniaxial loading condition during the bending test [5,7].

It is possible to obtain *direct* measures of the material properties of metallurgical coke, at least for individual samples of coke, using standard testing methods. A particular approach to determining the basic mechanical properties is the uniaxial compressive strength test [5]. In such a test, the sample is compressed with continually increasing strain and the associated load (stress) is measured, in order to obtain a stress-strain curve for the material. This process can be continued even after the sample yields. Of course, for an elastic material, the stress-strain curve is linear, with the slope representing the stiffness, or Young's modulus. However, for many real materials, particularly composites, the stress-strain curve will deviate from linearity in a range of ways, and it may be possible to determine properties of the structure from these deviations.

The idea of quantifying the damage induced by compression testing and its effect on the mechanical properties of a material has developed into its own field of study called damage mechanics [8]. The theory of

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https://doi.org/10.1016/j.fuel.2018.03.173
Received 26 October 2017; Received in revised form 22 March 2018; Accepted 27 March 2018

damage mechanics describes the change in material behaviour between the initial state and the fracture induced or failed state [8]. The concept of continuous measure of damage has been used to describe various types of failure in metals and other similar solids and these damage models are reviewed by Lemaitre and Chaboche [9]. In general damage based relationships can be applied to problems in which brittle fracturing has a large influence on the behaviour of the material [9,10], with metallurgical coke a prime example of this.

Previous studies by other researchers have applied the uniaxial compressive strength test to investigate the behaviour of coke under compressive loading. Amanat et al. [11] observed that coke shows linear elastic behaviour under compressive loading, which is followed by "brittle layered crushing" and "densification". This layered crushing was also observed in a finite element modelling study conducted by Tsafnat et al. [12], which used 3-D micro-CT images of coke before its compressive loading as the input to the linear elasticity model. Other studies [13–15] have focussed more on modelling the *diametral* compression test to investigate coke fracture behaviour and its deformation/damage under loading.

In this paper we describe uniaxial compression tests performed on a range of coke samples. These comprise tests where the sample is continuously strained until it yields, as well as so-called progressive loading tests where samples are repeatedly loaded, unloaded then loaded again to a higher strain, multiple times. The work is part of a long-term approach to understanding the mechanical strength of coke, its relationship to its microstructure and properties of the original coal (blend).

2. Experimental

2.1. Coal selection and coke preparation

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 variation in rank and vitrinite content. Different coking behaviour and pore size distribution within the final coke microstructure were therefore expected; we hypothesised that this would have an impact on the strength and effective Young's modulus of the coke under compressive loading.

The cokes were prepared in pilot coke ovens using operating conditions [16] which simulated commercial coking conditions. The coal grind for all cokes was ~85% passing 3.35 mm. Coke cold strength (tumble drum) indices are also shown in Table 1.

2.2. Sample preparation

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.

2.3. Compressive strength tests

Uniaxial compressive loading of cokes was conducted using a Shimadzu universal tester (Shimadzu Autograph AGS-10kND), pictured in Fig. 1. A minimum of three tests were conducted for each type of coke, using discs obtained from cores of at least two different coke lumps. Unless otherwise stated, a crosshead displacement rate of 1 mm/min was used. Load versus displacement graphs were recorded during compressive loading of the samples until failure. The maximum compressive strength was calculated from the maximum load at failure and the cross-sectional area of the sample.

2.4. Progressive loading tests

Progressive loading tests were conducted *via* the progressive application of a uniaxial compressive load to the samples using a Shimadzu universal tester, shown in Fig. 1. Samples were first loaded to 5% of the mean maximum compressive load at failure for that coke using a crosshead displacement rate of 1 mm/min. When 5% of the mean compressive load was reached, the crosshead was removed, i.e. the sample was unloaded. This cycle was repeated but the second time the sample was loaded to 10% of the mean maximum compressive load, followed by 15% and 20%. Next, the load was increased for each progressive loading cycle by 10% until 80% of the mean maximum compressive load was reached. The maximum load during each cycle was then increased in 5% increments, until failure of the sample occurred and the test was thus ceased. Load versus displacement graphs were recorded for each loading cycle. The load and displacement readings were converted to stress and strain measurements, respectively. The stress was calculated from the load using the sample cross-sectional area. The effective Young's modulus was calculated from the slope of the linear region of a stress versus strain graph.

2.5. Micro-CT imaging

Samples were imaged at the Imaging and Medical Beamline of the Australian Synchrotron, part of ANSTO, adapting a method we used in a previous study [17]. 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°. 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

Table 1
Basic properties of the coals used to form the pilot oven cokes studied, and measured coke properties and strength indices. N.B. ASTM stability and hardness indices were not measured for C135.

Coal/ Coke	Coal Properties		Coke Properties and Strength Indices							
	Vitrinite content (incl minerals basis, %)	Mean max vitrinite reflectance (%)	ASTM stability	ASTM hardness	CSR	Mean pore size (μm)	Std dev pore size (μm)	Mean porosity (fraction)	Mean max compressive strength (MPa)	Std dev max compressive strength (MPa)
C136	46.5	1.14	59.5	65.2	47.3	127	91	0.403	16.0	5.3
C144	50.3	1.22	55.1	65.1	70.3	164	134	0.371	19.2	5.7
C155	60.7	1.22	61.9	68.1	69.3	156	123	0.405	23.0	6.7
C134	70.8	1.38	63.8	67.6	71.1	164	116	0.516	22.2	4.8
C135	76.8	0.90			54.3	259	230	0.532	20.7	3.3