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Fractographic approach to metallurgical coke failure analysis. Part 1: Cokes of single coal origin

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HIGHLIGHTS

- Failure profiles for an individual metallurgical coke were constructed.
- Microstructural features in coke fracture surfaces were identified.
- Macro and micro failure mechanisms in the coke were quantified.

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ABSTRACT

Metallurgical coke is a complex brittle heterogeneous material consisting of carbon derived from fusible, semi-fusible and inert coal particles that forms a porous composite matrix. This paper presents a novel approach to assess and quantify the breakage behaviour and microstructural weaknesses in a pilot oven metallurgical coke. The approach uses fractography, a method commonly applied to determine the fracture behaviour and origin(s) in homogeneous materials, such as metals and ceramics. Determination of the fracture origin(s), paths of crack propagation and microstructural weaknesses in such a complex heterogeneous material as metallurgical coke represents a significant advance in both the application of fractography and the assessment of coke strength and breakage behaviour. Identification of the key features that contribute to the coke's failure will facilitate better prediction of coke strength from coal properties and ultimately optimisation of the coal blending process.

Key features and markings have been clearly identified on fracture surfaces that can either be traced back to the fracture origin or give an indication of the type of fracture or stresses to which the coke has been subjected, including the directionality and strength of those stresses. These markings include hackle, hackle twist and wallner lines, as well as markings generated by conchoidal and overload fractures.

A three-step approach was applied to determine the breakage behaviour in stabilized lumps of the pilot oven coke, in which fractured coke surfaces were analysed at the macro, micro and submicron levels. The observed mechanisms of failure were quantified and summarised using a radar diagram.

1. Introduction

Metallurgical coke is one of the key raw materials used in the blast furnace to reduce the ferrous burden (e.g., iron ore, sinter and pellets) to iron, which is the precursor step in the production of steel. In the blast furnace, metallurgical coke plays several essential roles, the most important of which are maintaining the permeability of the blast furnace and supporting the burden.

Typically the feed coke used in the blast furnace is not produced from one single coal but rather a blend of different coals. In order to predict the strength of a coke the coal industry uses models, which are largely empirical in nature. These models are based on the current standards that the coal and steel industry uses to assess coal and metallurgical coke strength, such as the coke strength after reaction with carbon dioxide (CSR) test. In some cases, these models fail to accurately predict coke behaviour/strength, particularly when coal blends are used. Fundamental research towards better prediction of coke behaviour/strength from its constituent coal/coal blend properties and coking behaviour is essential to

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improving the reliability of coke strength predictions and the models on which these predictions are based. To facilitate these improvements a more fundamental understanding of why coke fails is needed. We use a fractographic approach (see Section 1.3) to understand the sources of strength and weakness in coke.

1.1. Coke heterogeneity

One of the major reasons why the behaviour of metallurgical coke can be difficult to predict lies in its heterogeneity. Coke is a distinctive composite material comprising carbon forms derived from fusible, semi-fusible and non-fusible coal macerals as well as inorganic components. The reactive maceral derived component (RMDC) of the coke is the solidified product of organic material that fused during coke formation and acts as a binder. Inert maceral derived components (IMDC) arise from non-fusible and semi-fusible coal macerals that remain largely structurally unaltered during coke making. How a particular coke fractures under certain loads or stresses is therefore dependent not only on its composition, but also the properties and interactions at the interfaces between the various maceral derived components, known as 'textures'. Coke processing and the mechanical properties of its constituents e.g. brittleness/ductility, will also have a major impact on its ultimate strength and predominant mechanisms of failure.

1.2. Coke mechanical strength

To date, methods explored in the literature to estimate the mechanical strength of metallurgical coke include both hot strength (CSR) [1] and cold strength (tumble drum indices [2] and shatter tests [3]) techniques. These techniques use large numbers of coke lumps and so give a good average "strength" value for the coke. However, these techniques don't address the question of why the coke failed at the locations that it did. Knowing the mechanism of coke failure and the reasons it fractured where it did aids in the building of more fundamental models for predicting coke strength rather than relying on statistical comparisons of strength numbers with coal properties.

Early research, using alternative techniques, conducted by Patrick and Stacey investigated the tensile strength of coke as a means of measuring its mechanical strength [4]. This work was followed up with an investigation into the relation of coal properties and the resultant coke microstructure to coke tensile strength [5]; however, in the blast furnace the cokes are subjected to a number of additional and different forces, including compressive, shear and tensile.

Current techniques to assess the mechanical properties of the different textural components of coke rely, for example, on a nano-indentation type approach [6–8] which while excellent for establishing the properties at that precise point, does not allow for the heterogeneous nature of the coke itself and any interactions with mineral matter or inert material. It is also not capable of evaluating the grain boundary conditions, the degree of wetting (i.e. interaction) between any micro textural constituents or the stresses involved. Research work by Kubota et al. has demonstrated that additional factors have a significant impact on coke strength and its breakage behaviour, including the influence of inertinite size [9], pore structure [10], and pore roundness [11].

1.3. Fractography for coke failure analysis

This paper presents a novel approach to study the mechanisms by which coke fails, as well as acquire an enhanced understanding of the nature of and reasons behind the failure. Our approach uses fractography, a technique which has been used on a wide variety of materials including metals, polymers and ceramics [12–14] to determine both the microstructure of the material and the mechanism by which it fails. Following failure of a sample, the fractured surfaces are analysed to identify both the origin(s) and cause(s) of the failure. In the case of metallurgical coke, the structural and/or textural feature or boundary between textural components is identified as the source of stress concentration i.e. 'weak point' in the structure. These stress concentrators can result in failure of a coke at a considerably lower load than its predicted or theoretical maximum strength. Upon identification of the specific textural components and/or grain boundary (i.e. the interface between IMDC and RMDC) characteristics that act as stress concentrators in a coke of particular rank and type, steps could then be taken to remove or reduce these stress concentrators through linkage of these features to the properties of the parent coal(s).

Previous studies to assess coke strength and its breakage behaviour include a surface analysis based approach to assess coke breakage in response to tensile loading [15] and use of polarized light microscopy to classify and quantify the quality of interfaces present between the textural components in polished coke surfaces [16]. Later research by Andriopoulos et al. [2] indicated that the boundaries between different textural components are one of predominant regions at which stress concentration develops in metallurgical coke, if the interaction between these components is weak. Kanai et al. [17] advanced this concept by quantifying the area at which the interface between the RMDC and IMDC was deduced to be weak following visual examination of SEM micrographs, referring to these regions as 'non-adhesion grain boundaries'. Moreover, this quantification was undertaken using the fractured rather than polished surfaces, and was an improved technique on the previous work since it is more difficult to identify (micro) cracks at the grain boundaries and distinguish these from pores in polished surfaces [17]. Kanai et al. distinguished between cracks that occur around grains i.e. between the different textural components, known as intergranular cracking, and those that occur through grains/textures, known as transgranular cracking [17]. The main limitations of these earlier studies include: the breakage of the coke samples by diametral compression techniques which predisposed them to tensile failure mechanisms due to the orientation and loading of the testing rig, with respect to the coke sample; the use of small-sized (10 mm diameter and maximum 10 mm length) cylinders rather than whole stabilised coke lumps; and the lack of identification of the characteristic features responsible for the weak interaction between textural components.

In the fractographic approach presented in this paper, a three-step approach is applied to determine the breakage behaviour in stabilised lumps of a pilot oven coke, in which fractured coke surfaces were analysed at the macro, micro and submicron levels, as detailed in Section 2. The observed mechanisms of failure were quantified and summarised using a radar diagram, facilitating rapid, visual comparison between the dominant mechanisms contributing to the failure of cokes of different coal origin. To highlight this, comparison with a coke of different single coal origin has been included in this study.

2. Experimental

2.1. Metallurgical coal selection and coke preparation

A pilot oven coke from a low volatile coking coal was used in the initial study presented here, and will be referred to as 'coke A' in this paper. The coking conditions are summarised in Table 1 [18]. To identify the differences in coke breakage behaviour as a function of the original coal properties, the results were compared with a coke of different single coal origin, referred to as 'coke B'. The basic properties of the constituent coals A and B are summarised in Table 2.