



Influence of thermoplastic properties on coking pressure generation: Part 3 – Evidence and role of pore coalescence in the mechanism for pressure generation

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HIGHLIGHTS

- ▶ Pore behaviour during coal carbonisation has been tracked using rheometry.
- ▶ Plate gap and axial force accurately track pore nucleation, growth and coalescence.
- ▶ Open porosity increases when plate gap decreases and/or axial force decreases.
- ▶ Results reinforce hypothesis that pore coalescence reduces oven wall pressure.

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ABSTRACT

This work follows on from two papers which presented a hypothesis for the mechanism behind oven wall pressure. It was proposed that oven wall pressure results when the period of pore growth is lengthy prior to coalescence and/or the period of coalescence is minor ahead of resolidification. When this occurs a thick swollen band of closed pores in the viscoelastic medium results which exerts pressure on the oven walls. The band is a resistance to volatile transfer and the largely closed pore network remains throughout resolidification further exacerbating pressure on oven walls. This hypothesis has resulted from a mapping of the viscoelastic properties obtained from a rheometer against the plate behaviour of the rheometer where pore nucleation and growth has been characterised by an increase in plate gap (ΔL) when the plate is held under constant axial force (AF) or an increase AF when the plates are held at constant gap. Pore coalescence has been characterised by a peak in AF or a decrease in the rate of expansion of ΔL . Because the behaviour of either ΔL or AF has brought about our understanding of coking pressure this paper attempts to prove that pore coalescence can be monitored from plate behaviour.

As such this paper attempts to track pore growth and coalescence using mercury porosimetry, permeability, scanning electron microscopy and optical microscopy, all of which have been carried out on quenched coke samples. Scanning electron microscopy images showed that the ΔL profile provides an accurate account of the onset of bubble nucleation, growth and coalescence. Porosity measurements indicate that the proportion of open pores in the 1–100 μm range increases significantly after the peak in AF or decrease in the rate of expansion of ΔL , indicating that these points coincide with a bubble coalescence phenomena. Optical microscopy showed a distinct difference in pore structure between the image corresponding to the peak in axial force and images after, whereby the number of isolated cells decreased and pore walls became thinner. Permeability measurements on quenched samples throughout the softening process show an initial drop in permeability due to the loss of interparticle voidage and the onset of swelling followed by a steady increase coinciding with the peak in axial force and decrease in the rate of ΔL expansion. This work has helped to reinforce the proposed mechanism for coking pressure. Furthermore, permeability measurements of quenched samples at 550 °C show that the high pressure coals tend to have the lower permeabilities at this temperature.

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The finding that pore growth and coalescence behaviour is what impacts on coking pressure has opened the door for finding novel ways to manipulate pore behaviour and therefore manipulate coking pressure. Understanding and manipulating pore growth behaviour has other implications, largely the understanding for coke strength development, which is currently being explored by the authors.

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1. Introduction

Resistance to the escape of evolved volatile matter during carbonisation in slot-type ovens is the main reason for high oven wall pressures. Consequently, both plastic layer and semi-coke permeability are critical in the coking pressure mechanism, since the former will control swelling propensity and pore structure network development while the latter will control transport of volatile matter towards the oven walls.

Recent work by the authors using high temperature rheometry has indicated that it is the propensity for pore coalescence which dictates whether oven wall pressure will be high or low. Rheometry allows the subtle differences in coal properties to be measured and is ideal for aiding the identification of coking mechanisms. It has been proposed that if the pore growth region is sustained for a considerable period of time ahead of pore coalescence and/or coalescence does not occur prior to resolidification, oven wall pressure can escalate [1–3]. In these studies pore swelling and coalescence have been followed in a high temperature rheometer through either change in parallel plate gap (ΔL) under constant applied plate force or variation in axial plate force (AF) with constant gap. These parameters are monitored while simultaneously exposing the sample to a small oscillatory shear strain to determine viscoelastic properties. The magnitude of ΔL and AF can be related to the extent of swelling, while pore coalescence is intimated from a peak and subsequent drop in magnitude of ΔL or AF.

This mechanism is supported by the observations of Mott and Spooner [4] who prepared cokes at constant volume and rapidly quenched them at the various stages of carbonisation. Analysis of the coke porosity revealed a large increase in the proportion of open pores with temperature. Hays et al. [5] used optical microscopy to study pore evolution of samples quenched in a single wall heated oven. Different extents of pore evolution co-existed due to the thermal gradient across the oven and analysis showed that pores appeared at temperatures close to the onset of plasticity and increased in size and number up to a critical temperature, above which the number of pores decreased. This was again attributed to pore coalescence.

Although permeability of the resultant cokes were not evaluated in either of these studies permeability studies have been performed by Casal et al. [6] who measured plastic layer permeability *in situ* under constant volume conditions and found an initial drop in permeability at the temperature corresponding to coal softening. Permeability was found to remain low for a considerable period of time before increasing again. They found that for the low coking pressure coals the low permeability plateau was shorter and ended before resolidification. For the high pressure coal the low permeability region exceeded to the resolidification temperature, suggesting that permeability remains low in the semi-coke. Similar findings were also reported by Geny and Duchene [7] who showed that the greatest drop in internal gas pressure occurred during the thermoplastic temperature range for low pressure coals, but this drop in internal pressure occurred at higher temperatures for high pressure coals. These findings are consistent with those of Zubkova [8,9] who found that low fluidity coals, which are more likely to generate high oven wall pressures, retain higher gas pressures both during and after resolidification. Volume contraction was also found to be lower when gas pressure was high.

No studies have linked plastic layer permeability to pore structure although researchers at Loughborough University [10] found that the internal gas pressure generated by coals in a small double wall oven gave reasonably good correlation with a range of coal/coke derived parameters including closed porosity. More recently Casal et al. [11] studied the structure of a recently resolidified semicoke and found that the apparent density and porosity of the semicokes produced from high pressure and low pressure coals differed with the high pressure coals found to have a lower apparent density and greater porosity.

The primary purpose for this study is to confirm that the ΔL or AF profiles obtained from rheometer tests are a reliable reflection of the pore growth behaviour and in particular pore coalescence leading to an open pore network. This confirmation will help to validate the coking pressure mechanism proposed by the authors in earlier publications [1–3] and enable the rheometry methods to be used to develop new tools and methods for predicting and controlling coking pressure. To achieve this, samples have been quenched at various stages along the ΔL or AF profile and analysed for permeability, porosity, density and also examined visually using scanning electron microscopy (SEM) as well as optical microscopy.

2. Experimental

2.1. Coal and coke sample information

Rank information and measured oven wall pressures (OWP's) for the coals used in this study are shown in Table 1. Coal C, AA1 and R24 are similar, being from different parts of the same mine. AA2 is an oxidised variant of AA1, which has been included in the study because it gives a large increase in coking pressure compared with the freshly mined coal. Consequently it is possible to eliminate the effect of maceral and mineral matter composition on coking pressure.

Oven wall pressures were measured using the BHP Billiton Mitsubishi Alliance Research Coke Oven (RCO) in Newcastle, Australia. A full description of the RCO operation and its output has been given by Mahoney et al. [12]. Although coal BU has been measured in a separate movable wall oven, measured values for other coals have been found to be relatively consistent with those from the RCO oven and would indicate a coking pressure value between 120 and 200 kPa in the RCO oven. Coke samples from these coke oven tests were also obtained for analysis.

2.2. Sample quenching

Partially carbonised samples were quenched in both a stainless steel cylindrical retort and the rheometer. For the retort, 20 g samples were charged at a bulk density of approximately 900 kg/m³. The cylinder was placed into the centre of a quartz refractory tube located in a split-horizontal furnace and heated under a constant stream of nitrogen to 300 °C at 20 °C/min, then at 3 °C/min up to the 550 °C at which stage the cylinder was removed from the furnace and quenched under a stream of nitrogen. This temperature was chosen to ensure samples had sufficient strength to be cut to specific sizes for gas permeability testing and to allow bulk densities to be estimated using the weight/volume method.

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