GRAPHITISING CARBONS FROM COAL VIA SOLVENT EXTRACTION: EFFECTS OF PROCESSING CONDITIONS ON PRODUCT QUALITY

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Introduction

The National Coal Board's basic process for the production of high purity coke from coal via solvent extraction has been described previously[1,2]. This paper outlines how the properties of coal extract cokes are dependent on the original coal and the manner in which it is processed. The ways in which the growth and coalescence of mesophase during coking influences the thermal expansion of graphite rods made from these extract cokes are described.

Experimental

A range of U.K. coals were digested in anthracene oil (a coal tar distillate boiling nominally 250-400°C) at 360-420°C for up to 5 hours. Either a 50 Kg batch rig or a 100 Kg h^{-1} continuous process development unit was used at pressures of up to 1 Nmm^{-2}. After filtration at 200-250°C the resulting coal solutions were coked either in 15 kg batches to a final temperature of 500°C or in a 120 kg hr^{-1} delayed coking facility with its associated rapid preheating system. Amongst the characterisation techniques applied to the cokes were measurements on 15 mm dia. extruded rods made from the coke after the appropriate calcining, forming, baking and graphitising. It should be noted that in strict contrast to Jasienko[3] no hydrogen is added.

Results and Discussion

The low temperature (0-100°C) longitudinal coefficient of thermal expansion of graphite rods (CTE) has long been used as a measure of the degree of needle likeness and quality of petroleum cokes. Coal extract cokes have been found to obey a similar relationship in that the larger the extent of anisotropy visible microscopically the lower the CTE. However, it is not yet established whether the same absolute relationship between CTE and quality applies to both types of cokes particularly when trying to correlate with the overall performance of a graphite electrode in an arc furnace. For example coal extract coke graphites exhibit a greater resistance to thermal shock than might be expected.

It appeared from initial tests that extract coke properties were very dependent on the parent coal. This aspect was studied first using fixed extraction and coking conditions; Fig. 1 shows the effect of coal rank on CTE for these conditions, with the higher rank coals generally giving the lower CTE's. However, there is an appreciable scatter about the mean line and this is due in part to differences in petrological composition of coals.

In the series of experiments when only the digestion time and temperature were varied, Fig. 2, it was found that short times and low temperatures gave small mesophase growth when the filtrate was eventually coked. Thus CTE reduced as residence time was increased. One of the mechanisms contributing to this is the depolymerisation of the coal which continues during prolonged digestion causing atoms and groups, that could sterically hinder alignment into graphite precursors, to be evolved.

The separation of the undissolved coal particles (mainly mineral matter and fusinite) from a coal digest is an

expensive operation and it is thus important economically to determine the effects of any deviations from highest efficiency that can technically be achieved. The results of experiments where a portion of the digest was made to bypass the filter are shown in Fig.3, where the ash in the coke is taken as a measure of the total solids passing the filter. The solids that pollute the filtrate inhibit mesophase growth and coalescence during coking thus giving a higher CTE. However, in the CRE process, after digestion under a certain range of conditions, the residue particles build up into a filter cake which is efficient both in retaining sub-micron particles and in giving a high filtering rate.

The series of experiments in which the preheat temperature (i.e. that at the inlet to the delayed coker) was altered shows that even having started with a good coal, and having digested and filtered it properly it is possible by incorrect coking, to produce isotropic rather than anisotropic coke. Fig.4 shows that unless a certain threshold temperature is achieved a high CTE graphite will eventually result. The macrostructure of the coke is dependent on how the polymerising feed (with the changing viscosity) is affected by the disturbances caused by gas and vapour, both in the preheater where there is a high degree of turbulence and in the coke bed itself. In some of these respects coal extract solutions are different to the feeds used to make petroleum cokes.

Acknowledgements

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References

1. J. Owen, 12th Conf. Carbon 1975.
2. USP 3,966,585 1976.
3. S. Jasienko, 12th Conf. Carbon 1975.

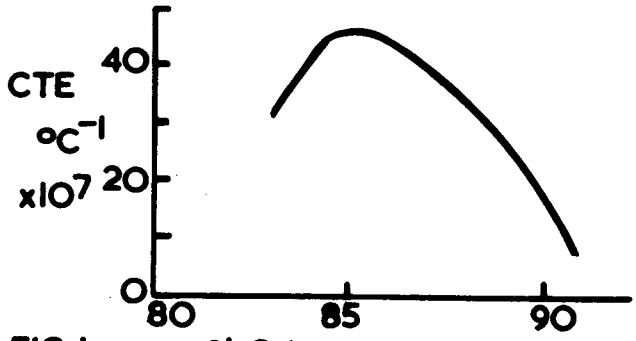

FIG.1.

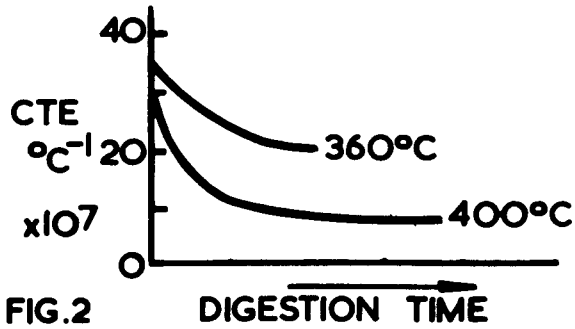

FIG.2

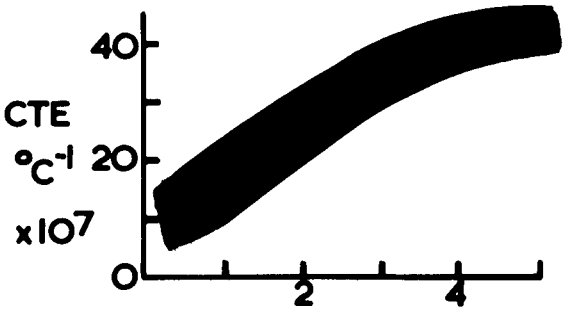

FIG.3

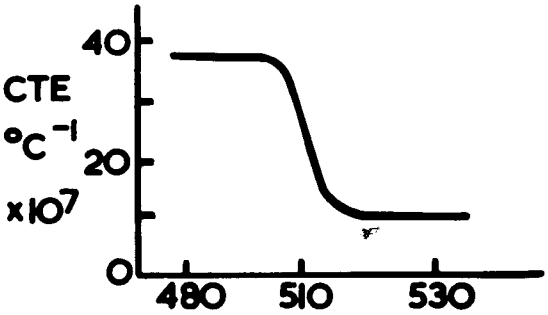

FIG.4