Wetting of Filler by Binder - A Simple Apparatus for Determining Wetting Temperatures

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Abstract. A rapid, simple method of determining wetting temperatures of pitch binders on coke fillers on a comparative basis was deemed important in order to control the quality of the final electrothermic graphite. To that end, a simple apparatus was designed and constructed. The method is applicable to the determination of the wetting temperature as measured by the change in contact angle of a liquid pitch sphere on a surface of carbonaceous materials. The carbonaceous materials can be green or calcined coke, carbon black, baked carbon or graphite. The effect of coke calcination temperature is used as a typical application.

Introduction

Many industrial processes require that one component be uniformly wet by another during some stage of the manufacturing process. One such example is the wetting of a petroleum coke filler by a coal tar pitch binder in the initial mixing step of the manufacture of artificial graphite.[1] A rapid, simple method of determining wetting temperatures of coal tar pitch binders on petroleum coke fillers on a comparative basis was deemed important in order to control the quality of the final electrothermic graphite. Although the following description was designed specifically for the wetting of a petroleum coke by a coal tar pitch, it should be applicable for the determination of comparative wetting temperatures of most liquids on most solids by appropriate modifications; as, for example, the wetting of a petroleum pitch on a baked carbon artifact.

Experimental

A simple apparatus has been assembled from standard taper joints and bushings with an immersion thermometer to measure temperature. This unit is placed on a standard laboratory hot plate which has a graphite crucible sitting on top. The crucible has been so designed that it fits under the assembled apparatus in such a way that when the thermometer bulb is placed into the center of the crucible cavity, it is completely covered with the sample.

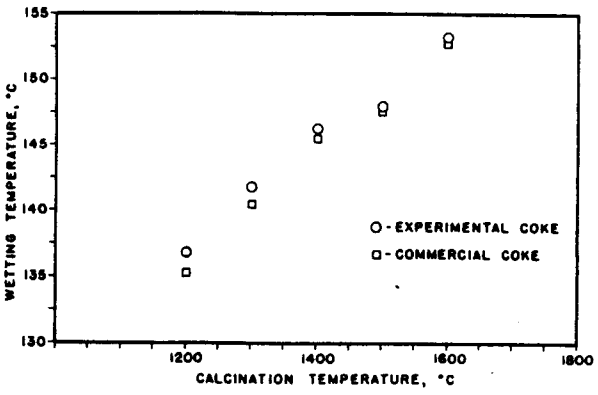
In principle, the test can be performed on any size coke particle. However, the more premium a coke becomes, the more porous the surface becomes. Further, the surface also becomes more uneven due to the nonuniform stacking of the needle-like components of a premium coke. Too coarse a particle will allow the pitch spheres to literally drop into a crevice. In such a situation, it will be impossible to measure the change from nonwetting to wetting. After some preliminary experimentation, it was found that a particle size obtained by passing the coke sample through a 0.425mm sieve but retained on an 0.180mm sieve, gave samples which were readily prepared and gave reproducible results with respect to wetting temperature from one determination to another.

In the specific use for which this system was developed, the material actually wetting the coke is a solid at room temperature, generally having a softening point of ca. 110° C.[2,3] Therefore, one could prepare small particles of solid coal-tar pitch having a particle size sufficient to pass through a 1.70mm sieve but retained on a 1.40mm sieve. It was found that this solid pitch particle readily changed into a sphere at the softening point of the pitch.

In a typical run, the particular coke sample is added to the crucible and four pieces of coal tar pitch are placed on the surface. The crucible is then placed on a laboratory hot plate capable of attaining a heating rate of 5°C per minute. That rate is typical of heating schedules reported to be used in the plant mixing of coke and pitch.[3] Tests are always run with the initial temperature taken at room temperature. The test continues until the pitch wets the coke sample; this can be well above 150°C (but usually less than 200°C), depending on both the coke and pitch being studied.[2,4,5] The wetting temperature for a given coke and pitch should not vary by more than 2°C from one pitch sphere to another.

Discussion

To test the usefulness of the system, green samples of commercially available premium cokes and two green samples of an experimental super premium coke were carefully sized and calcined to various temperatures in the 1200 to 1600°C range as described earlier.[6] The individual coke samples were loaded into the crucible and placed in the wetting apparatus. It was found that three commercially available premium cokes were indistinguishable from one another when wet by a standard binder pitch having a softening point near 110°C.[2,5] Further, when the wetting of the experimentally prepared super premium cokes was measured, it was found that they were wet by the same pitch some 2-4°C higher than the standard cokes. These data are shown graphically on the figure, where each point represents the average of 20 individual measurements.



Wetting Temperature Versus Calcination Temperature for Two Classes of Coke

This number of measurements was deemed necessary in order to ensure that the consistent higher wetting temperature at calcination temperature of the experimental cokes was real. This higher wetting temperature of the experimental cokes is probably due to the fact that they are more highly aligned than the commercial cokes. That is, they are more developed in a graphitic sense and should be more difficult to wet. For the sake of comparison, pure natural graphite is wet by the same pitch at 192°C. However, if an unpurified sample of natural graphite is used, the wetting temperature is decreased to 180°C.

Several factors can influence the wetting of a coke by a pitch.[4,5] As the calcination temperature increases, the coke surface is purified and the crystallites tend to align themselves more perfectly.[4] As the softening point of the pitch increases, so will the wetting temperature of a given coke. Wetting is viscosity dependent,[5] thus wetting probably occurs at the point of constant viscosity from pitch to pitch.[3] The calcination atmosphere can also affect the wetting temperature, as it can change both the chemical and physical characteristics of a coke.[4] Obviously, from the brief observation concerning purified and unpurified natural graphite, inorganic impurities also influence the pitch-coke interaction.

References

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