STRUCTURAL CHANGE DURING DESULFURIZATION OF PETROLEUM COKE

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Experiments were carried out with the aim of removing the sulfur contained in petroleum coke by treatment at a temperature lower than the temperature at which it departs spontaneously. The coke used had already been calcined at 1300°C and had a sulfur content of 1.27%. Two desulfurization methods were tried: treatment with hydrogen and vacuum degassing. During these treatments, four phenomena occur simultaneously:

1. The departure of part of the sulfur.
2. A wright loss of the coke, partly caused by formation of methane in the case of the hydrogen treatment.
3. The transformation of a small fraction of the coke into well crystallized graphite ($\frac{c}{2}$=3.36 Å, Lc≃500Å), which in indicated by the appearance of a second peak in the (002) line.
4. The formation of small cracks or granulations in the coke.

With the hydrogen treatment there is no departure of sulfur at 1200°C or below. At 1300°C, desulfurization starts after a few hours, and proceeds slowly: after 750h, the sulfur content of the coke is 0,5%. The weight loss is 14%. At that time, about 15% of the coke is transformed into graphite. In an identical thermal treatment under argon carried out for comparison, there is neither elimination of sulfur nor formation of graphite, and the weight loss amounts to only 2.5%.

In the treatments under vacuum (1300°, 1400°, 1500°C), the sulfur content at first rapidly decreases to a value that depends on the temperature, then keeps on decreasing very slowly. About 5% of the coke gets transformed into graphite. It is concluded that desulfurization by hydrogen or vacuum treatment is neither rapid nor total. The process of formation of graphite during these treatments is different from the normal graphitization process.