

Development of Pore Size Distribution and Physical Properties of Carbons During Baking

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Introduction

The physical properties of carbon artefacts are influenced by the composition of the mix on the one hand and the green density, the heating rate and the final heat treatment temperature on the other hand. Therefore the process parameters ought to be optimized for every grade. In order to elucidate to a certain extent the interdependence of physical properties and process variables the following experiments had been performed.

Experimental

Samples of different grades had been pressed at pressures between 500 and 2.000 atmospheres and were heat treated to temperatures of 275, 350, 500 and 930 °C at different heating rates. The baking was performed in a laboratory furnace under inert atmosphere. The physical properties in the green and baked state as well as the pore size distribution were measured.

Results

Increasing pressure enhanced the green density and resulted in a lower porosity in the green state as could be expected. The pore size distribution of the green samples was not affected by the pressure as can be seen from fig. 1. A decrease of the pressure by the factor 4 resulted in doubling the pore volume. The pore sizes remained the same. The pore size distributions for different grades were different; the character of the curves for one grade baked at different heat treatment temperatures remains more or less the same depending on the binding system.

The biggest difference found can be seen from fig. 2. Graphitization can result in a shift of the mean pore size by one order of magnitude in the direction of bigger pores. The heating rate is of small influence at low pressures but increases the pore volume of samples densified at higher pressures.

Fig. 4 compares the pore size distributions measured on samples of one grade in the green state and after heat treatment to different final heat treatment temperatures with two different heating rates. The binder used was a soft pitch. One can see an increase of the total porosity with increasing final heat treatment temperature

up to 500 °C. At higher HTT the porosity decreases again because of the shrinkage of the samples. Slow heating rates lead to higher porosities because of a longer residence time in the "distillation range" of the pyrolysis.

Figure 5 gives the results of the same experiments performed with a grade containing polymerizing agent in the binder system. The influence of the final HTT on the porosity is much lower. The effect of the heating rate is inverse as compared with fig. 4. A lower heating rate results in a higher condensation and polymerisation of the binder which takes place already at temperatures where distillation is still negligible.

In comparison to fig. 4, one cannot find pores in the range of 0,3 to 1 micron and one can see a narrow pore size distribution. But the mean pore size for both grades is about 1,5 micron.

Fig. 6 shows the physical properties for the "soft pitch grade" for different pressures, heating rates and final HTT.

As it was to be expected increasing HTT decreases the electrical resistivity. For lower heating rates the decrease is even bigger. The hardness and the flexural strength at low HTT (275, 350 °C) are higher at high heating rates because the residence time in the "distillation range" is shorter. At 500 °C already shrinkage occurs.

The dependence of the density on heating rate and final HTT can be explained by the same influence of shrinkage and distillation.

Fig. 7 shows the results of the same experiments for grade 2. The influence of the heating rate and the final HTT on the physical properties is much smaller due to the polymerization of the binding system.

The flexural strength after heat treatment at 275 °C is already 25 % of that at 930 °C.

Discussion

It could be expected that the final physical properties of carbon artefacts would be already predetermined at temperatures of about 450 °C i.e. when mesophase formation takes place. But as can be seen from fig. 5 the character of a carbon artefact can have developed itself at a much lower temperature.

