

STUDY OF SUPERFICIAL GROUPS OF SEVERAL GRAPHITE OXIDES IN HELIUM AND HYDROGEN.

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Introduction

A study of the relation between thermal decomposition of several graphite oxides and their degrees of oxidation has been performed by means of thermogravimetry (TG) and differential scanning calorimetry (DSC). The evolution of different gases has been studied by mass spectrometry (MS).

The graphite oxidation products were obtained in liquid medium, following the Staudenmaier [1] method.

From previous studies [2-6]* concerning the characterization of graphite oxides, it is well known that in the structure hydroxyl, carbonyl, ether, epoxy and peroxy groups are present. In the thermal decomposition, these groups lead to the production of CO₂, CO and H₂O mainly.

In the present study, the TG, DSC and MS experiments were carried out under two different atmospheres (H₂ and He) in order to analyze the influence of the surrounding gas over the decomposition processes.

Experimental

Several graphite oxides with different oxidation degrees were prepared as above described. All the employed products were reagent grade. The samples were denoted with a G letter followed by the oxidation time, in hours.

TG and DSC studies were carried out with samples of about 25 mg, under dynamic atmospheres of H₂ and He, with a gas flow of 50 cm³/min, using a heating rate of 1°C/min from 30° to 300°C. MS analyses were performed out simultaneously with TG experiments.

Thermal analyses were performed in a SEIKO SSC/5200 system with TG/DTA 320 and DSC 320 cells. MS studies were carried out by means of a BALZERS INSTRUMENTS THERMOSTAR GSD 300T quadrupole spectrometer.

Results and discussion

TG curves, showed in figure 1, indicate that weight losses are similar as well in hydrogen as helium atmosphere [2]. From this fact we could conclude that the employed surrounding atmospheres are not a factor capable of influencing the decomposition processes in the pressure conditions of the experiments.

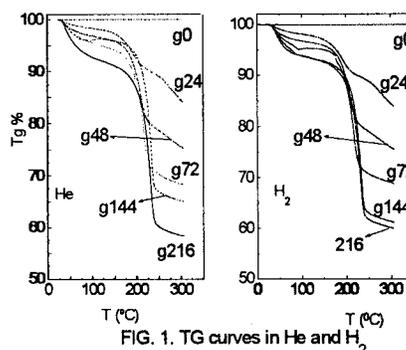


FIG. 1. TG curves in He and H₂

Two different process with weight loss are detected. In the first one, at 60-70°C, the weight loss was about 5% in all the studied samples. The second, and more important weight loss takes place between 175 and 250°C, and it is proportional to the oxidation degree of the samples. In the most oxidized one, G-216, the measured total weight loss is about 40%, whereas in the less oxidized samples (G-24) the detected weight loss was only about 10%.

The first of these processes corresponds with the water elimination as can be easily see from the mass spectrum (Figure 2). The similar shape of curves corresponding to the experiments performed out in H₂ and He atmospheres, and the low temperature at which the weight loss takes place seem indicate that these water molecules proceed from the surface of the oxides graphites.

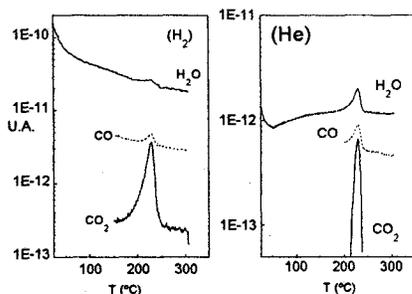


FIG. 2. Mass spectra of sample g-168 in H₂ and He

The second decomposition process corresponds to the elimination of oxygenated molecules proceeding from surface functional groups, as MS spectra show. So, the amount of produced gases increase with the oxidation degree of the material.

The mass spectra of graphite oxides are different under helium or hydrogen atmospheres. The evolved gases are, in both cases, CO₂, CO and H₂O. Evolution of CO₂ is proportional to the oxidation degree of the sample and takes place between 175 and 250°C, in good agreement with the TG dates. Evolution of CO, not detected in the less oxidized samples, takes place at higher temperatures.

Nevertheless, the composition of the gaseous mixture is depending on the surrounding gas. This fact could be interpreted assuming the occurrence of reactions between the evolved gases proceeding from the decomposition reactions and the H₂ of the ambience. Such reactions are not possible under helium and, thus, the composition of the gaseous phase in bothe series of experiments are different.

This is in good agreement with the enthalpy values obtained from DSC measurements. (Figure 3).

References

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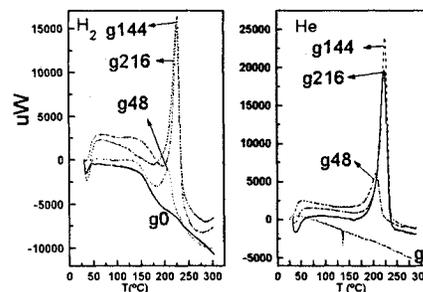


FIGURE 2. DSC curves in H₂ and He