NANOSTRUCTURED CARBON OBTAINED BY LASER PYROLYSIS FROM GAS-PHASE REACTANTS

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Introduction

The laser synthesis of nanostructured powders is based on the resonance between the laser radiation and the chemical reactants or sensitizer. The high temperature gradients and very rapid reaction time involved in the process of laser pyrolysis are characteristics of the process leading to the very fine powders. Our results of study concerning the synthesis of nanostructured carbon by laser pyrolysis from gas-phase hydrocarbons are reported here.

Experimental

The usual design for the synthesis apparatus allows for a cross flow configuration where the beam of a CW CO_2 laser (λ=10.6µm) orthogonal intersects the flow of emergent gases. For obtaining carbon nanopowders one should use carbon-containing precursors and we report here our results concerning acetylene and ethylene as carbon donors. Since acetylene does not absorb the laser radiation, a sensitizer should be used as energy transfer agent, which may be either SF_6 or C_2H_4. Different experiments were performed by varying one parameter and trying to keep all the others constant. The powder efficiency (%) and productivity (g/h) in the case of runs with different laser power values (100-900 W), pressures (250-950 mbarr), gas flows (100-300 sccm) were quantified and the powder characteristics were investigated by TEM coupled with an energy dispersive x-ray spectrometer (EDAX), HRTEM, XRD and Raman spectroscopy. The exhaust gases and the toluene extracts of carbon powders were analyzed by IR spectroscopy.

Results and Discussions

As in our experimental conditions SF_6 is not chemically inert and could undergo different degrees of dissociation and also reactions with other species, C_2H_4 was also used as carbon-donor in order to avoid the powder contamination. The IR spectrum of exhaust gases in a run using SF_6 as sensitizer (Fig. 1) shows the presence of gaseous species produced by the partial dissociation of SF_6. Investigations with a TEM with EDAX showed the presence of sulfur in carbon powders, where higher sulfur concentration belong to smaller carbon particles (Fig. 2). TEM analysis of the samples shows that these particles may be well dispersed having a rather spherical shape with diameter around 20 nm (Fig. 3) or may coalesce in bigger particles. The SAED analysis as well as the Raman spectra (Fig. 4) suggests the presence of the amorphous carbon (D band) and partial crystalline carbon with a small crystallite size (G band). In addition, the XRD spectrum (Fig. 5) revealed the presence of rather uniform round particles with a turbostratic structure of around 5 graphitic planes spaced at 3.66Å. The parametric dependence of soot formation shows that powder output and productivity as well as powder morphology depend on the experimental parameters like gas-flows, nature of precursors, laser power, temperature, pressure and reaction time. Using C_2H_4 as precursor, high pressure (up to 850 mbarr) and higher gas-flows lead to better productivity of carbon powder (Fig. 6). The using of a C_2H_2/SF_6 mixture at a lower pressure (~250 mbarr) leads to carbon powders composed by small particles (15-20 nm), characterized by a higher content of graphitic structure.

Conclusions

The particles composing the carbon powders synthesized by laser pyrolysis of gas-phase hydrocarbons are generally well dispersed and have a rather spherical shape with diameters depending on experimental conditions. Their characterization shows the presence of amorphous carbon and crystallites having usually a turbostratic structure.

Acknowledgements

This work was supported by the NATO under contract number NATO-SfP-974214 and by the Romanian Ministry of Education & Research under contract number RO 99404.

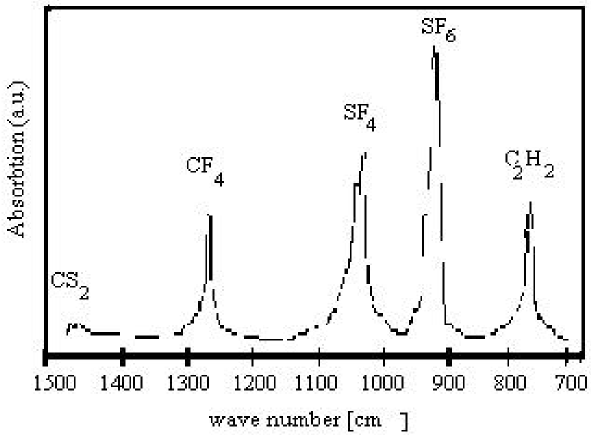

Fig. 1. The IR spectrum of exhaust gases in a run using C_2H_2/SF_6 mixture

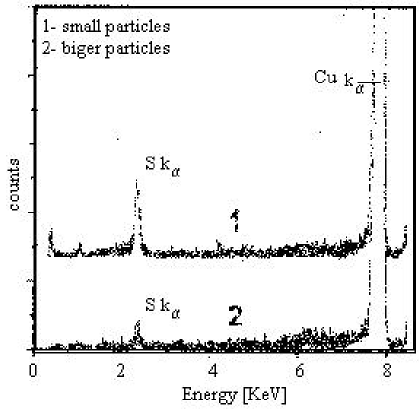

Fig. 2. The EDAX spectra showing sulfur concentrations for particles of different sizes

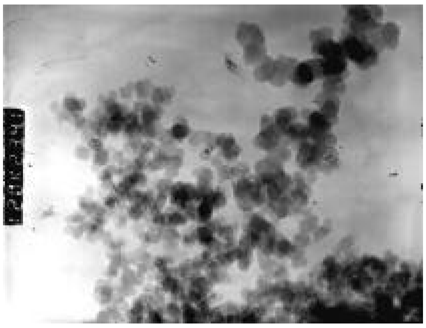

Fig. 3. TEM image of carbon powder of a run using acetylene as precursor

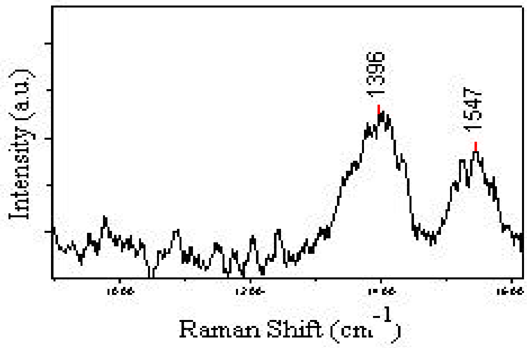

Fig. 4. The 1600-1100 cm^{-1} region of Raman spectrum

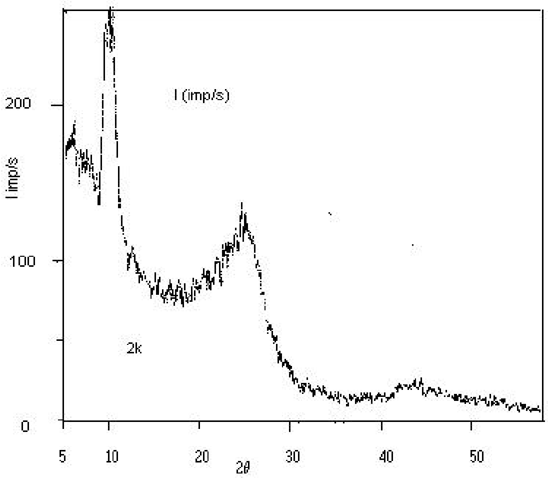

Fig. 5. XRD spectrum

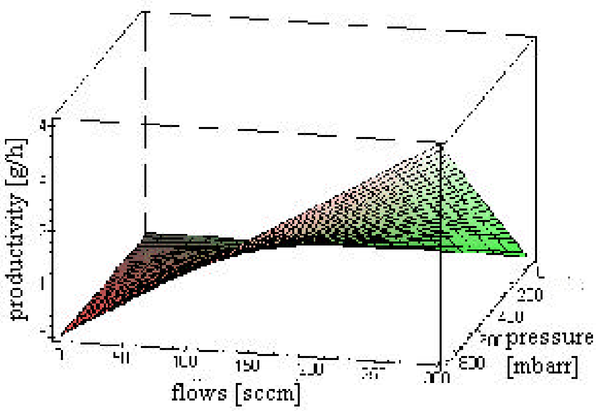

Fig. 6. The productivity (g/h) of carbon powder vs. pressure and gas -flow