Synthesis of Metal—Carbon Conglomerates under the Action of Pulsed High-Voltage Discharge in a Liquid Dielectric Medium

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Abstract—At present a variety of technologies for the fabrication and storage of nanosized particles of metals and conglomerates, which are used as catalysts in different petrochemical processes, in particular, the Fischer—Tropsch process, has been developed and introduced in industry. Iron and cobalt catalysts have gained the greatest acceptance.

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The major advantage iron–carbon catalysts offer for the Fischer–Tropsch process is their ability to work efficiently in the carbon monoxide hydrogenation system at the CO: H_2 ratio of 1: 2, as well as the low cost of these catalysts.

In the present work we propose a technology for the fabrication of nanodispersed iron—carbon conglomerates under the action of a pulsed nanosecond high-voltage discharge in *o*-xylene.

The setup for the fabrication of iron–carbon conglomerates is a discharge system with a reaction chamber immersed into a liquid dielectric (Fig. 1). The high-voltage electrode implemented as a set of various-diameter steel grids (30–150 μ m) is connected to a pulse generator. A plane ground electrode is placed a few millimeters apart from the grids and perpendicular to them.

Discharge pulse is generated after the discharge capacitor C has been charged to a required voltage and

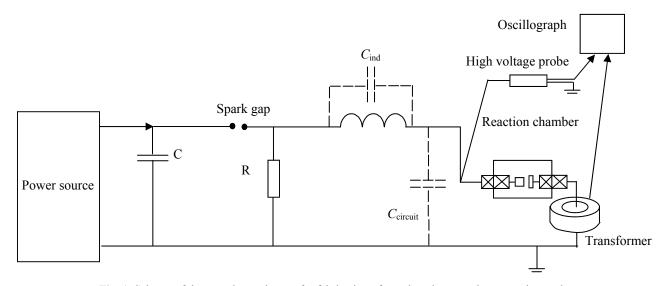


Fig. 1. Scheme of the experimental setup for fabrication of metal–carbon conglomerates in o-xylene.

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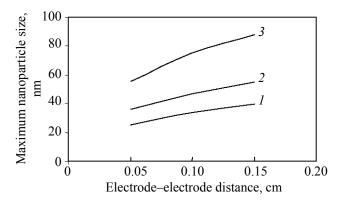


Fig. 2. Dependence of the maximum radius of iron nanoparticles on the electrode–electrode distance at the voltage of (1) 20, (2) 25, and (3) 30 kV.

the spark gap has broken down. The capacitor C voltage is supplied to the resistor R and further transmitted, through a high voltage probe, to the electrode of the discharge (reaction) chamber. Therewith, a fast potential jump arises at the high-voltage electrode of the reaction chamber. Further on a slower inductivity $C_{\rm ind}$ rise takes place.

Subsequently, the second stage of breakdown development, a discharge of the main working chamber through the working inductor L. The discharge current grows very slowly with a time constant.

Xylene is exposed to pulsed high-voltage discharge for 1 h at a pulse frequency of 10 Hz. Iron nanoparticles are formed as the result of erosion of the electrode at the "fast" discharge stage. The diameter of the high-voltage electrode grid used in our experiments was 30 μ m. A strong electric field is created at the electrode edge, and this filed breaks the particles formed by electrode erosion to particles a few tens of nanometers in size (Fig. 2).

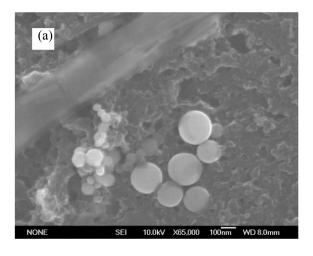
The "slow" stage of discharge in *o*-xylene involves pyrolysis to form a carbon material.

Figure 3 shows photo images of iron and ironcarbon conglomerate nanoparticles obtained under the action of pulsed high-voltage discharge in a liquid dielectric medium.

Nanoparticle conglomerates formed due to drying droplets of suspensions containing a nanosized catalyst, deposited on silicon surface. The average size of conglomerates is 600–800 nm. They consist of particles with characteristic radii of 10–100 nm. The size distribution of iron nanoparticles is presented in Fig. 4.

The elemental composition of the iron–carbon catalyst was studied by mapping a single crystal with a deposited catalyst; the chemical composition was determined in lighter fields (Fig. 5).

As seen, iron nanoparticles are distributed over the entire volume of the studied field. The catalytic activity of the iron–carbon nanostructures fabricated by pulsed high-voltage discharge in a liquid *o*-xylene medium was assessed to show that this type of catalysts offers advantages over traditional catalysts in



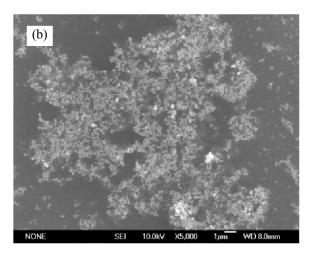


Fig. 3. Nanoparticles of (a) iron and (b) iron–carbon conglomerate, obtained under the action of pulsed high-voltage discharge in *o*-xelene.

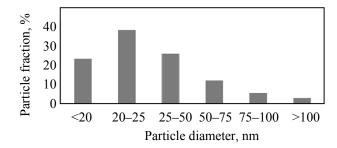


Fig. 4. Diameter distribution of iron nanoparticles.

the Fischer–Tropsch process. The conversion of carbon monoxide with the iron–carbon catalyst reaches 70%, and the yield of target products (liquid hydrocarbons) is \sim 100 g m⁻³, which corresponds to the selectivity parameter of about 70%.

Thus, nanodispersed iron-carbon catalysts are promising candidates for industrial applications. These catalysts are superior in performance [about 0.4 kg C5+/(kg Fe×h)] to known industrial catalysts 1.5–2 times.

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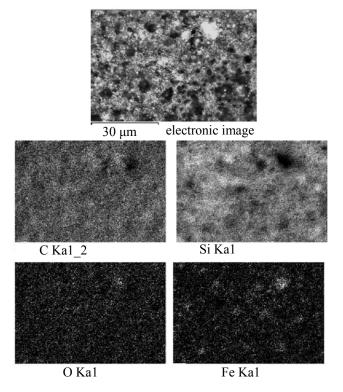


Fig. 5. Results of study of the elemental composition of the iron carbon conglomerate on the surface of a silicon single crystal.

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