IRON PARTICLE FORMATION BY PHOTO-DISSOCIATION OF $Fe(CO)_5$

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The formation of iron particles during the thermal decomposition of $Fe(CO)_5$ has been investigated in many works [1, 2]. In this study the new method of iron particles formation at room temperature by the photo-dissociation of $Fe(CO)_5$ vapor was applied [3]. Under ultra-violet radiation in the range $200 < \lambda < 350$ nm $Fe(CO)_5$ dissociates to one iron atom and five stable CO molecules [4], yielding supersaturated iron vapor with well-controlled properties. Then, the formation and growth of iron particles can be observed depending on iron concentration, bath gas composition and pressure. The method of UV-laser particle photosynthesis provides conditions that are convenient to study the detailed mechanism of an iron particles growth in gas the phase. Besides that, this method of particles synthesis can be very useful for different applications. Thus, the main goal of this study is the investigation of the iron particles condensation process and the determination of conditions for formation of iron particles with adjusted properties.

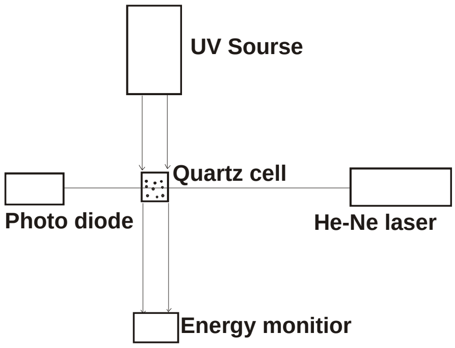

Fig. 1. The experimental setup for iron particle producing and diagnostics

The experimental setup for iron particles formation study is presented in Fig.1. A reaction volume - quartz cell (20×10×10 mm) was evacuated and filled with the vapor of pure $Fe(CO)_5$ or $Fe(CO)_5$ diluted by Ar at a variable mixture ratio and total pressures from 1 mbar to 1 bar. For photo-dissociation of $Fe(CO)_5$ a beam from a flash xenon lamp (spectra range 200-400 nm, 10 mJ per pulse, the time of the pulse of about 5 μs was adjusted to the size of the reaction cell in order to homogeneously illuminate the entire reaction volume. The energy of each light pulse was measured before and during the experiment to evaluate the energy absorbed by $Fe(CO)_5$ molecules. The diagnostic part consisted of the laser light extinction measurements based on the He-Ne laser beam attenuation by the iron particles. In Fig.2 the example of an experimental extinction signal from growing iron particles is shown. The optical density of final iron particles was extracted from the extinction time profiles. In Fig.3 the optical density of final iron particles is demonstrated in dependence of $Fe(CO)_5$ pressure and fitted by the exponential damping curve. It is clearly seen that optical density of iron particles increases with the pressure rise up to the saturated vapor pressure of $Fe(CO)_5$ (20 mbar).

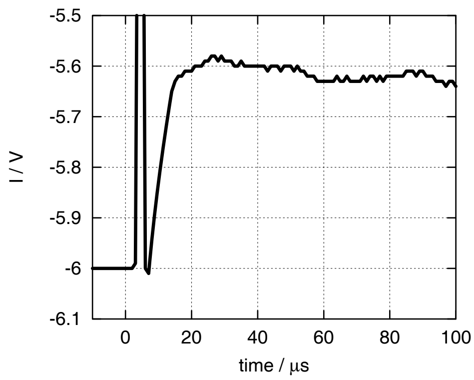

Fig. 2. Experimental time-profile of extinction from iron particles

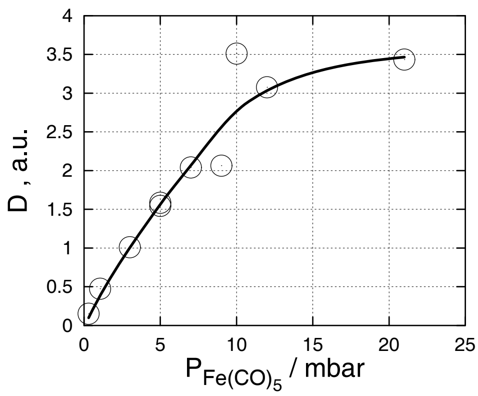

Fig. 3. Optical density of final iron particles versus pressure of $Fe(CO)_5$

The sizes and structure of iron particles were analysed by transmission electron microscopy (TEM) and microdiffraction measurements (MDF). The particles were collected from the gas phase under the natural gravity conditions on the TEM grid deposited on the bottom of the quartz cell. In dependence of gas-diluter pressure the iron particles had quite different sizes and structure. In the experiments with pure $Fe(CO)_5$ the particles look like the big agglomerates with sizes of 40-60 nm. MDF showed that their structure was amorphous without any crystallized regions. At intermediate argon pressure (100 mbar) the micrographs represented the chains of iron particle with the sizes of about 10-20 nm with small crystalline regions inside. At the highest investigated pressure - 1 bar of argon yielded the particles of rather small sizes of 4-8 nm combined the thin web on the surface of the TEM grid. These particles were fully crystallized. In Fig. 4, the micrographs and diffraction pictures of the iron particles formed at different conditions are shown.

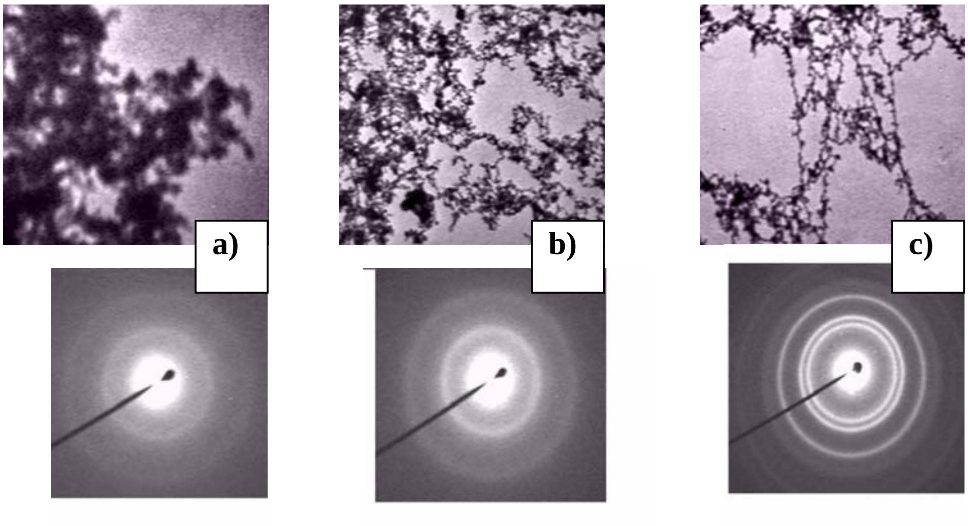

Fig. 4. The micrographs and microdiffraction pictures of the iron particles formed at 10 mbar of $Fe(CO)_5$ - a), at 10 mbar $Fe(CO)_5$+100 mbar Ar -b) and at 10 mbar $Fe(CO)_5$+1 bar Ar - c). Magnification - 10 000

Thus, the first experiments of iron particle formation in the gas phase under pulse photolysis of $Fe(CO)_5$ were carried out. The optical density extracted from experiments gave the kinetic information about the iron particle growth. The TEM and MDF measurements resulted in the final particles sizes and structure. The yield of the particles and their size and structure depend on the $Fe(CO)_5$ concentration and the pressure of gas-diluter.

Acknowledgements

The authors thank Prof. V.G. Babaev (Moscow State University) for TEM and MDF measurements. This work was supported by INTAS and Russian Foundation for Basic Research.

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