Synthesis of Titanium Nanoparticles in Liquid Xenon by a High-Voltage Discharge[1]

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Abstract—The formation of titanium nanoparticles (NPs) in a high-voltage electric discharge between titanium electrodes in liquid xenon at a temperature of $-105°C$ has been observed. It has been shown that these titanium nanoparticles have a spherical shape with an average diameter of <50 nm and they possess high chemical activity. This makes it possible when a relative mass concentration of NP reaches $\sim 10^{-6}$ to efficiently purify xenon from electronegative impurities for its use as a working medium for a new generation of high-efficiency nuclear radiation detectors.

Over the past few decades, there has been significant progress in the study of nanomaterials and their unique electrical, optical, magnetic and catalytic properties [1]. Particular attention has been paid to the synthesis of metallic nanoparticles and study of their properties [2]. In particular, it was shown that the chemical activity of nanodispersed titanium in gases significantly exceeds the chemical activity of standard nonevaporable getters based on sponge titanium with a micron pore size [3]. For this reason, methods of generating titanium nanoparticles in inert gases are being developed [4]. A spark method for the liquefied noble gases purification based on generation of a fine-dispersed titanium dust by an electric discharge is also known [5, 6]. According to the published data, liquids such as distilled water, ethanol, or solutions of metal salts in water are usually used for the synthesis of metallic nanoparticles in liquid media [1].

In the present study, the formation of titanium nanoparticles (NPs) in macroscopic samples of liquid xenon has been observed for the first time. The exper-imental setup is a stainless steel cryostat in which up to 100 L of xenon with a total mass of up to 300 kg can be condensed [7]. To disperse titanium in liquid xenon, a two-electrode "blade-plane" discharge system with an interelectrode gap of an order of 1 mm has been used. The electrodes were made of titanium sheet of VT1-0 type with a thickness of 1.5 mm. The high-voltage cathode ("blade") is a cylinder with a diameter of 20 cm and a height of 3 cm, and the grounded anode is made as a flat ring. The discharge circuit was formed by a high-voltage 6-m cable and a 600-pF capacitor and a VS20-10 high-voltage dc power supply unit as shown in Fig. 1a. The typical current versus voltage characteristic of the high-voltage discharge in liquid xenon is shown in Fig. 1b. A waveform of the discharge current is shown in Fig. 1c. In the case of substantially pure xenon (when the lifetime of electrons before the capture by electronegative impurities exceeds 10 μs), there is a clearly manifested relatively low-voltage branch of a corona discharge on the current-voltage curve, which transforms into a spark discharge when the voltage between electrodes exceeds 12.5 kV.

The monitoring of degree of liquid xenon contamination by electronegative impurities was based on the

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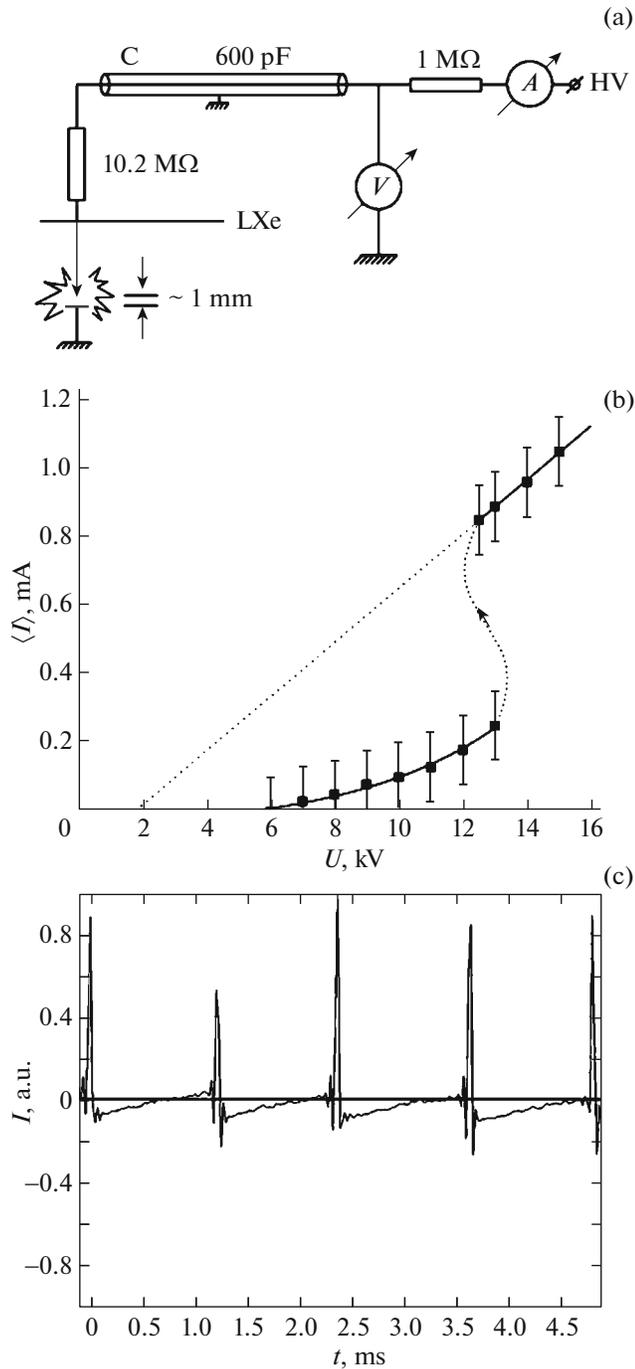[1]The article was translated by the authors.

Fig. 1. (a) Schematic drawing of the discharge circuit (C is a cable with a capacity of 600 pF, LXe is the level of liquid xenon, HV is the high voltage), (b) current–voltage characteristic of the discharge, and (c) waveform of the discharge current.
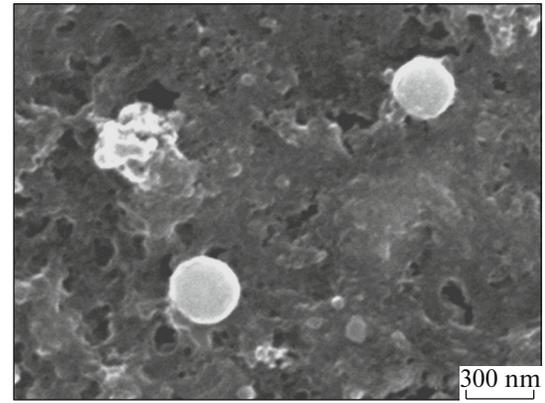

Fig. 2. An image of titanium nanoparticles deposited on a silver-coated copper pad after evaporation of liquid xenon (obtained with Carl Zeiss EVO 50 scanning electron microscope).
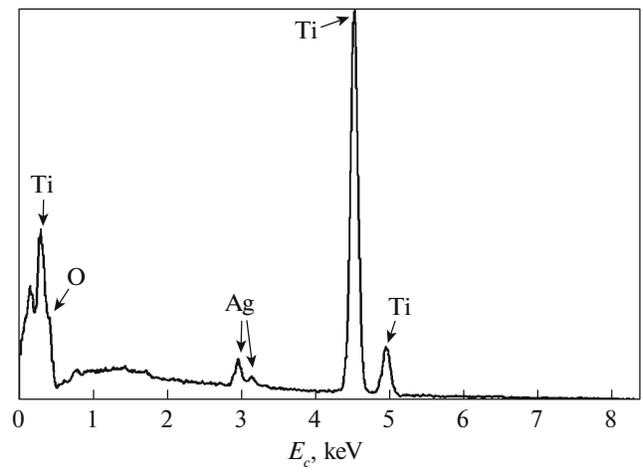

Fig. 3. Elemental composition of titanium nanoparticles measured by energy dispersive X-ray spectroscopy on a Carl Zeiss EVO 50 scanning electron microscope (E_c is the characteristic radiation photon energy).

measurement of lifetime of quasi-free electrons before their capture using a pulsed X-ray ionization chamber installed in the discharge chamber [7]. For this study, samples of liquid xenon with a mass of 10–205 kg were used. With an average discharge power of 2 W, it was necessary to generate a spark discharge of at least 50 h

in order to achieve a noticeable increase in the lifetime of quasi-free electrons from the initial value of the lifetime ≪1 μs to more than 100 μs at the final stage of the experiment (in the range of drift fields from 10 to 500 V/cm). The lifetime of quasi-free electrons before the capture by electronegative impurities increased exponentially at a rate of

$$\tau_e = \tau_0 \exp(t/t_0),$$

where $t_0 \approx 40$ h. After removing the xenon and opening the chamber, the titanium dust was analyzed with a Carl Zeiss EVO 50 scanning electron microscope. It was found that titanium dust is produced in the liquid xenon by the method described above. It consists of NPs with shapes that are close to spherical (Fig. 2) and the average diameter of which does not exceed 50 nm.

Table 1. Content of molecular impurities in xenon before and after purification by nanodispersed titanium

Impurity	H_2	N_2	O_2	CF_4	CH_4	CO_2	H_2O	SF_6	$C_nH_mR_k$
Impurity content in the initial sample, ppm	0.325	0.950	0.180	<0.001	0.210	0.835	1.4	0.015	7.450
Impurity content upon purification, ppm	0.600	0.700	0.050	<0.001	0.065	0.015	1.0	<0.0001	1.520

The elemental composition of NP was determined by a method of energy-dispersive X-ray spectroscopy. It was shown that the nanoparticles mainly consist of titanium (Fig. 3). The presence of oxygen is probably related to the interaction of electronegative impurities in the liquid xenon with a chemically pure surface of titanium NPs. The presence of silver is explained by the silver coating of the metal substrate on which titanium dust was deposited.

The production efficiency of nanodispersed titanium was 60 μg/C in the samples of liquid xenon with a mass of several tens of kilograms, which roughly corresponds to the production efficiency of the titanium getter in the work of Pokachalov et al. [8] in samples of liquid xenon with a mass of about 100 g.

The quantitative analysis of produced nanoparticles has shown that the beginning of the electronegative impurities' effective absorption corresponds to the achievement of the order of 1.2 mg/L NP mass concentration in liquid xenon. It corresponds to approximately 100 mg of the total mass of the NPs produced in the experiment with 205 kg of liquid xenon. The total surface area of the nanoparticles was then of the order of 5 m^2. This means that the mass ratio of the titanium-based getter to its effective area is several orders of magnitude smaller than that of the widely known St122 (Ti-St707) hot metal getter type produced by SAES Pure Gases. This getter was not able to provide purification of xenon contaminated with high molecular weight impurities and SF_6 gas in the initial gas with concentrations specified in the attached Table 1. Thus, it has been shown that NP of titanium can be used for purification of macroscopic samples of liquid xenon used as operating media for modern nuclear radiation and elementary particle detectors [6, 7].

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