



Purification of tellurium through thermal decomposition of plasma prepared tellurium hydride

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ABSTRACT

For the first time tellurium has been purified by means of plasma-enhanced chemical transport reaction with intermediate formation of tellurium hydride (H_2Te) and the following its thermal decomposition with formation of high-purity tellurium and hydrogen. Recently developed direct interaction of hydrogen with tellurium vapors at low pressure in inductively coupled non-equilibrium plasma discharge underlain the novel approach of purification. The metal and carbon-containing impurities behavior was also studied. The possible mechanism of their conversion during the process has been discussed. In order to demonstrate effectiveness of the suggested method, As_2Te_3 films have been prepared from the initial tellurium and tellurium after purification. They were studied by mass-spectrometry technique for comparison.

1. Introduction

High-purity tellurium is one of the most important components of chalcogenide glasses transparent in the middle and near infrared ranges [1]. In addition, it is the basic element of semiconductor materials [2,3]. Finally, prediction of high- T_c superconductivity in tellurium hydrides stabilized at megabar pressures has recently been reported [4]. The degree of purity of the initial substances mainly determines properties and practical applicability of the final materials [5]. That is why development of technologies of deep purification of chalcogens, in general, and tellurium, in particular, causes an increasing interest.

As a rule, purification tellurium from impurities is a multi-stage process including mechanical, chemical, and physicochemical steps. For example, the methods described in [6–8] consist of dissolution of technical tellurium in hydrogen peroxide, boiling the resulting telluric acid with addition of tellurium, preparation of solution of potassium tellurate and deposition of the purified tellurium by means of electrolysis. However, in this case, extra contamination of the final tellurium coming from the reagents of the solution should be taken into account. The additional appearance of impurities in the final tellurium may be because of incomplete conversion of precursors as well [9,10].

As the examples of physical methods of tellurium purification from impurities are its sublimation in vacuum in atmosphere of hydrogen or inert gases, zone melting or directional crystallization [11,12]. The

main disadvantage of the mentioned methods, as well as other methods of physical purification of tellurium, is the contamination of tellurium with materials of setup due to a high temperature, long duration and a continuous contact of the purified tellurium with the walls of the reactor as the authors concluded in [10]. In addition, it has been established in [13] that all known methods of physical purification do not provide the purification of tellurium from nanoscale hetero inclusions of different nature. There are a lot of strong scientific investigations devoted to the theme of nanoparticles, e.g. [14–21].

In this connection, the hydrides of elements seem to be an attractive chemical form of the chalcogens in terms of obtaining them in a high-purity state. Indeed, H_2S , H_2Se and H_2Te possess low values of melting and boiling temperatures, low viscosity and surface tension to be purified effectively by simple distillation or rectification [22]. The possibility of implementation of the process as the entire multistage one including preparation, purification and decomposition of hydrides becomes the main advantage of the hydride distillation/rectification technique [22].

The traditional method of “wet” chemistry for synthesis of hydrides of chalcogens H_2S and H_2Se is the acid hydrolysis of magnesium, zinc and aluminum chalcogenides. Tellurium hydride H_2Te was also synthesized by this approach with the yield close to 80% of the theoretically possible, but with a negligible quantity of yield of a few grams [23].

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The most technologically convenient technique has turned out to be the electrochemical synthesis of H_2Te implemented by electrolysis of sulfuric or orthophosphoric acid with tellurium cathode [24,25]. The maximum yield of H_2Te in the initial stage of the process reaches 80% from the theoretically possible, but it drastically decreases with time due to accumulation of tellurium particles in electrolyte. Furthermore, extra contamination of the final tellurium hydride coming from the reagents of the solution should be taken into account, too [26,27].

That is why it is of great interest to develop a method of the one-stage purification of tellurium based on formation and decomposition of its hydride, especially, if it is based on the physical “contactless” methods of activation of chemical interaction, e.g. by electron impacts in plasma discharge.

The goals of this work are, on the one hand, to suggest a novel method of tellurium purification based on plasma-chemical direct synthesis of H_2Te by low-temperature hydrogen RF inductively coupled plasma discharge at low pressure, and, on the other hand, to implement the process as the entire multistage one including preparation, purification and decomposition of hydride.

2. Experimental

The commercial tellurium 3N and the hydrogen of high purity (the hydrogen content was at least 99.9999%, oxygen – $2 \cdot 10^{-5}\%$, argon – $2 \cdot 10^{-5}\%$, nitrogen – $8 \cdot 10^{-5}\%$ and water vapor – $1 \cdot 10^{-4}\%$) were used as the precursors in our experiments. The plasma-chemical device designed for purification of tellurium via its hydride is shown in Fig. 1. The setup consists of a system of input of the initial commercial tellurium, a glass tube plasma-chemical reactor equipped with an external inductor, a trap cooled by liquid nitrogen to collect the obtained H_2Te , and a RF generator (40 MHz). The input system comprises the quartz ampoule with external furnace. Hydrogen of high-purity was utilized as a plasma feed gas, as a carrier gas, and as a precursor. It was blown through the quartz plasma-chemical reactor with the rate 10 mL/min.

The total pressure in the system was kept constantly 13 Pa. The temperature of the external heater of the initial tellurium quartz reservoir was maintained constantly $570^\circ C$ during the all experiments. The plasma prepared H_2Te was accumulated in the trap cooled by liquid nitrogen located just after plasma-chemical reactor. Then, H_2Te was decomposed by its slow heating to $0^\circ C$. The “telluride mirror” reaction was observed” (Fig. 2) [28]:



The beginning of the tellurium hydride decomposition process is presented in Fig. 2a, when the liquid tellurium is still seemed in the

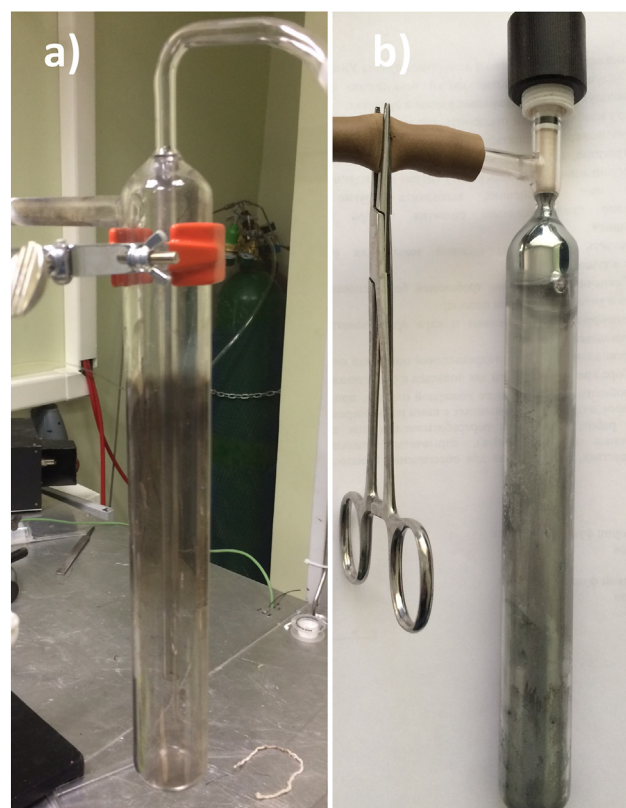


Fig. 2. The “telluride mirror” reaction in the walls of the trap walls.

bottom of the trap. Fig. 2a portrays the final state of the ampoule with the deposited “telluride mirror” on the walls.

The output of tellurium hydride and purified tellurium was determined by gravimetric method with accuracy of 1×10^{-4} g. The elemental analysis of the precipitated tellurium was carried out by atomic emission spectroscopy with DC arc discharge as a light source (the spectroscope with crossed dispersion STE-1). «Ex-situ» qualitative analysis of the major gas phase reaction products was carried out on GCMS – QP2010Plus (Shimadzu, Japan) with a vacuum sample inlet system through the automatic injection valve (Valco Instruments Co Inc, USA). The reaction products were separated on an Agilent capillary column Select for Permanent Gases/ CO_2 with the set of two parallel columns: CP-Molsieve 5 \AA for permanent gases and PoraBOND Q for CO_2 analysis in accordance with the following temperature program:

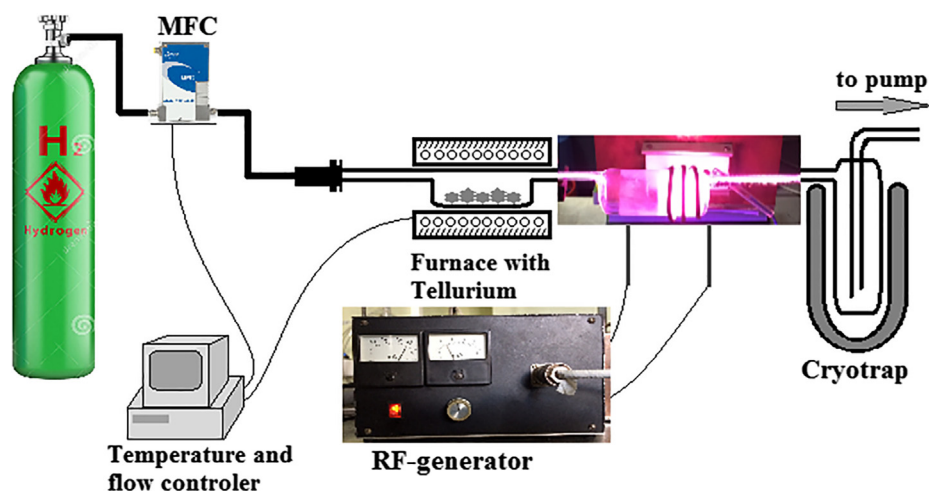


Fig. 1. Plasma-chemical setup for purification of H_2Te .

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