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Application of impedance spectroscopy to study oxidized powders of titanium hydride

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ABSTRACT

Results of studies of current-conducting properties of titanium hydride powders oxidized by air oxygen at different modes in order to form oxide and oxohydride coatings at the particles surface which enhance heat resistance of the hydride are presented. Method of impedance spectroscopy is used to determine contribution of resistance of the formed coatings and contribution of resistance of the grains of titanium hydride powders into the powder impedance. Method aspects of conducting measurements of the impedance of powder materials by stepwise sample compression are discussed. General approach to experimental data processing related to study of metal hydrides which contain shielding films of various chemical compositions at the particle surface is described.

1. Introduction

It is known that a resistometric research method is one of the most available and sensitive methods for studying the condition of the particle contact surface in powder materials (see, for example, [1–9]). This method is based on processing of experimentally determined electrical resistance of powder material measured under direct current as a function of its density [2–9]. The above dependence is determined in the process of stepwise powder compression; its pattern is similar in each case and is described by equation set forth in [3–6].

As shown, for instance, in works [3–9] carried out at RFNC-VNIIEF by the team headed by Mokrushin, application of a resistometric method enables to solve a wide range of fundamental and applied problems including those related to study of metal hydrides. In particular, this method permitted to uniquely define optimum conditions for vacuum annealing of titanium [4] and zirconium [7] powders aiming at their transfer in an active state relative to hydrogen; to define the most appropriate conditions for oxidation of titanium hydride powder in order to form the tightest shielding coating at its surface [8], and other.

Improvements of a resistometric method also led to rapid development of the method of impedance spectroscopy of powder materials which is based on measurements of powder electric impedance under alternate current. This research method is widely used, primarily, in electrochemistry [10–12]. However, its application for studying physical and chemical properties of powders has been quite limited so far due the lack of generally recognized approaches for conducting such measurements.

In general, impedance spectroscopy is based on taking impedance time curve of a system under investigation in a wide range of AC frequencies; on selection of an equivalent electrical circuit which adequately describes this system and on measurements of numerical values of its parameters [10–12]. Therefore, apparent advantages of impedance spectroscopy incorporate feasibility of experimental determination not only of the impedance of the entire system but also the resistance of its individual components. As applied to powder materials it permits to directly measure resistance of interparticle contacts as well as resistance of grains of powder particles [9,13–15].

Objective of this work lies in applying an impedance spectroscopy method for determining contribution of resistance of oxide and oxohydride coatings at particle surfaces as well as resistance of particle grains into impedance of pre-oxidized titanium hydride powders.

2. Materials and methods

Similar to resistometric method [2–9], a methodological approach to conducting impedance measurements is based on a stepwise powder material compression with simultaneous taking of time curves at each fixed sample density value. In this case, a set of impedance time curves taken at different degrees of compaction will be a result of powder material impedance measurements [9,15]. At that, configuration of these time curves is individual in each specific case and is determined by the properties of a test specimen. General approaches for processing and interpretation of experimental data acquired during powder material impedance measurements by the method under discussion are indicated in [14,15].

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Measurements of impedance in this work were performed using a tailored contact device with a measuring cell similar to one described in [16] which enabled to work with powders in inert environment. A powder sample (~0.5 g) was placed inside a measuring cell of a contact device where it was subject to stepwise compaction with simultaneous taking of impedance time curves using a computer-aided system consisting of an electrochemical interface and a frequency analyzer. Electrical parameters were controlled and recorded by a multi-channel AD converter and information was downloaded into PC. A basic diagram of a measuring system is shown in Fig. 1. In taking impedance time curves in this work AC frequency range varied from 0.1 Hz up to 1 MHz and measuring signal amplitude was 300 mV.

Commercial titanium hydride powder (GT brand) made of a titanium sponge served as a subject of inquiry. Average size of powder particles measured by optical microscopy was ~7 μm: specific gas content in initial hydride – 442.7 cm^3/g. The specific gas content was determined volumetrically by measuring the volume of hydrogen released from titanium hydride after its complete decomposition as a result of alloying with copper at 1100 °C. Separate samples of parent powder were preliminary oxidized by air oxygen to form oxide and oxohydride coatings of various thickness at the particle surface [17–19]. Oxidation was carried out by heating of powder in the air atmosphere within 1 h at different temperatures varying in the range of 440 °C up to 540 °C thus enabling to escape significant hydrogen losses [8,17,18]. Preliminary thermal treatment permitted to produce several batches of powders which differed in thicknesses of inhibitive coating at titanium hydride particles. More experimental details are given in [8].

3. Results

Measurements of impedance of separate batches of titanium hydride powders oxidized at different temperatures were performed. As an example, Fig. 2 presents impedance time curves taken for powders at various densities that were oxidized at temperatures 460 °C (a) and 520 °C (b) respectively. Similar shape of time curves is characteristic for all other batches of studied materials.

It is evident from Fig. 2 that impedance time curves of the powders under investigation have a form of semi-circumferences which intercept at *X*-axis. Regular decrease of semi-circumference

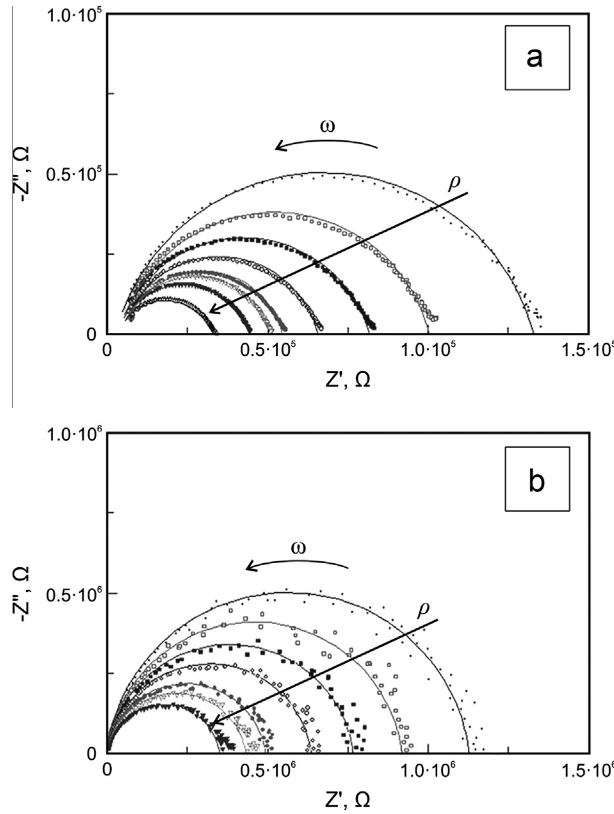

Fig. 2. Time curves of impedance of titanium hydride powders oxidized at 460 °C (a) and 520 °C (b) within 1 h; the arrows show directions of AC frequency (ρ) and powder compression density growth (ω).

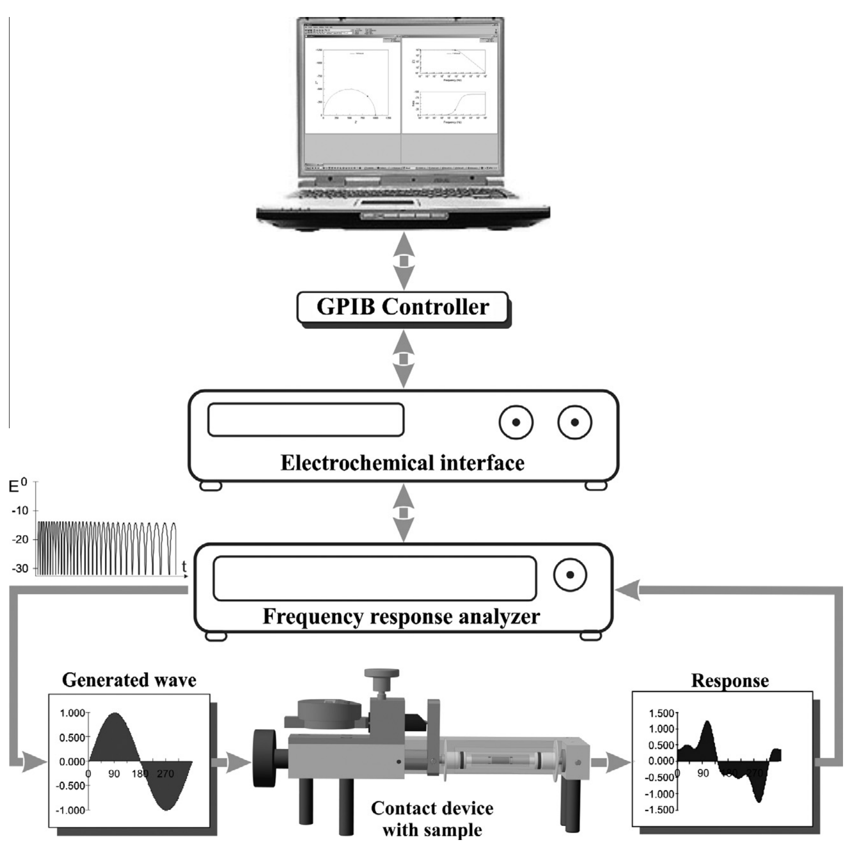

Fig. 1. Basic diagram of a system for measurements of powder materials impedance.