# DEVELOPMENT OF METHOD FOR PREPARATION CALIBRATION SAMPLES OF TITANIUM WITH HYDROGEN FOR GD-OES

Natalia Pushilina[1], Victor Kudiiarov[1], Andrey Lider[1], Patrick Chapon[2]

pushilina@tpu.ru, lider@tpu.ru, viktor.kudiiarov@gmail.com, patrick.chapon@horiba.com

[1]National Research Tomsk Polytechnic University, Lenin Avenue, 30, Tomsk, Russia

[2]HORIBA Jobin Yvon S.A.S. 16-18, rue du Canal - 91165 Longjumeau cedex -France

Currently there are not enough of calibration samples with certified values of hydrogen, and commercially available calibration samples are not suitable for the calibration of glow discharge optical emission spectrometers (GD-OES), as it does not fulfill the necessary requirements for the shape and dimensions. Preparation of samples with various geometrical dimensions and with a wide range of hydrogen concentration is possible using method of hydrogen saturation at gas atmosphere at high temperatures and pressures. The aim of this work is development of method for preparation calibration samples of titanium with hydrogen for glow discharge optical emission spectrometry. The method was implemented for commercially pure titanium alloy, but it can be suitable for calibration samples preparation of different titanium and zirconium alloys.

## Materials and Research Methods

The cylindrical samples of commercially pure titanium alloy with sizes 20x5 mm were prepared for investigation. The samples surface was mechanically grinded and polished. Then vacuum annealing was carried out at temperature 750 °C during 60 minutes with subsequent slow cooling in vacuum. Hydrogenation at gas atmosphere was carried out using automated complex Gas Reaction Controller LP at temperature 500 °C and pressure 2 atm. Hydrogenation was performed in an automated mode until hydrogen concentration reached values 500, 1500 and 2500 ppm. The evolution of the diffusion coefficient of hydrogen as a function of the temperature is governed by an Arrhenius-type equation. Using the material's constants values proposed in the literature for titanium: $D_0$ = 5,5·10$^{-3}$ sm$^2$/s, Q = 42950 J/mol. This diffusion coefficient was used to compute the numerical hydrogen distributions. So annealing in a helium environment at a temperature 500 °C for 90 minutes was carried out by melting in an inert gas using a hydrogen analyzer RHEN602 by LECO. Figure 1 shows the samples geometry and extracted samples for hydrogen measurements by RHEN602.

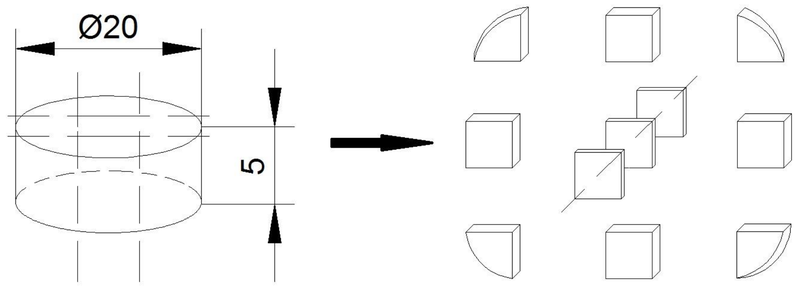


Fig. 1 – The samples geometry and extracted samples for hydrogen measurements by RHEN602

Table 1 – Hydrogen concentration in samples, ppm

| Hydrogen concentration in samples, ppm | | | | | |
|---|---|---|---|---|---|
| 1 | | 2 | | 3 | |
| 513 | 520 | 1533 | 1495 | 2603 | 2559 |
| 475 | 480 | 1492 | 1489 | 2534 | 2512 |
| 537 | 509 | 1493 | 1547 | 2566 | 2589 |

Figure 3 shows the calibration curves for hydrogen at the experimental conditions mentioned above.

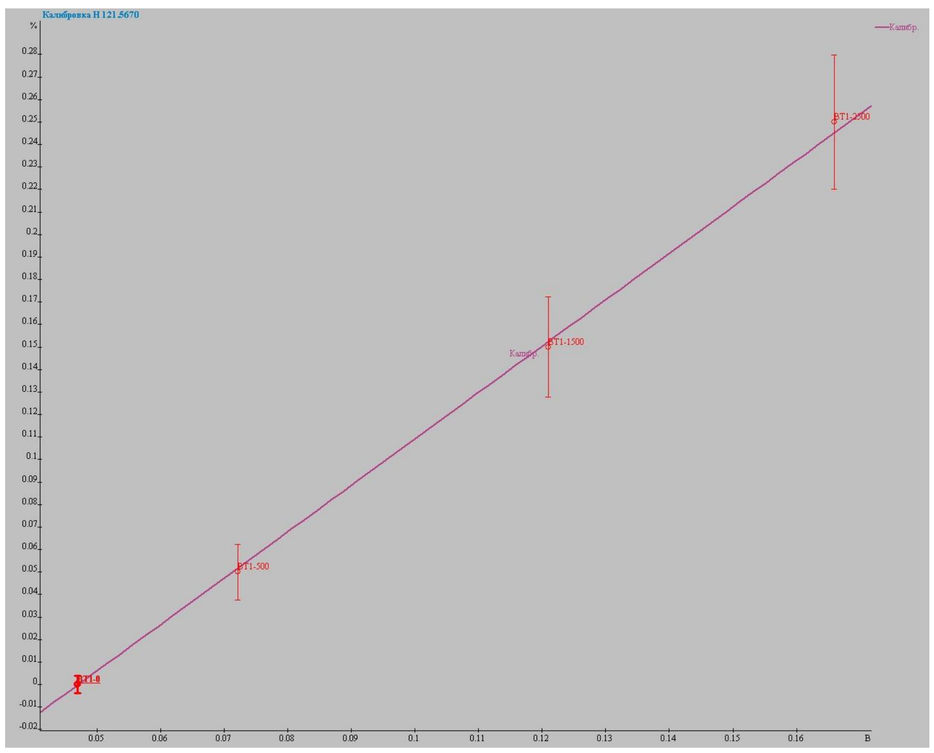


Fig. 3 – Calibration curve for H

High quality GD analysis of samples mostly depends on the depth resolution and, therefore, on the experimental conditions selected for the analysis. Experimental conditions of 700 Pa and 50 W were found to be a good compromise for the analysis of samples under study.

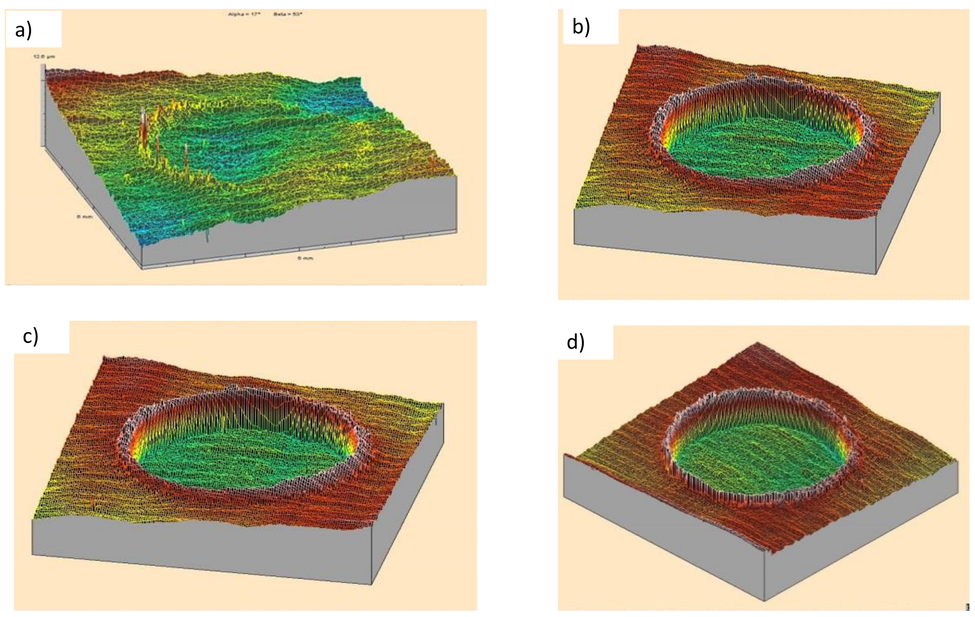


Fig. 2 – 3D-profiles of craters on titanium alloy surface depending on gas pressure a) 20 W; b) 30 W c) 40 W; d) 45 W

The method of hydrogen saturation at gas atmosphere at high temperatures and pressures can be used for preparation calibration samples of titanium with hydrogen for glow discharge optical emission spectrometry.

## The promising ways for improvement calibration accuracy:

- The development of new special sample chamber.
- The using of nitrogen eliminator.