NUCLEAR METHOD FOR DETERMINING HYDROGEN IN METALS AT THE SKIF FACILITY

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INTRODUCTION

Determining the concentration of gas impurities accumulated in the materials of nuclear reactors is of great scientific and applied importance. To conduct experimental studies in this area, the accelerator facility "SKIF" was manufactured at NSC KIPT.

The SKIF facility (Fig. 1) allows the following experiments to be carried out:

1. Studying the introduction and thermal desorption of gas ions from metallic and non-metallic samples in the temperature range of 77...1800 K. In this case, the targets irradiated in chamber I to a given dose are heated with an approximately linear law of temperature increase. The heating rate can be changed within the range from 0.5 to 10 $K \cdot c^{-1}$.

2. Monitoring the growth of deuterium concentration in the samples was performed using the nuclear reactions method (d – d).

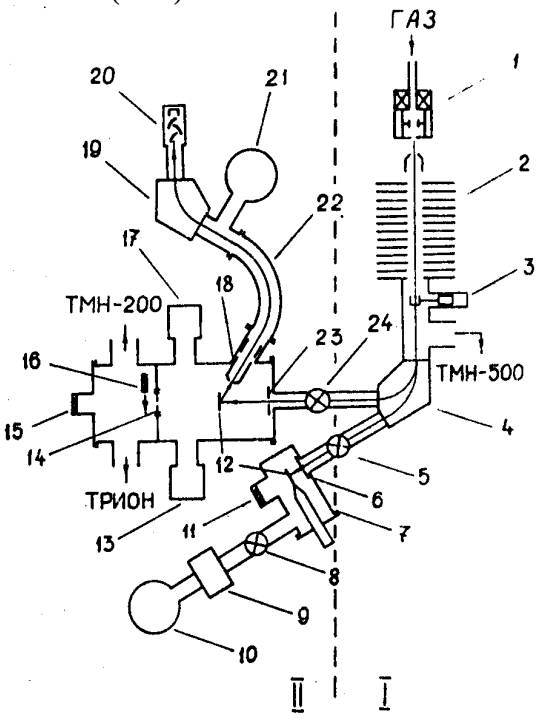

Fig. 1. Schematic diagram of the SKIF setup: I – accelerator section; II – output device section. 1 – ion source; 2 – electrostatic accelerating tube; 3 – Faraday cup with electromagnetic drive; 4 – mass separator; 5,24 – valves; 6 diaphragm; 7 – chamber 2; 8 – DU-50 valve; 9 – nitrogen trap; 10, 21 – NMD-0.025 magnetic discharge pumps; 11, 15 – windows; 12 – target; 13 – APDM-1 mass spectrometer sensor; 14 – iris diaphragm; 16 – quartz screen; 17 – MX-7304 mass spectrometer sensor; 18 – extraction lens of the secondary ion mass spectrometric analyzer; 19 – mass separator; 20 – VEU; 22 – energy analyzer; 23 – chamber 1

TDS METHODOLOGY

When solving a number of problems in solid state physics using thermal desorption spectrometry, it becomes necessary to irradiate the samples under study with ion or electron beams at liquid nitrogen boiling temperatures (~78 K), and also to smoothly change the sample temperature during or after irradiation in the maximum possible range of values. Using the thermal desorption technique together with electron diffraction studies of ion-induced phases of the metal-gas system allows identifying peaks of gas evolution with the breakup of the corresponding phases and, accordingly, establishing the thermodynamic parameters of the breakup of the phase states of the metal-gas system (temperature ranges, activation energy, reaction order).

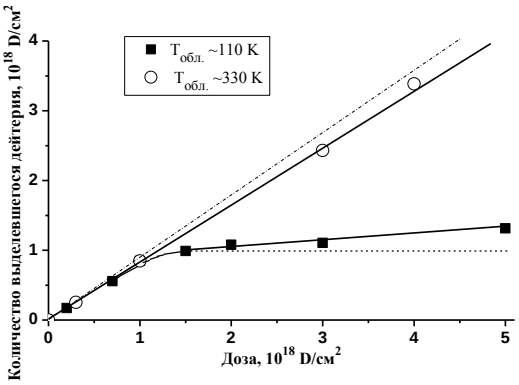

Fig. 2. Dependence of the total amount of desorbed deuterium on the irradiation dose for titanium implanted with deuterium at temperatures of ~ 110 and ~ 330 K

Dependence of the total amount of desorbed deuterium on the irradiation dose for titanium implanted with deuterium at temperatures of ~110 K (–■–) and ~330 K (–○–), -- 100% capture of implanted deuterium at a temperature of ~110 K, - - -hypothetical saturation level for a zirconium sample in the case of saturation of a smaller sample, for example, a zirconium film. In the case of target irradiation at room temperature, the linear dependence of the amount of implanted deuterium on the implantation dose is maintained over the entire studied dose range from $7x10^{15}$ to $7x10^{18}$ D/cm^2. The deuterium capture coefficient is ~95%. This behavior of the dependence indicates the absence of any signs of metal saturation with deuterium at a temperature of $T_{irradiation}$~330 K. The absence of saturation in our case at an irradiation dose exceeding the dose by 3 times can be explained only by the fact that at $T_{irradiation}$~330 K, deuterium in the metal has a sufficiently high diffusion mobility and, as a result, "runs away" in the sample, which has a large capacity, without leading to a noticeable increase in concentration in the region of ion beam deceleration. In connection with the above, we came to the

conclusion that at room temperature, it is practically impossible to achieve a concentration of metal saturation with deuterium in a massive sample in a reasonable experimental time. To estimate the concentration of deuterium in the implantation layer of the metal, the nuclear reactions (d-d) method was used.

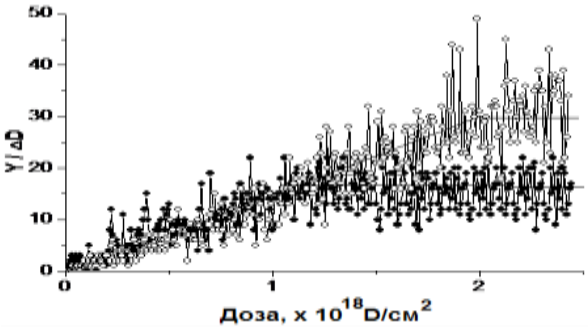

Fig. 3. Dependence of the total amount of desorbed deuterium on the irradiation dose for titanium implanted with deuterium at temperatures of ~110 K (–■–) and ~330 K (–○–), -- 100% capture of implanted deuterium at a temperature of ~110 K, - - - hypothetical saturation level for a zirconium sample in the case of saturation of a smaller volume sample, for example, a zirconium film

NUCLEAR TECHNIQUE (d-d)

The growth of deuterium concentration in the sample is monitored by plotting the dependence of Y/ΔD on the dose, where Y is the number of particles of a given type (synthesis products) emitted from the target surface during irradiation and recorded by the detector over a period of time Δt, ΔD is the dose increase over the same period of time, and D is the irradiation dose. The relationship between Y/ΔD and D can be obtained from the equality

$$\frac{Y}{\eta \cdot S \cdot \Delta t} = \sigma_i \cdot l_{eff} \cdot n_t \cdot \frac{\Delta N}{S \cdot \Delta t} \ ,$$

where η – is the efficiency of particle counting by the detector; S – is the area of the irradiated surface and, accordingly, the area from which the reaction products are emitted; σ_I – is the cross-section for the i-th synthesis channel; l_{eff} - is the effective thickness of the target – the implanted layer in which the reactions occur.

The nuclear reaction method was used to estimate the deuterium concentration in the titanium implantation layer. The energy spectra of charged particles obtained during the irradiation of titanium samples with D ions showed that the deuterium concentration in the titanium sample increases until a dose of $2x10^{18}$ D/cm^2 is reached and then saturation occurs.

The data obtained indicate that deuterium initially accumulates in the deposition profile to a certain concentration and the subsequent implantation of deuterium is accompanied by the diffusion of deuterium from the implantation layer deep into the sample.

CONCLUSIONS

A comprehensive methodology for experimental study of the behavior of ion-implanted deuterium in metals has been developed, based on mass-spectrometric registration of the implanted deuterium released during linear and isochronous heating of the target and control of the amount of deuterium accumulated in the bedding profile. The data obtained indicate that deuterium accumulates in the bedding profile to a certain concentration and subsequent implantation of deuterium is accompanied by diffusion of deuterium from the implantation layer deep into the sample.

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