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An Integrated Approach to the use of Ion Implantation (ii), Thermal Desorption Spectroscopy (tds), Nuclear Reactions (d-d) Methods for the Determination of Hydrogen Concentration in Metals using the Example of a System Zirconium-Deuterium (Zr-D)

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Abstract

To determine certain characteristics of hydrogen in metals (concentration, depth distribution, and location in the crystal lattice), we propose using a combined approach, including thermal desorp-tion spectroscopy (tds), nuclear reaction analysis (d-d), ion implantation (ii).

An integrated approach to the use of ion implantation (ii), thermal desorption spectroscopy (tds), nuclear reactions (d-d) methods for the determination of hydrogen concentration in metals using the example of a system Zirconium-Deuterium (Zr-D).

To determine certain characteristics of hydrogen in metals (concentration, depth distribution, lo-cation in the crystal lattice), it is proposed to use a combined approach, including thermal desorp-tion spectroscopy (tds) and nuclear reaction (d-d) analysis.

The combined use of these research methods allows us to determine both the total hydrogen con-tent and the nature of its retention in traps, allowing for a more complete description of the pro-cesses of hydrogen dissolution, diffusion, and degassing in metals.

Application ion implantation (ii), thermal desorption spectroscopy (tds), nuclear reactions (d-d) methods for the determination of concentration hydrogen in metals using the zirconium-deuterium (Zr-D) system as an example.

The combined use of these research methods allows us to determine both the total hydrogen con-tent and the nature of its retention in traps, enabling a more complete description of the processes of hydrogen dissolu-tion, diffusion, and degassing in metals.

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Background

The interaction of hydrogen with metals is one of the most fascinating topics in modern metallurgy and solid-state physics. This is due to the unusual behavior of this element in condensed phases and the practical need to explain the long-known strong negative effect of hydrogen on the properties of most metals and alloys.

Currently, laboratories are conducting hydrogen-related research in several areas: - the production of lightweight porous materials – gasars – and the study of their properties for subsequent indus-trial implementation.

When hydrogen dissolves in metal during a polymorphic transformation, it can form layers with a high hydrogen content (up to tens of atomic percent) (H-layers), which dramatically alter the properties of metals. It has been established that the H-layer leads to a sharp change in the physical and mechanical properties of polymorphic metals (a decrease of 3-4 orders of magnitude due to a reduction in metal-to-metal interatomic bonds) during the polymorphic transformation, causing their spontaneous deformation. Iron and manganese do not change their physical and me-chanical properties after treatment in a hydrogen atmosphere, while hydride-forming metals (Ti, Zr) increase their brittleness due to the formation of hydrides in their structure.

Thermal cycling of metals in hydrogen significantly coarsens their grain size, creating a cast-like structure with a wide grain size distribution. This process occurs due to the interaction of the H-layer with structural defects in the metal (vacancies, dislocations). Structure purification hinders the nucleation of a new α-phase. The movement of H-layers in metals leads to the formation of micro- and macro pores within them due to the transfer of structural defects into the interior of the samples.It is of significant scientific interest to use several methods; we are attempting to implement this approach using our Skif facility [1].

To determine certain characteristics of hydrogen in metals (concentration, depth distribution, localization in the crystal lattice), we propose using a combined approach, including thermal desorption spectroscopy (tds), nuclear reaction analysis (d-d), and ion implantation (ii).

Methods

The ion implantation method (ii) allows implantation/ introduction of a given amount of ions of both hydrogen and deuterium in the sample for research.

The thermal desorption spectroscopy (tds) method (tds spectrum) allows one to determine the temperature ranges of hydrogen and deuterium desorption in the test sample and the amount of deuterium released. The presence of two or more peaks in the thermal desorption spectrum indi-cates the presence of different mechanisms of deuterium retention in the sample. By comparing the areas of the thermal desorption spectra obtained by heating similar samples at the same heat-ing rate, pre-irradiated with different doses of deuterium ions at the same temperature, we obtain the dose dependence of deuterium retention (accumulation) in the sample area. Similarly, by comparing the areas of the TDS spectra of deuterium after irradiation with the same doses of deuterium ions, but at different temperatures of the irradiated samples, we obtain the depend-ence of the amount of retained deuterium on the sample temperature during irradiation. [2-3].

Irradiating samples with deuterium ions (instead of hydrogen ions) helps eliminate the influence of background hydrogen on the thermal desorption spectrum. Irradiating samples with deu-terium ions also enables the use of nuclear physics (d-d) techniques, which allow us to determine the dependence of deuterium concentration in the sample's depth profile on the deuterium ion irradiation dose by recording nuclear d-d reactions.The combined use of these two research methods allows us to determine both the total hydrogen content and the deuterium distribution profile across the

sample depth, enabling a more complete description of the processes of hydrogen dissolution, diffusion, and degassing in metals.

The use of the nuclear reaction method (d-d) results in the emission of secondary particles (protons, neutrons, and alpha particles—the products of nuclear reactions). By recording the energy and number of secondary particles, the deuterium concentration in the implantation layer is determined and its distribution profile across the sample depth is determined.

The combined use of these research methods allows us to determine both the total hydrogen con-tent and the deuterium distribution profile across the sample's depth, enabling a more complete description of hydrogen dissolution, diffusion, and degassing processes in metals.

Nuclear reaction method (d-d)

The use of the nuclear reaction method (d-d) results in the emission of secondary particles (pro-tons, neutrons, alpha particles—products of nuclear reactions). By recording the energy and quantity of secondary particles, the deuterium concentration in the implantation layer is deter-mined and its depth profile is determined [4-6].

Results and Discussion

Application of the thermal desorption spectrometry (tds) method

Studies of the thermal desorption of deuterium from zirconium were conducted taking into ac-count the deuterium irradiation dose and sample temperature ~100 K. The dependence of the amount of released deuterium on the dose of implanted deuterium was plotted (Figure 1).

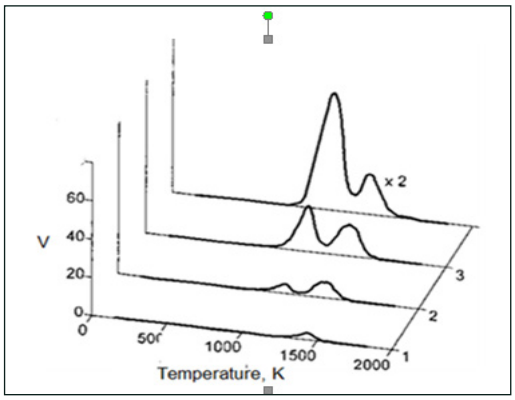

Figure 1: Thermal Desorption Spectra of Deuterium Implanted into Zirconiu.

$T_{irr.}$ ~ 100 K for different doses: (1) – $7x10^{16}$ cm^{-2}, (2) –$3x10^{17}$ cm^{-2}; (3) – $1x10^{18}$ cm^{-2}; (4) – $4x10^{18}$ cm^{-2}.

Based on the analysis of experimentally measured deuterium thermal desorption spectra from zir-conium, Arrhenius polytherms were constructed and thermal desorption activation energies were determined for peaks with maximum temperatures of ~1030 K and ~1350 K. [Arrhenius poly-therms, These values were found to be ~3.1 eV (Figure 2) and ~1.2 eV (Figure 3) for peaks with maximum temperatures of ~1350 K and ~1030 K, respectively. The reaction rate order γ=1 [7]. The activation energies for deuterium thermal desorption from zirconium were obtained using the well-known desorption kinetics equation spectrum from deuterium-implanted zirconium at Tirr ~100 K with a dose of ~6×10^{16} D/cm^2 (peak with a maximum temperature of ~1360 K.

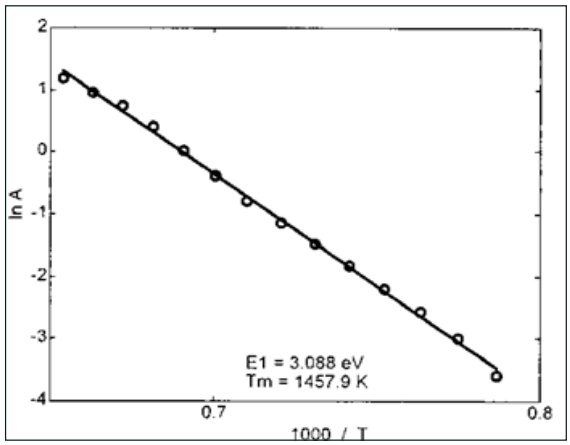

Figure 2: Dependence of Ln[Dn_i /Ddt/n] on the Reciprocal Temperature for the Peak of the Deuterium Thermal Desorption (peak with a maximum temperature of ~1460 K).

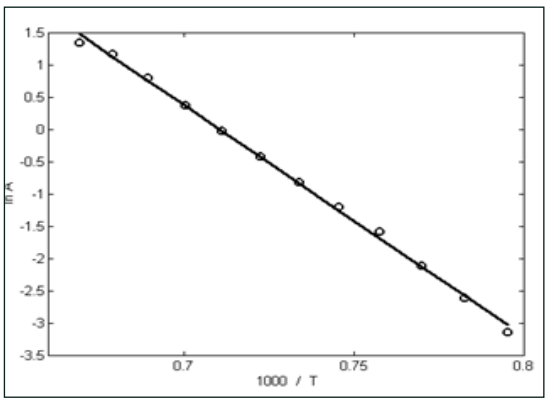

Figure 3: Dependence of ln[dn_i /dT/n] on the reciprocal temperature for the peak of the deuterium thermal

desorption spectrum from deuterium-implanted zirconium at T_{irr} ~ 100 K with a dose of ~ 7×10^{16} D/cm^2 (peak with a maximum temperature of ~1360 K).

The spectra presented in Figure 1 show that the change in the thermal desorption spectrum with increasing irradiation dose is in good agreement with the concepts of the emergence of a two-phase system (α- and β-) in the zirconium-deuterium system [8-10].

The TDS method revealed the presence of several peaks in the spectrum. Analysis of the spectra revealed that most of the observed peaks were due to the decomposition of hydrides formed dur-ing the deuterium implantation process. Further analysis of the observed peaks in the spectrum determined the temperature ranges of both the formation and decomposition of the hydride phases, as well as the chemical composition of the hydride phases.

Estimation of Deuterium Concentration in the Zirconium Implantation layer

When irradiating a massive zirconium target at room temperature, the linear relationship between the amount of implanted deuterium and the implantation dose is maintained over the entire stud-ied dose range from ~7×10^{15} D/cm^2 to ~7×10^{18} D/cm^2. The deuterium capture efficiency is **~95%.**

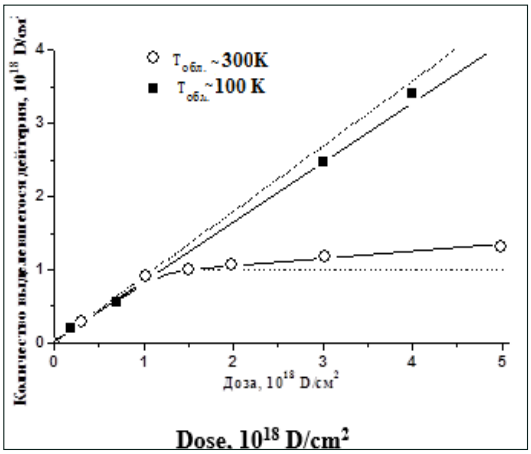

Figure 4: Dependence of the Total Amount of Desorbed Deuterium on the Irradiation Dose for Zirconium Implanted with Deuterium at Temperatures of ~100 K (–■–) And ~300 K (–O–), - --- 100% Capture Of Implanted Deuterium at A Temperature Of ~100 K, - - - Hypothetical Saturation Level for a Zirconium Sample in the Case of Saturation of a Smaller Sample, for Example, a Zirconium Film.

The lack of deuterium saturation in the sample, in our case, can be explained by the fact that at T_{ir}~ 300 K, deuterium in zirconium has a fairly high diffusion mobility and, as a result, is "absorbed" in the sample, which has a high capacity, without leading to a noticeable increase in concentration in the ion beam deceleration region. This behavior of the dependence indicates the absence of any signs of deuterium saturation in zirconium. In Wert C. A. Hydrogen in metals. II: Topics in applied physics [11]. In this regard, we determined that at room temperature it is practi-cally impossible to achieve the saturation concentration of zirconium with deuterium in a massive sample in a reasonable experimental time, **or the TDS method does not register the true value of the deuterium concentration in the implantation profile.**

To estimate the actual deuterium concentration in the zirconium implantation layer at tempera-tures of 100 K and 300 K, the d-d nuclear reaction method was used. The energy spectra of charged particles obtained during irradiation of zirconium samples with D ions showed that the deuterium concentration in the zirconium sample at T~100 K increases until a dose of ~2×10^{18} D/cm^2 is reached, and at a sample temperature of 300 K until a dose of 1.2×10^{18} D/cm^2 is reached. At a sample temperature of 300 K and further, the signal reaches saturation (Figure 5).

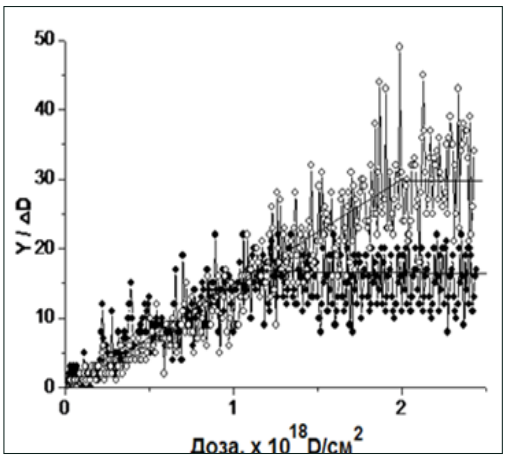

Figure 5: Energy Spectra of Charged Particles Obtained During Irradiation of Zirconium Samples With D Ions (--O--- T=100 K; --■---T=300 K)

To estimate the actual deuterium concentration in the zirconium implantation layer at tempera-tures of 100 K and 300 K, the nuclear reaction method (d-d) was used. Energy spectra of charged particles obtained during D-ion irradiation of zirconium samples showed that

the deuter-ium concentration in the zirconium sample increases at T~100 K until a dose of ~$2x10^{18}$ D/cm^2 is reached, and at a sample temperature of 300 K until a dose of $1.2x10^{18}$ D/cm^2 is reached at a sample temperature of 300 K and beyond, the signal reaches saturation (Figure 5).

The obtained data show that the accumulation of deuterium occurs in the deposition profile be-fore the occurrence of consequences and, upon subsequent implantation of deuterium, is accom-panied by the diffusion of deuterium from the implantation structure of the layer. (This is evi-denced by the saturation of the dependencies shown in Figure 5.

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