

COMPLEX INVESTIGATION OF GAS DIFFUSION PROCESSES IN VACUUM TECHNOLOGY MATERIALS

1. Automatically-Controlled Plant for Study of Hydrogen Permeability of Metals With Simultaneous Diagnostics of Membrane Material

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1. INTRODUCTION

From the standpoint of vacuum technology, the hydrogen-metal system is of special interest, since the presence of hydrogen in a solid and on its surface can radically change the physico-chemical characteristics of vacuum equipment operating elements.

In modern practice the permeability method is a basic method for studying the processes of hydrogen-metal interaction in the course of hydrogen diffusion through thin membranes.

In the course of hydrogen permeability in solids the high chemical activity of hydrogen brings about the occurrence of different-type diffusion anomalies associated with the interaction between hydrogen and impurities or defects: deformation of kinetic diffusion curves, occurrence of breaks on the diffusion- or solubility-coefficient versus temperature curves plotted in the Arrhenius coordinates, appearance of effective diffusion coefficient dependence on partial hydrogen pressure at the membrane inlet or on the membrane thickness, etc. The interpretation of such anomalies is impossible without using different methods for studying the surface and volumetric properties of the membrane material and their changes in the course of diffusion.

The present paper reports on the development of complex equipment using in combination diffusion diagnostics, nuclear-physical and physico-chemical methods of material diagnostics.

2. APPLICATION, COMPLETE SET AND BASIC TECHNICAL DATA OF THE AUTOMATICALLY CONTROLLED PLANT FOR THE COMPLEX INVESTIGATION OF DIFFUSION PROCESSES

An experimental flexible complex of the DAM-1 model has been designed for studying the kinetics of gas diffusion and dissolution in various kinds materials under thermal, radiation, mechanical and chemical effects on the sample, as well as for studying the changes in the elemental and chemical compositions and in the structure of the material by electron probing techniques and different methods of gamma-ray resonance spectroscopy.

This DAM-1 automatically-controlled plant can be applied to various objectives: the determination of fundamental parameters of gas-solid interaction and transport parameters of gases in different materials; the study of membrane catalysis processes; adjustment and prediction of gas-separation filter service life; the development of hydrogen brittleness control methods; the development of methods for detecting the weld discontinuity; the search for selective membranes for the liberation, purification and concentration of hydrogen; the finding of materials for the first wall of thermonuclear reactors and for hydrogen transport and storage systems; the providing of optimum operating conditions for the degassing of vacuum technology materials; the prediction of

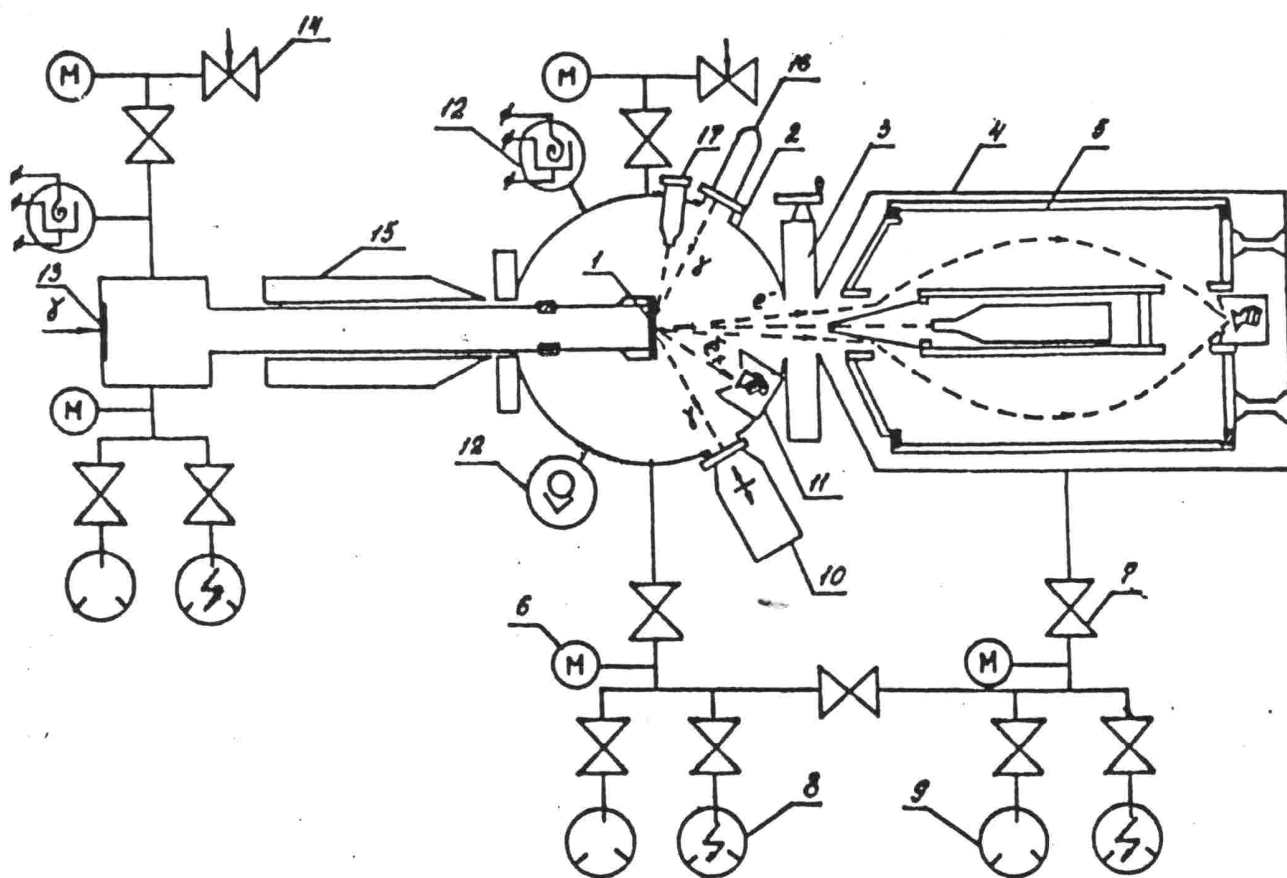


Figure 1. Block diagram of an experimental plant. 1—studied sample, 2—research chamber, 3—free-flow valve, 4—Auger spectrometer chamber, 5—energy analyzer, 6—vacuum gauges 7—vacuum valves, 8—sputter ion pumps, 9—adsorption pumps, 10—vibrator, 11—electron detector, 12—partial pressure, 13—beryllium window, 14—dosing valves, 15—manipulator, 16—gamma-radiation detector, 17—ion gun.

structural material capabilities in the hydrogenous medium; the preparation of materials with chemically clean surfaces; and the measurement of isotope effects, etc.

The present automatically controlled plant is universal, can be easily rearranged, permits change over from one method to another without any readjustment of the equipment, to use several methods simultaneously by the implementation of the software in real time.

The experimental complex makes it possible:

1) to measure the kinetics of the diffusion of stable or radioactive gases or gas mixtures through various materials (metals, ceramics, glass), using different diffusion methods: permeability methods, sorption and thermodesorption methods, frequency probing methods and others;

2) to study the diffusion under thermal effects (temperature range from 300 to 1200 K), chemical effects (treatment of the sample with chemically active gases, conducting catalytic reactions on one or both sides of the membrane), and under radio-

active actions on the sample (electron-radiation, gamma-radiation, x-radiation);

3) to study the material surface state and structure by diffusion gas probe methods: tritium labelling, stable and radioactive inert gas probes, including thermodesorption spectroscopy, emanation thermoanalysis;

4) to carry out elemental analysis of the outlet side surface of the membrane by Auger electron spectroscopy with layer-by-layer ionic etching of the sample

5) to perform an analysis of the surface layers and volume of the metal by methods of nuclear gamma-ray resonance spectroscopy of conversion electrons;

6) to study the pattern for diffusion radioactive gas distribution over the surface and in the volume of the metal by autoradiography methods.

Figure 1 shows a block-diagram of the experimental diffusion complex. The automatically controlled plant consists of:

- a diffusion cell divided into three sections by a free-flow valve and the membrane being studied;
- a oil-free evacuation system comprising of three independent vacuum units and providing ultrahigh vacuum in every section of the diffusion cell;
- a gas and gas mixture purification and bleeding-in system (provision is made to bleed-in the gas on the inlet or outlet side surface of the membrane);
- a system of recording the changes in partial gas pressures, comprising two mass-spectrometers for the determination of light and heavy masses on the outlet side of the sample and a mass-spectrometer in the inlet chamber, which

- allows carrying out diffusion experiments under conditions of changing the direction of hydrogen flow through the membrane;
- ionizing radiation counter and radiation sensitive instrument set for studying the diffusion processes of radioactive gases (tritium included);
- Auger spectrometer employing an energy analyzer of the cylindrical-mirror type;
- Mossbauer spectrometer;
- a beryllium window for the introduction of intensive ionizing radiation into the diffusion chamber to study diffusion processes in the radiation field;
- devices for the deformation of the sample in the course of diffusion processes;

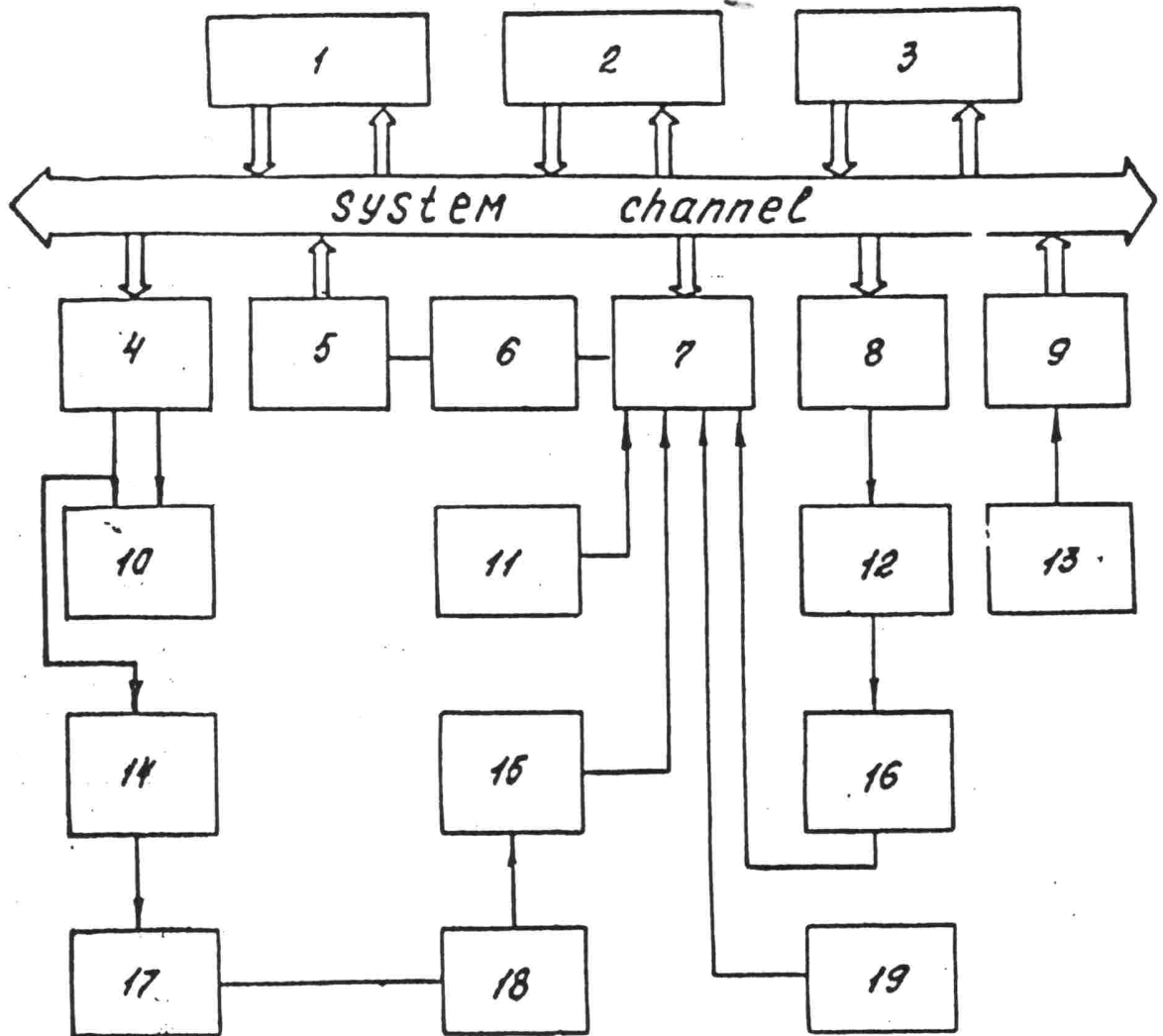


Figure 2. Automatic control block diagram. 1—DBK-3 computer; 2—alphanumeric display, 3—printer, 4—plotter coupling unit, 5—digital voltmeter (DV) coupling unit, 6—digital voltmeter (DV) 7—DV switch, 8—heating control unit, 9—timer, 10—plotter, 11—mass-spectrometer of inlet chamber, 12—heater; 13—frequency meter-generator, 14—AES sweep unit, 15—synchronous detector, 16—thermocouple signal amplifier, 17—analyzer of cylindrical-mirror type, 18—electronic multiplier, 19—mass-spectrometer

— a DVK-3 computer-based automatic control system providing for control of all the experiments, as well as the processing and interpretation of the results.

The automatic control block-diagram is given in Figure 2.

Materials being studied: metals, alloys, materials with coats and welds, ceramics, glasses.

Diffusants: hydrogen isotopes (protium, deuterium, tritium), air components, inert gases (stable and radioactive).

Temperature range	300–1200 K
Ultimate vacuum	10^{-8} Pa
Inlet gas pressure range	10^{-6} – 10^{-1} Pa
Partial hydrogen pressure sensitivity	10^{-10} Pa
Monatomic sensitivity for GRS	0.1%
Absolute sensitivity of AES	10^{-14} at. %
Relative sensitivity to impurity of volumetric origin for AES	10^{-3}
Limit of element detection by AES	10 10 atom/sq.cm

3. EQUIPMENT AND METHODS FOR MEASUREMENT OF HYDROGEN DIFFUSION IN METALS

The research chamber is a basic part of the experimental complex. It is provided with a window for the visual observation of the sample, with flanges for connecting the stable and radioactive gas transducers, a free-flow valve with a chamber for electron probing diagnostics of materials, pipes for supplying the gases being studied to the inlet and/or outlet side surfaces of the sample, and a beryllium window for the introduction of external ionizing radiation into the chamber.

A sample holder arranges the membrane in the center of the research chamber, opposite the free-flow valve.

A heater and a control thermocouple are fastened on the sample holder. The control thermocouple is in the center of the membrane. The heater control system makes it possible to carry out experiments at a constant temperature or in the programmed heating regime (linear, as a rule). The supply of liquid nitrogen vapor allows for carrying out the experiments at low temperatures. The sample holder is seated on a sealed transfer device which serves for focusing the Auger-spectrometer. The same transfer device can be used for the deformation of the sample.

The gas purification and bleeding-in system comprises a palladium-silver filter and a silver filter for bleeding in hydrogen and oxygen, respectively; a controllable piezoelectric-ceramic leak for bleeding any gases; a quartz capillary tube or a needle leak for helium bleeding-in.

The plant design permits conducting diffusion experiments by different methods.

The permeability method is based on the measurement of gas diffusion through a thin membrane [1]. At the initial moment the gas is fed into the vessel and the process of the gas passing into the receiver is studied. During the experiment the gas flow is recorded by the time variation of the partial gas pressure detected by the mass-spectrometer transducer. From the non-stationary portion of the curve (Figure 3), for instance by the time of the half value of the stationary flow (i.e., $J_{1/2} = 0.5 J_{\infty}$, where J_{∞} is the stationary flow value) we can determine the diffusion coefficient:

$$D = L^2/7.19 \tau_{1/2} \quad (1)$$

where L is the membrane thickness.

Find the permeability constant from the stationary flow formula:

$$P_H = J_{\infty} L / S P_0^{1/2} \quad (2)$$

where S is the membrane surface area, P_0 is the partial hydrogen pressure at the inlet to the membrane.

Then the solubility constant is

$$K_I = P_H / D \quad (3)$$

The afore-cited formula the estimation of the coefficients of diffusion, solubility and permeability were derived on the assumption of the classical diffusion mechanism described by a simplest form of the Fick law. The classical mechanism is realized very seldom in experiments for studying the hydrogen permeability kinetics, therefore the processing of the results was performed by the least-squares technique with the use of the DIGS program package [2].

In addition to the step-by-step supply of gas or gas mixture to the sample inlet, the equipment makes possible conducting the experiments under conditions of partial pressure variation at the membrane inlet in accordance with some prescribed rules [3]. The hydrogen concentration was fed as a square pulse of different height and width, as a pulse train (provision was made for changing the width of pulses, pulse spacing and pulse repetition frequency) or sine wave oscillations (provision was made for changing the amplitude and frequency of the concentration wave).

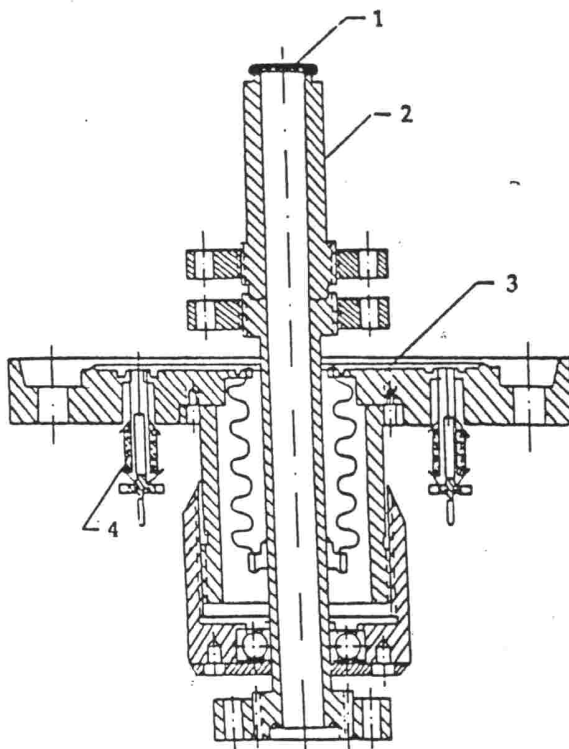


Figure 3. Diffusion Cell Construction
1-membrane, 2-Transfer device rod,
3-sample transfer device flange,
4-pressure-seal feed-through

The pulse variant of the permeability method was used to reduce the diffusion experiment time, and the gas mixture (or hydrogen isotopes) pulse was applied to improve the selectivity factor. The pulse train was employed to increase the dynamics of the permeability method, which made it possible to study phase transitions and relaxation processes occurring in the membrane matrix. The concentration wave method was used to prevent nonlinear effects (concentration dependence of the effective diffusion coefficient, abnormal form of the kinetic curve, etc.). Such effects occur in the presence of hydrogen capture by limited capacity defects or second-order chemical reactions on the membrane surfaces or in its volume.

The sorption method is based on the measurement of the kinetics of gas diffusion into the solid and makes possible determining coefficient and solubility constant [4]. During the experiment the gas was fed to the both sides of the membrane (there was not hydrogen pressure difference on the membrane) and the equilibrium state was awaited. To determine the absorbed gas quantity, the sample was heated to high temperatures and the liberated gas quantity was measured. The sorption experiments were carried out at successively increasing partial pressures of hydrogen, which

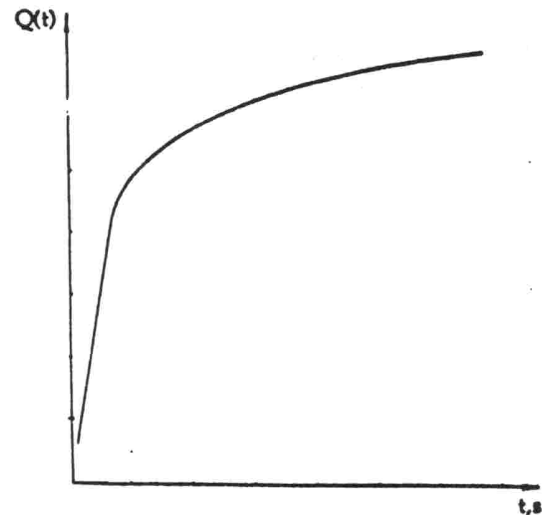


Figure 4. Kinetic curve of hydrogen penetration in nickel. ($T = 993 \text{ K}$, $L = 2.0 \text{ mm}$, $P_{\text{H}_2} = 10 \text{ Pa}$)

permitted obtaining the gas sorption isotherm to classify it (i.e., to determine whether it relates to the Henry isotherm or to the Langmuir one) and to calculate the sorption isotherm parameters.

The desorption method (DS) and the method of thermally stimulated gas liberation (TSGL) are based on studying the kinetics of gas liberation from the solid. The desorption study is carried out at a constant temperature, while the TSGL study is performed in the linear heating regime [5]. The liberated gas flow is monitored under continuous evacuation conditions during the experiment. The TSGL method allows for judging of the sample surface cleanliness, the presence, type and concentration of defects in the solid volume, and the presence and intensity of solid-phase processes in the sample under heating. Using the experimental data, spectra of the initial gas state and diffusion activation energies were produced [6,7].

The availability of stable- and radioactive-gas detectors makes it possible to use the DAM-1 complex for the determination of isotopic effects (in case hydrogen is employed—for the measurement of the diffusion coefficients of stable isotopes: protium, deuterium and the radioactive isotope—tritium) and their temperature dependences. Besides, radionuclides can be used for improving the recording sensitivity (tracer method regime) and for checking the pattern of hydrogen (tritium) distribution in the volume and over the surface of the sample during diffusion. In the latter case, the autoradiographic variant (AR) of the permeability method [8] is effective. Figure 4 shows an example of the distribution of the

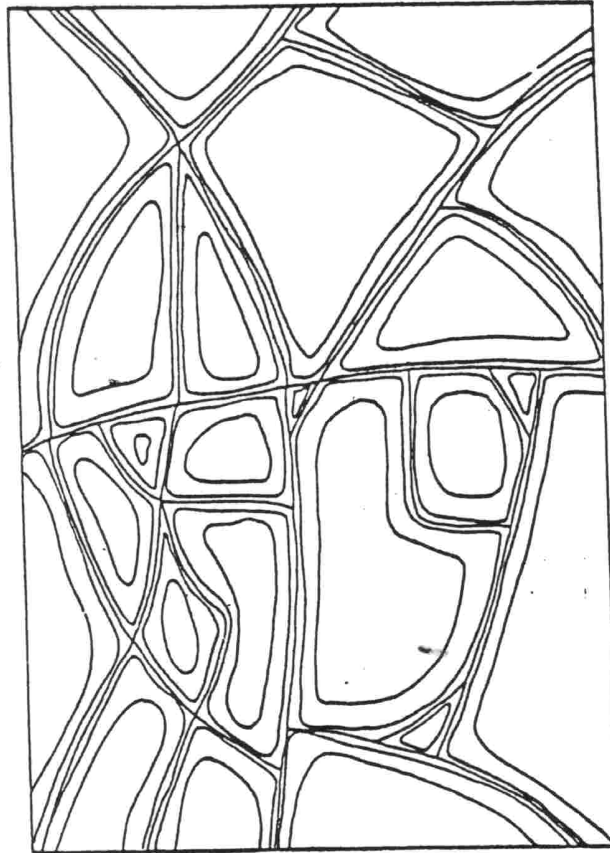


Figure 5. Tritium distribution over inlet surface of aluminium membrane by autoradiography data.

autoradiogram darkening density obtained in the study of the tritium concentration over the outlet side surface of an aluminium membrane. The distribution is seen to have a nonuniform pattern: tritium is concentrated at the grain boundaries. The sampling of the obtained distribution (Figure 5) permits quantitative assessment of the degree of distribution nonuniformity. A study of the pattern for hydrogen (tritium) distribution in the material volume is performed by the longitudinal shearing method and autoradiography technique.

4. EQUIPMENT AND METHODS FOR MEMBRANE MATERIAL DIAGNOSTICS DURING DIFFUSION

4.1 Gas Diffusion Probe Method

The gas diffusion probe method is based on the use of a specially selected gas the diffusion of which is sensitive to structural nonuniformities of the object being studied and to solid-phase processes occurring because of the internal instability of the substance or external effects on the sample

[9]. Tritium microquantities, periodically introduced into hydrogen at different stages of permeability, can be used as a probe detecting individual stages of the complicated hydrogen permeability process accompanied by different chemical reactions and sample structure changes.

From the standpoint of defectoscopy, tritium is an optimum probe, as it has small dimensions and diffuses quickly in metals by the interstitial-site mechanism. Therefore, inert gases are widely used in diffusion-structural analysis. The inert gas transport is not complicated by processes of the chemical interaction with the material under study, the molecules have one-atom structure and the atoms are spherically symmetric. Inert gases are practically insoluble in metals and hence are easily released from the metal lattice and condensed in micro- or macrodefects, which in turn facilitates solution of the defectoscopy problem. A set of inert gases allows for realizing various diffusion mechanisms. Thus, helium usually diffuses in metals by the interstitial-site mechanism, neon and argon by the dissociation mechanism (i.e., over interstitial sites and vacancies), krypton by the vacancy mechanism, while xenon and radon over

defect-clusters consisting of two or more vacancies. This permits changing the diagnostic sensitivity to different-type defects. Inasmuch as the presence of dislocations, microbubbles and grain boundaries exerts a great influence on the diffusion processes, macrodefects can also be easily detected when using inert gas diffusion probes.

When performing the material defectoscopy, inert gases were introduced into the membrane outlet surface by ion bombardment (in glow discharges, in high frequency Tesla discharges or by means of ion sources). Through changing the bombardment energy, the probe introduction depth was altered. The spectra of the thermally-stimulated gas liberation kinetics were taken in the linear heating regime. The TSGL spectra reflected the inert gas initial state spectra (and consequently the spectrum of defects), the diffusion activation energies spectrum (i.e., local diffusion coefficient spectrum) and solid-phase processes taking place in the sample under heating.

From the standpoint of membrane defectoscopy, the combination of the tracer method (i.e., the study of the permeability process peculiarities by using hydrogen isotopes), the hydrogen thermodesorption method and the inert (radioactive or stable) gas probe method are especially effective. It is hoped that a combination of these methods will make it possible to determine the type, concentration and dimensions of the defects, as well as to study the evolution of the degree of the metal defects with various external effects on the sample.

4.2 Auger Electron Spectroscopy (AES)

Auger electron spectroscopy relates to the probing methods for the surface study based on particle emission or radiation as a result of the action of the probing flux of particles (electrons, positrons, ions) or electromagnetic radiation on the surface being studied [10]. Central to the AES is the measurement of the energy and number of Auger electrons being emitted from the bombardment of the solid surface by an electron beam.

The Auger spectrum form reflects the composition of the surface layer of the material, 2-4 atomic layers in thickness. See Figure 6 for an example of the Auger spectrum of the real nickel surface. If impurity atoms are applied to the surface the AES makes possible detecting about 10^{12} coat monolayers of these atoms. The absolute sensitivity of this method makes up 10^{-14} at.%, sensitivity to impurities of the volumetric nature is about $10T^{-3}\%$. The method can be employed for

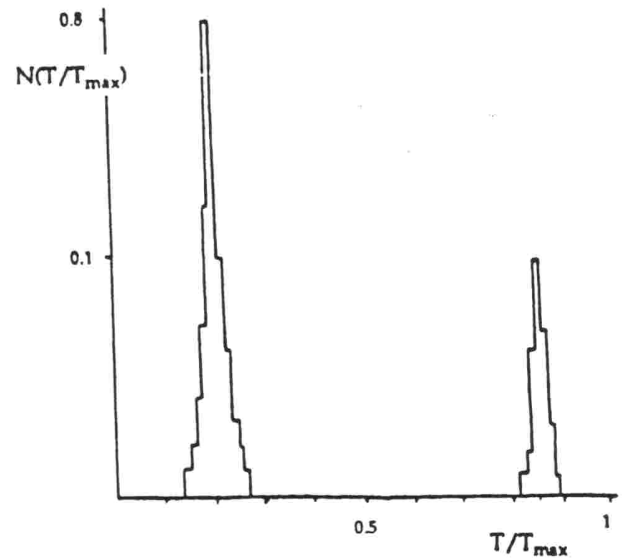


Figure 6. Local solubility constant spectrum (by the results of processing data of Figure 5).

the qualitative and quantitative analysis of all elements, except for hydrogen and helium. A distinctive feature of the AES method is the Auger transition sensitivity to the chemical nature of the analyzed elements interaction on the surface or pre-surface layer of the material. This interaction is reflected on the Auger spectra as chemical shifts and as newly appearing peaks corresponding to the Auger transitions between the inner shells and the valence band.

Thus, the AES presents reliable quantitative information about the surface composition (elemental and chemical). In a layer-by-layer analysis made with the use of ionic etching, the AES permits studying the chemical elements distribution pattern in the volume or along the chosen section. The AES method makes possible monitoring the variations in the state of the surface and pre-surface layer during the permeability process, including when the sample is subject to external actions. Besides, within the bounds of the present work the AES method is applicable for studying the adsorption, desorption, of impurities from the volume to the surface, chemical reactions on the membrane outlet surface, impurities segregation at the grain boundaries under thermal treatment of metals and upon exposure to mechanical loading, oxidation and corrosion.

In the experimental DAM-1 complex, the Auger spectrometer was arranged in the electron probing diagnostics chamber separated from the research chamber by a free-flow valve and pro-

vided with an individual vacuum unit. The complex employs an electrostatic energy analyzer of the deflection type [11].

A Auger spectrometer was used in this work for monitoring the state of the membrane outlet surface prior to the beginning of the diffusion experiment (qualitative and quantitative analyses of the elemental surface composition), in the course of the diffusion experiment (monitoring of variations in the concentration of one of the elements), and after the diffusion experiment. When the free-flow valve was closed, the investigated sample chamber was chemically treated (reduction—oxidation reactions were conducted at different temperatures and partial pressures of chemically active gasses; membrane catalysis was performed) or the sample was subjected to mechanical deformation. After this the research chamber was again evacuated and Auger spectra of the modified sample were taken.

4.3. Nuclear Gamma-Ray Resonance Spectroscopy Method (NGRS)

The essence of the nuclear gamma-ray resonance spectroscopy (Mossbauer effect) is in the effect of resonance absorption and emission of gamma quanta by nuclei in the solid without loss of energy by nucleus recoil. A necessary condition for this resonance absorption is the equality which is consumed by the gamma quantum for the nucleus excitation and the gamma transition energy, i.e., the difference in the inner nucleus energies between the excited state and main state.

The NGRS method is effective for the solution of such physical metallurgy problems as the determination of the phase state of steels and alloys, the study of phase transformations, atomic and magnetic ordering processes, diffusion mechanisms in solids, interaction between gases and alloys, as well as local mechanical stresses. The method makes it possible to qualitatively evaluate interatomic bond forces. The observation of the resonance spectra both on gamma quanta and conversion electrons widens the opportunities of Mossbauer spectroscopy. The method requires that the sample contains certain nuclides—Mossbauer isotopes of iron, tin, antimony, tungsten, and others.

For studying dynamic effects (diffusion of hydrogen atoms and other impurity atoms) the NGRS employs the dependence of the Mossbauer effect probability on the crystal lattice dynamics, because the energy distribution on Mossbauer gamma quanta and conversion electrons changes

during the atom diffusion in the solid for time periods comparable to the time of the Mossbauer levels life [12].

In studying diffusion in solids the NGRS method makes it possible to take measurements on one and the same sample, and the diffusing atom concentration gradient is not obligatory. This method permits revealing a concrete mechanism for mass transfer, as the line spreading is connected with jumps occurring for the time

$$\tau_D < T$$

(τ_j is the jump time, τ is the nucleus life time) and the method allows also for obtaining data on the diffusion shock anisotropy.

The NGRS method was used for the study of dynamic effects during hydrogen permeability of iron. A membrane of iron enriched with isotope ^{57}Fe (98%) was used to this end. The source was isotope ^{57}Co placed on a vibrator (see Figure 1). The source gamma rays passed the beryllium window in the electron probing chamber and got onto the membrane. The reflected gamma quantum flux passed through another beryllium window and got onto the scintillation ionizing-radiation detector. On the whole, the standard Mossbauer investigation equipment of the Medeya type was used for the control of NGR spectra.

When NGR spectra of conversion electrons were taken, gamma quanta from the source also got onto the membrane outlet surface, but only reflected conversion electrons were recorded.

5. CONCLUSION

The DAM-1 Complex is included in the equipment assembly, designed by the authors which provides a means for the measurement of dissolution kinetics and diffusion of both stable and radioactive gases, using all basic methods of gas diffusion under various external effects [1,4,8,13–15]. A characteristic property of the DAM-1 Complex is its modular construction the basic model being a research chamber with a vacuum unit, mass-spectrometer and gas bleeding-in system. The basic model allows conducting the diffusion experiment by principal diffusion methods. Equipment for various diagnostic methods is connected to the research chamber as needed. And the plant does not require any significant modification to this end. In addition to the above-described methods, the complex can use such methods as electron-photon emission, total-current spectroscopy, exoelectron emission and others [16].

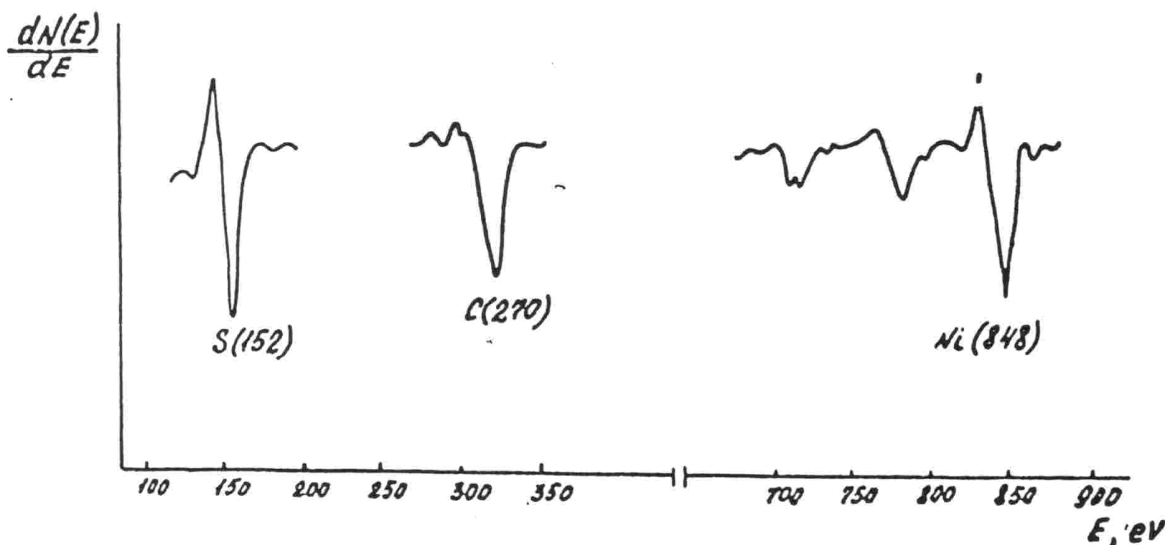


Figure 7. Auger spectrum of real nickel surface at room temperature.

Experience for the use of the proposed equipment has demonstrated that an adequate interpretation of experiments for studying the processes of interaction between hydrogen and metals, hydrogen and impurities on the surface or in the volume of the solid, as well as the processes of translational diffusion is possible only with the use of a diagnostic equipment set.

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