The Determination of the Small Hydrogen Traps as Nucleus of Fatigue and Destruction

Institute Problems of Machinery RAS
St.-Petersburg

V. A. Polyanskiy ***vapol@electronbeamtech.com***

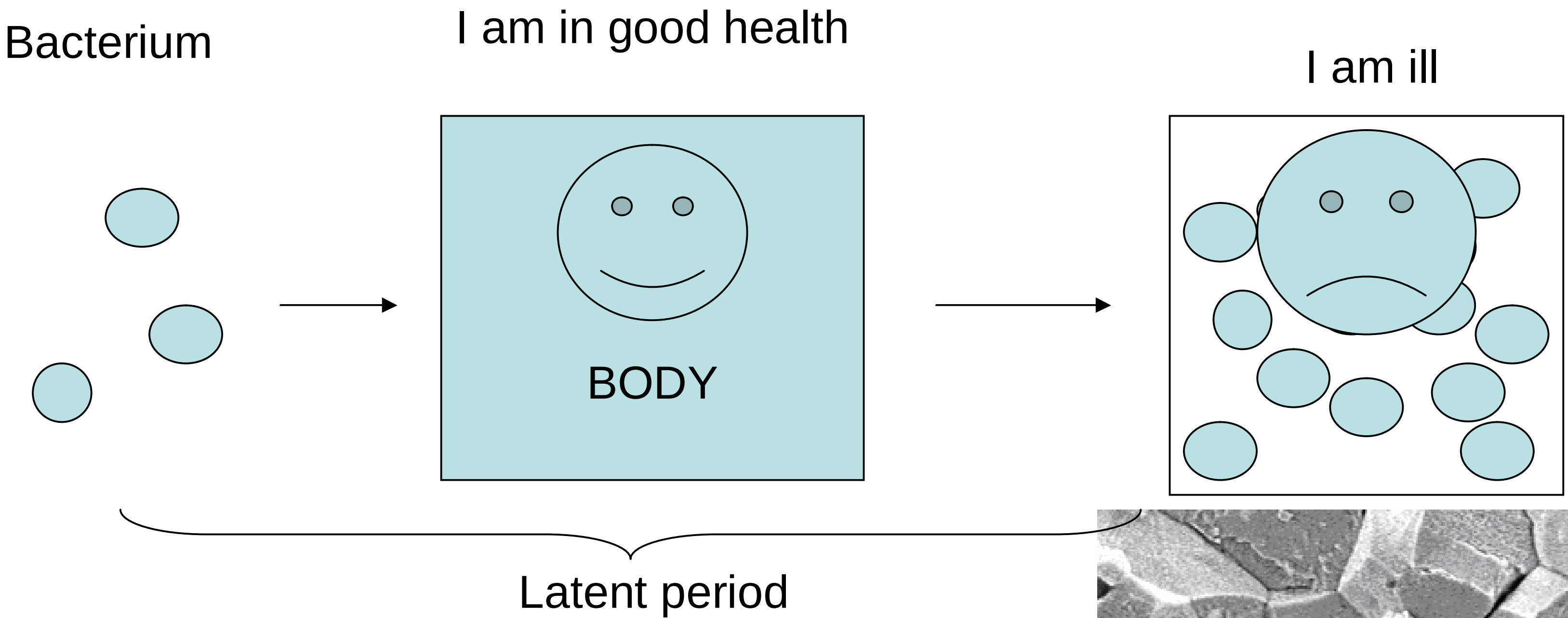
V. A. Polyanskiy *, A. M. Polyanskiy,† A. K. Belyaev,‡ Yu. A. Yakovlev,*
* St.-Petersburg State Polytechnic University, Polytekhnicheskaya, 29, St.-Petersburg, 195251, Russia
† RDC Electronbeamtech, St.-Petersburg, Bronevaya 6, 198188, Russia
‡ Institute of Problems in Mechanical Engineering of the Russian Academy of Sciences, Bolshoy pr. V.O. 61, St.-Petersburg, 199178, Russia

Acknowledgements

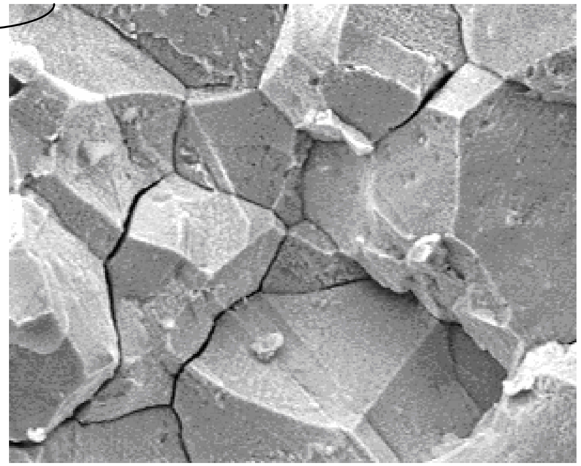
The financial support of the Sandia Corporation, contract 891092, and the Russian Foundation for Basic Research, grants 08-08-00737 and 08-01-12017, is acknowledged.

Content

Hydrogen embrittlement's Health analogy



Micro cracks, diffusion channels, hydrogen accumulation



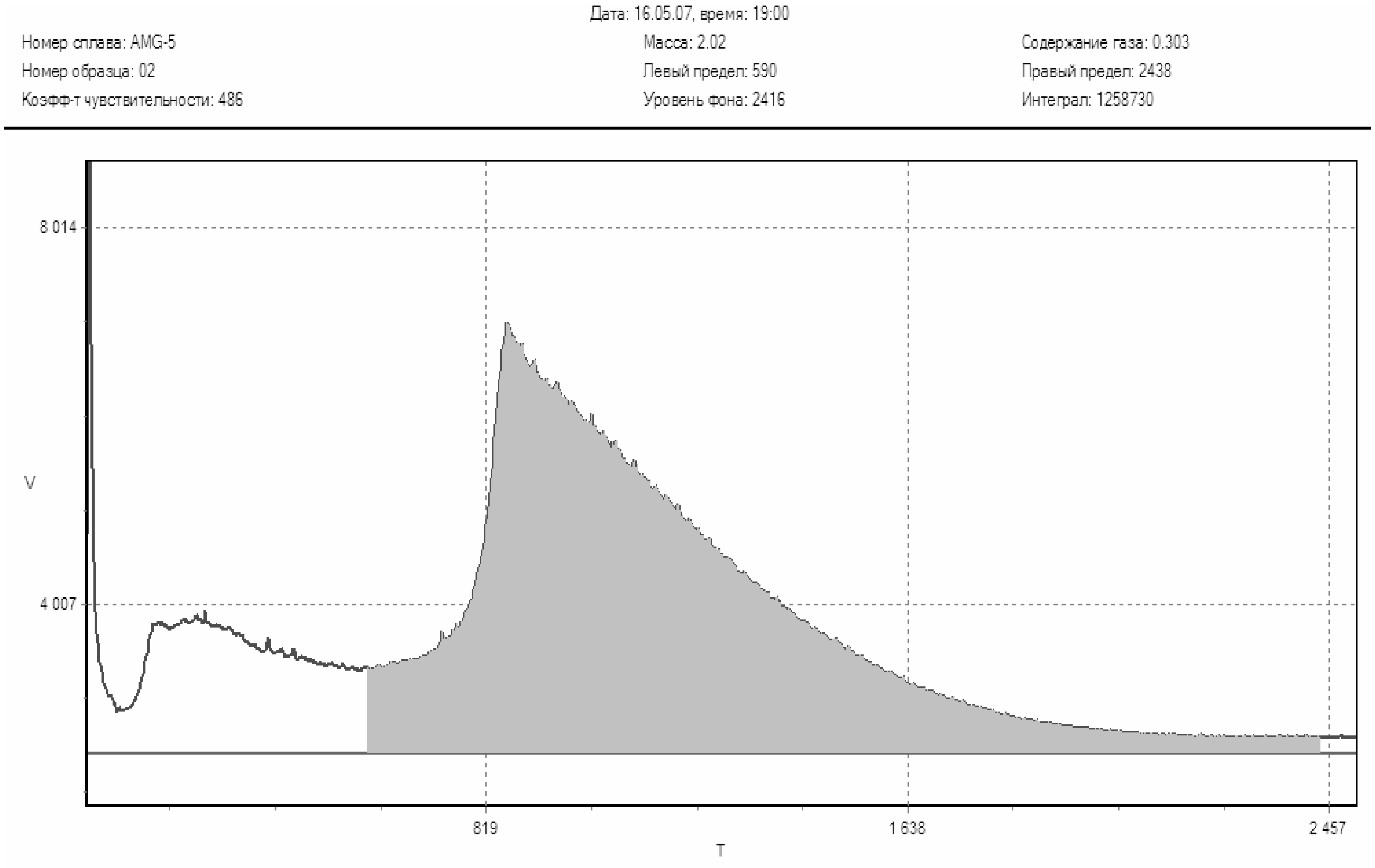
Hydrogen embrittlement, hydrides

Hydrogen analyzer AV-1

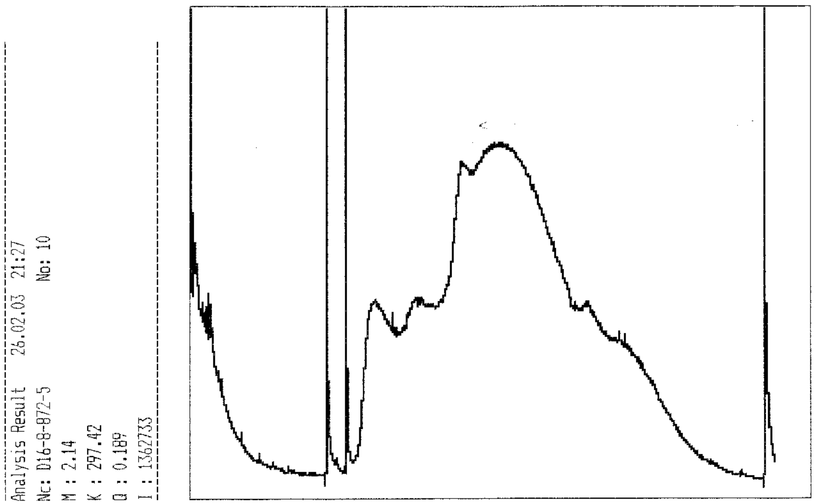
Comparison between the measurement characteristics of RH402 and AV-1

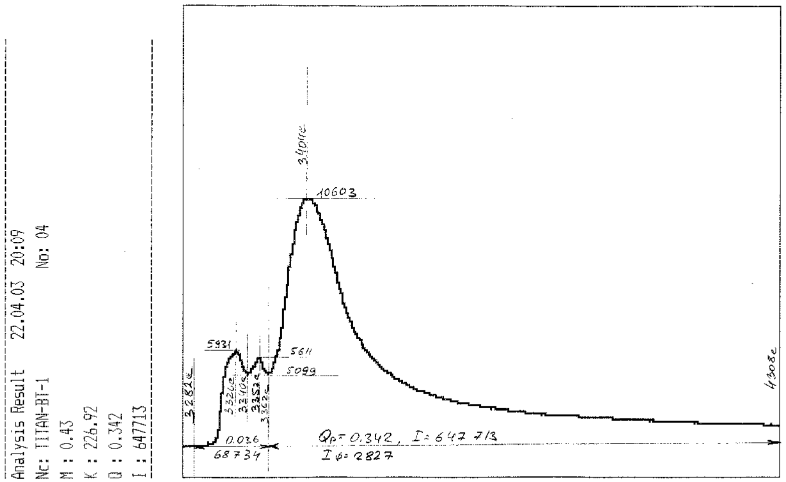
1	**The type of analyzer**	**RH 402**	**AV-1**
2	Least value of hydrogen content's measurements in solid probe	0,01 ppm	0,01 ppm
3	Mass of the probe	1g -10g	0,001g – 5g
4	The type of the probe's material	Metals without Mg & Li as alloy Component	Any metal and inorganic materials
5	Instrument sensitivity	0,001 ppm	10^{-5} ppm
6	The principle of operation	The measurement of thermal conductivity of gas carrier with probe's emitted hydrogen	The mass-Spectrometric analysis of probe's emitted gases

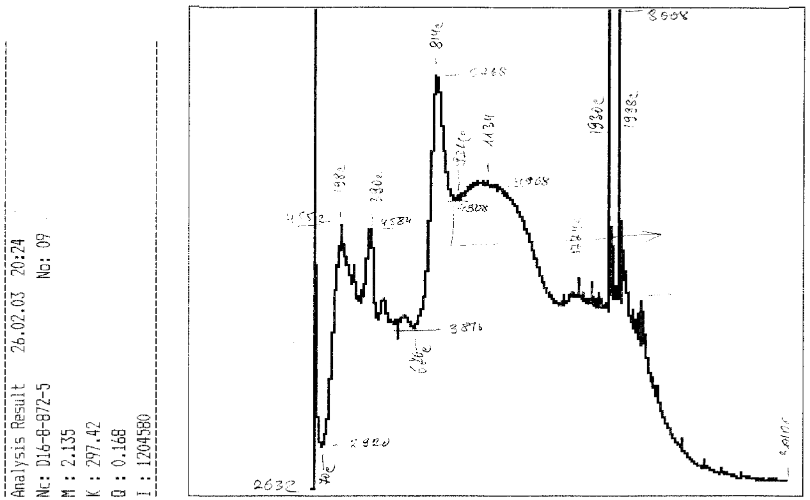
Extraction curve for the alloy AMg5

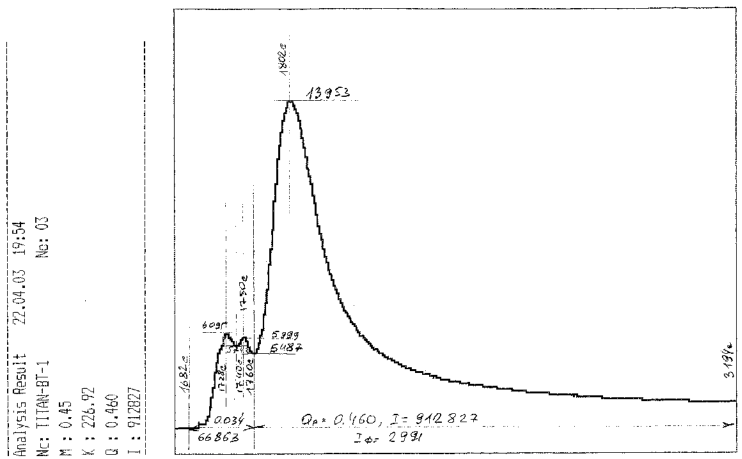


Extraction curves for the aluminum alloy D16 and titanium alloy VT-1



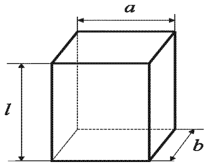






Estimation of hydrogen binding energy

Probe for analysis



The heat flux absorbed by the probe

$$\frac{dQ}{dt} = s\,S\,e_t\,(T_0^4 - T^4), \qquad (1)$$

where
σ = 5.6687·10^{-8}W/m^{2}K^4 is the Stephan-Boltzmann constant,
S is the probe surface area,
T is the probe temperature,
ε_t is the absorption coefficient

$$e_t = 0.2 \qquad (2)$$

The heat *dQ* absorbed by the probe

$$dQ = C\,r\,VdT\ , \qquad (3)$$

where
ρ and *V* are the probe density and volume
C=0.6kJ/kg·K is the heat capacity

equation for the probe heating:

$$\frac{dT}{dt} = \frac{s\,S}{C\,r\,V} \cdot 0.2 \cdot (T_0^4 - T^4). \qquad (4)$$

The equation for time-dependent hydrogen diffusion on in the probe is

$$\Delta C = \frac{1}{D}\frac{\partial C}{\partial t} \quad C\,|_{t=0} = C_0 \quad C\,|_S = 0 \qquad (5)$$

where
C is the hydrogen concentration in the probe,

$D = D_0 \cdot \exp(-\frac{u}{kT})$ is the diffusion coefficient for hydrogen in metal,
u is activation energy,
D_0 is diffusion constant,
k is the Boltzmann constant.

Estimation of hydrogen binding energy

First term of the Fourier expansion of diffusion's equation

$$C(x,y,z,t) = \frac{C_0 p^3}{8} \sin\frac{px}{a} \cdot \sin\frac{py}{b} \cdot \sin\frac{pz}{l} \cdot f_1(t,u,D_0), \quad (6)$$

where

l, a and *b* are the height, width and depth of the probe

*f*1(*t,u,D*0) is the solution of the equation:

$$\dot{f}_1 + D_0 \cdot \exp(-\frac{u}{kT})(\frac{p^2}{a^2} + \frac{p^2}{b^2} + \frac{p^2}{l^2}) f_1 = 0 \qquad (7)$$

$$f_1(0,u,D_0) = 1$$

The hydrogen flux is

$$q(t) = -\int_S D \frac{dC}{dn} dS \,, \qquad (8)$$

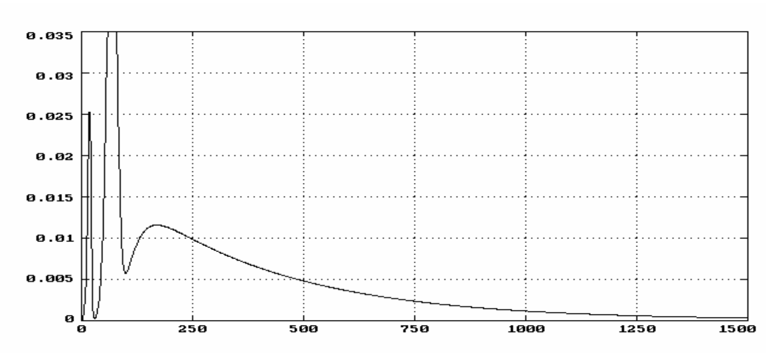
$$q(t) = \frac{16C_0}{p^2} \cdot \left[\frac{1}{a^2} + \frac{1}{b^2} + \frac{1}{l^2}\right] \cdot D_0 \cdot \exp(-\frac{u}{kT}) \cdot f_1(t,u,D_0). \qquad (9)$$

$$q(t) = \frac{16}{p^2} \cdot \left[\frac{1}{a^2} + \frac{1}{b^2} + \frac{1}{l^2}\right] \cdot \sum_i C_{0i} \cdot D_{0i} \cdot \exp(-\frac{u_i}{kT}) \cdot f_1(t,u_i,D_{0i}),$$

Approximation of the experimental extraction curve by the calculated curve

q(t)

q(t)



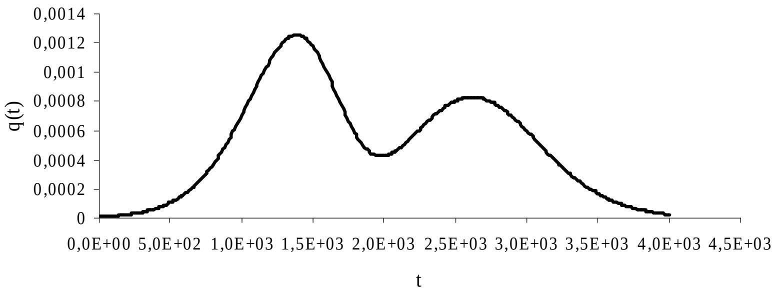
t, [s]

The approximating curve for the case of three maxima

q(t)

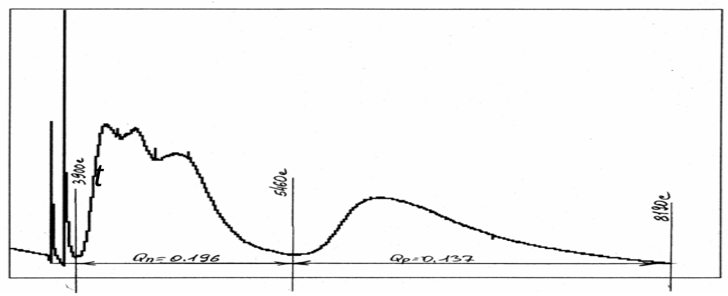
t

The experimental curves for the titanium



The approximating curve for the case of two maxima

q(t)

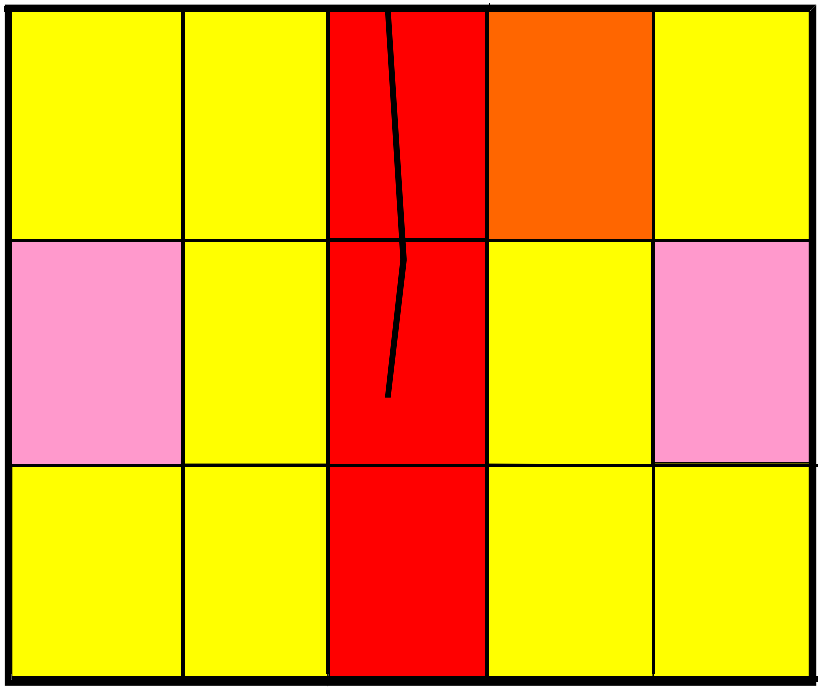


t

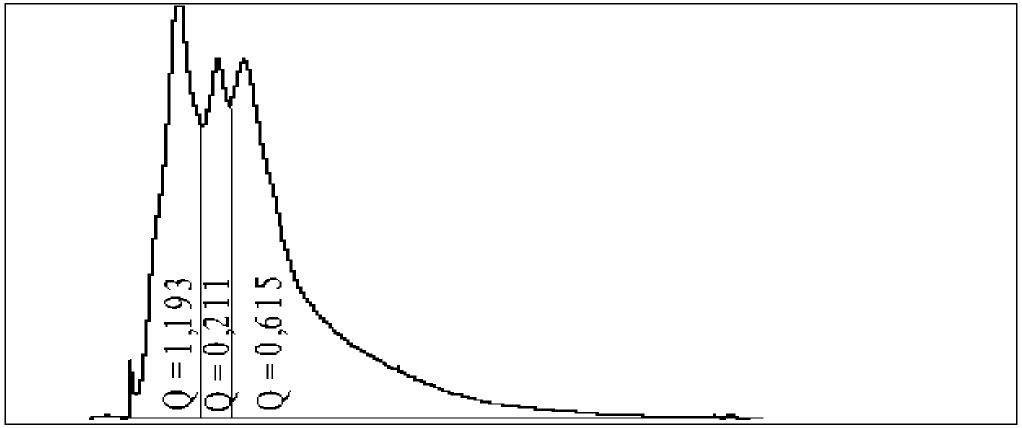
The experimental curves for the aluminium

Map of hydrogen distribution in the plate

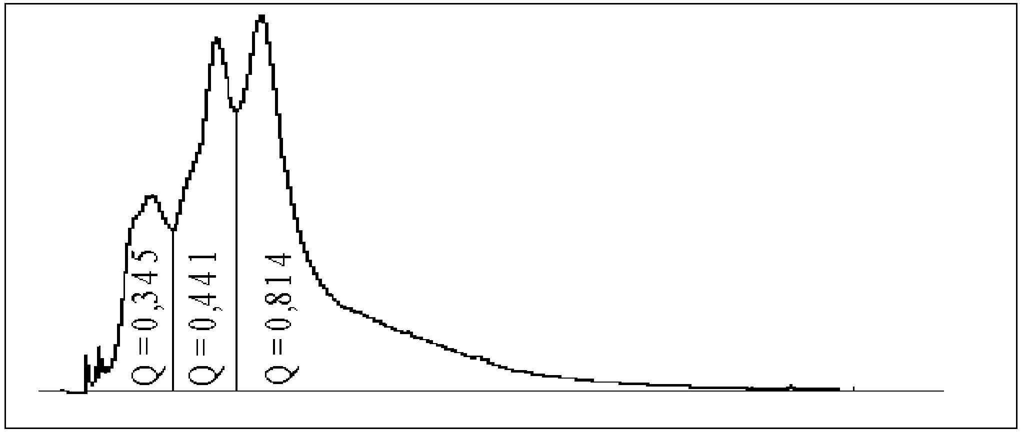
Red – 2.0 ppm;
Orange – 1.7 ppm;
Yellow – 1.3-1.4 ppm:
Violet – 1.2 ppm.



Redistribution of the hydrogen among the energy levels



Extraction curve for simple from the line of the formation of the crack

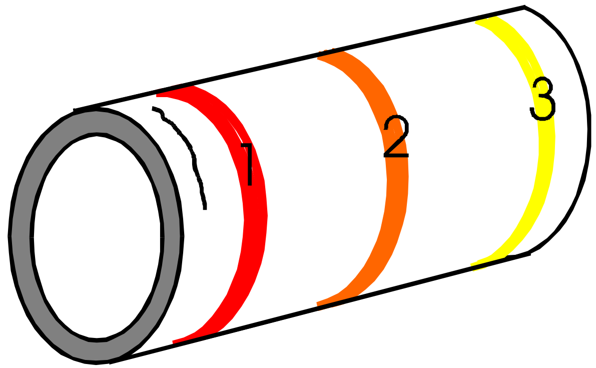


Extraction curve for simple from the background

Results of analysis of hydrogen content in titanium tubes

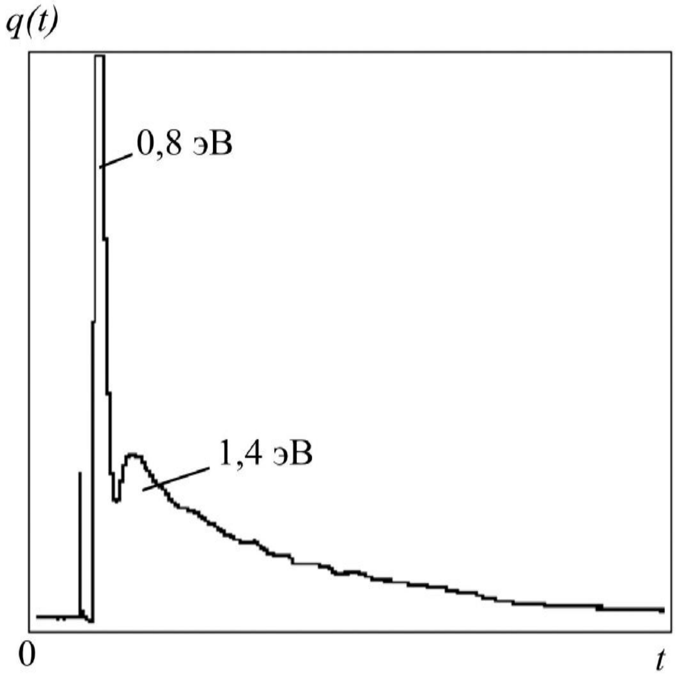
Results of analysis of hydrogen content

Probe number	Extraction temperature	Probe mass (mg)	Hydrogen Concentration [%] of mass
1	800 C	95	0,056
2	800 C	90	0,037
3	800 C	90	0,021

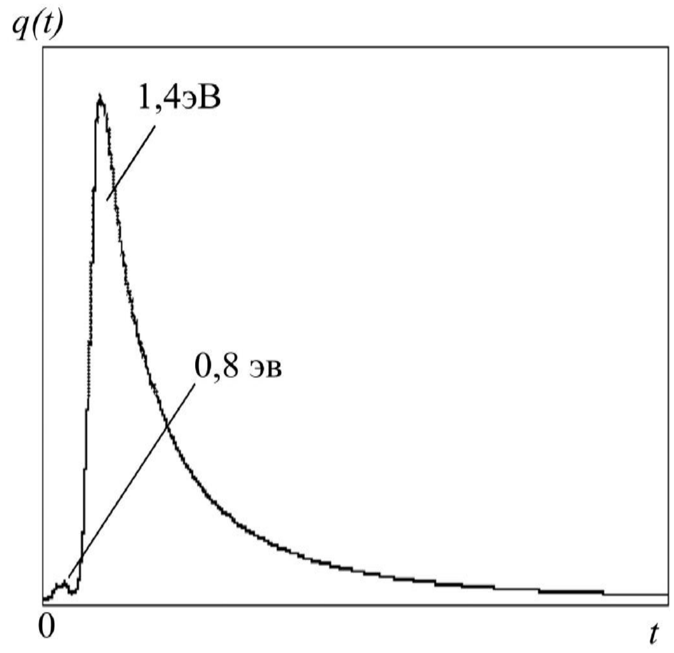


Scheme of probe position with respect to the crack in titanium tube.

Results of analysis of hydrogen content in titanium tubes



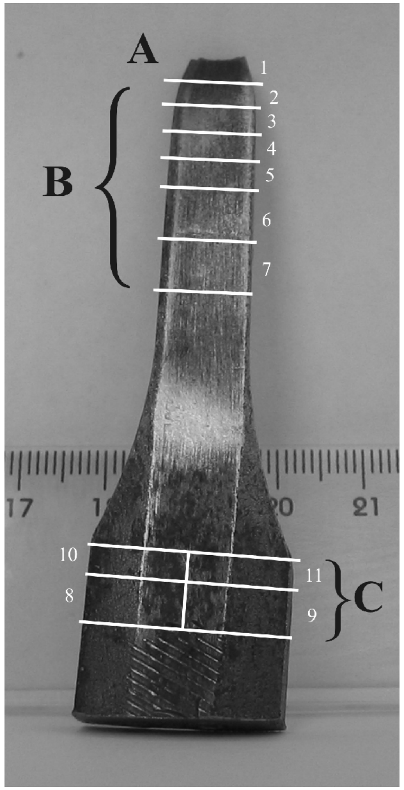
Probe #3

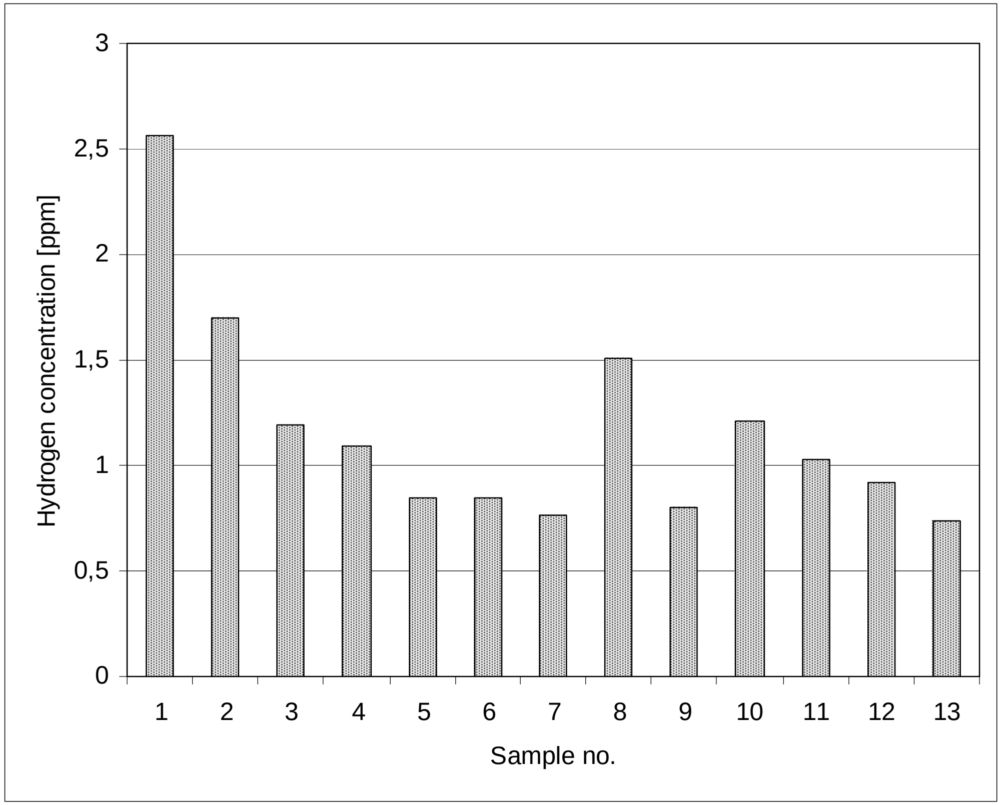


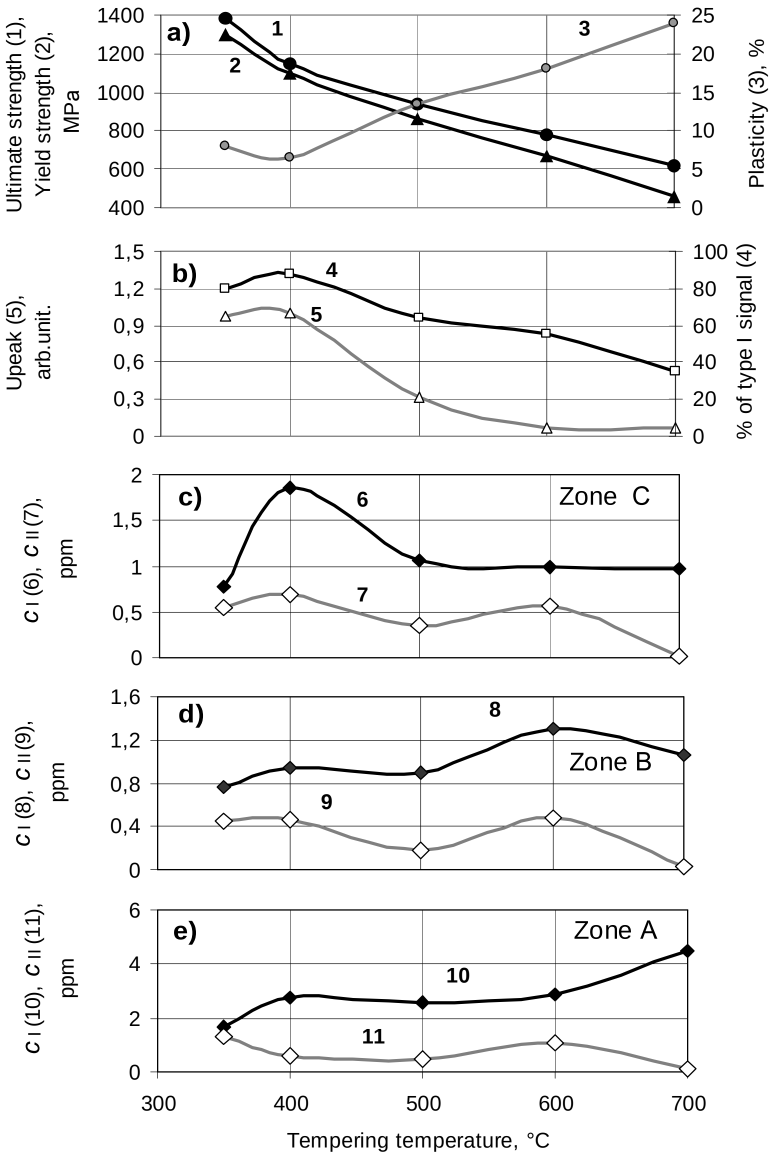
Probe #1

The sample of 35G2 steel fractured under axial tension showing the schematic of test sample cutting.

Upper part (zone A) - zone of fracture, lower part (zone C) - sample grip zone in the test bed



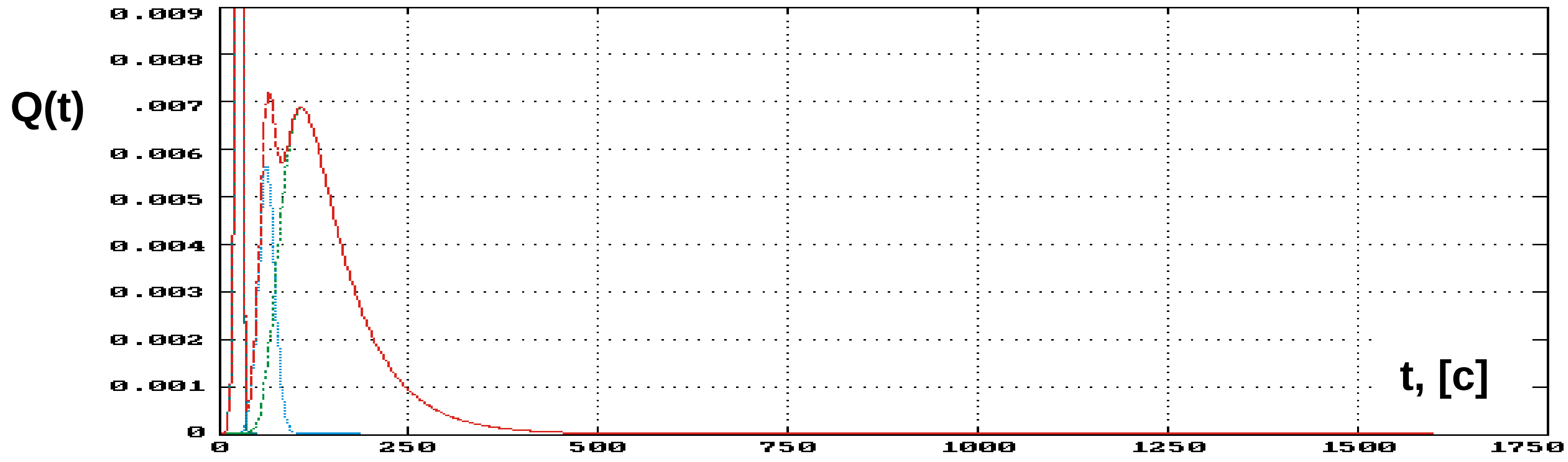


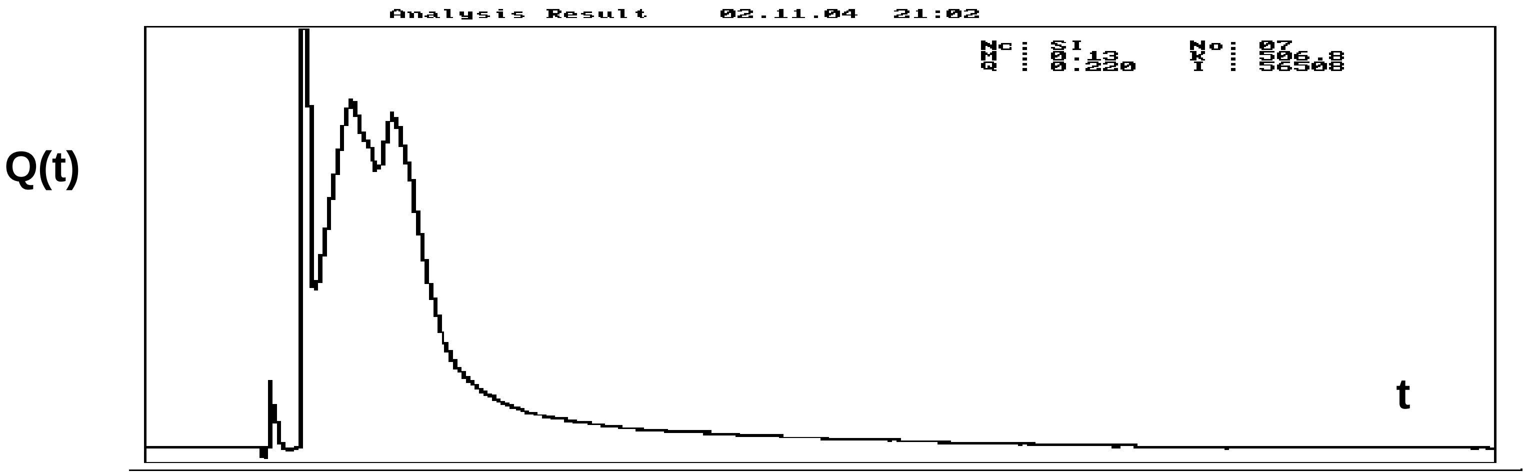


Mechanical (a) and acoustic (b) characteristics and concentrations of diffusely (cI) and bound (cII) hydrogen (c-e)

plotted vs. tempering temperature for 35G2 steel samples quenched from 850oC in water. 1– ultimate strength; 2 - yield strength; 3- relative residual elongation,; 4 - percentage of type I signals (group 1); 5 - AE envelope peak height; 6, 8, 10 - concentrations of diffusion-active hydrogen in the unstrained zone C and zones of strong (zone A) and weak (zone B) strain, respectively; 7, 9, 11 - concentrations of strongly bound hydrogen in zones C, B, and A, accordingly

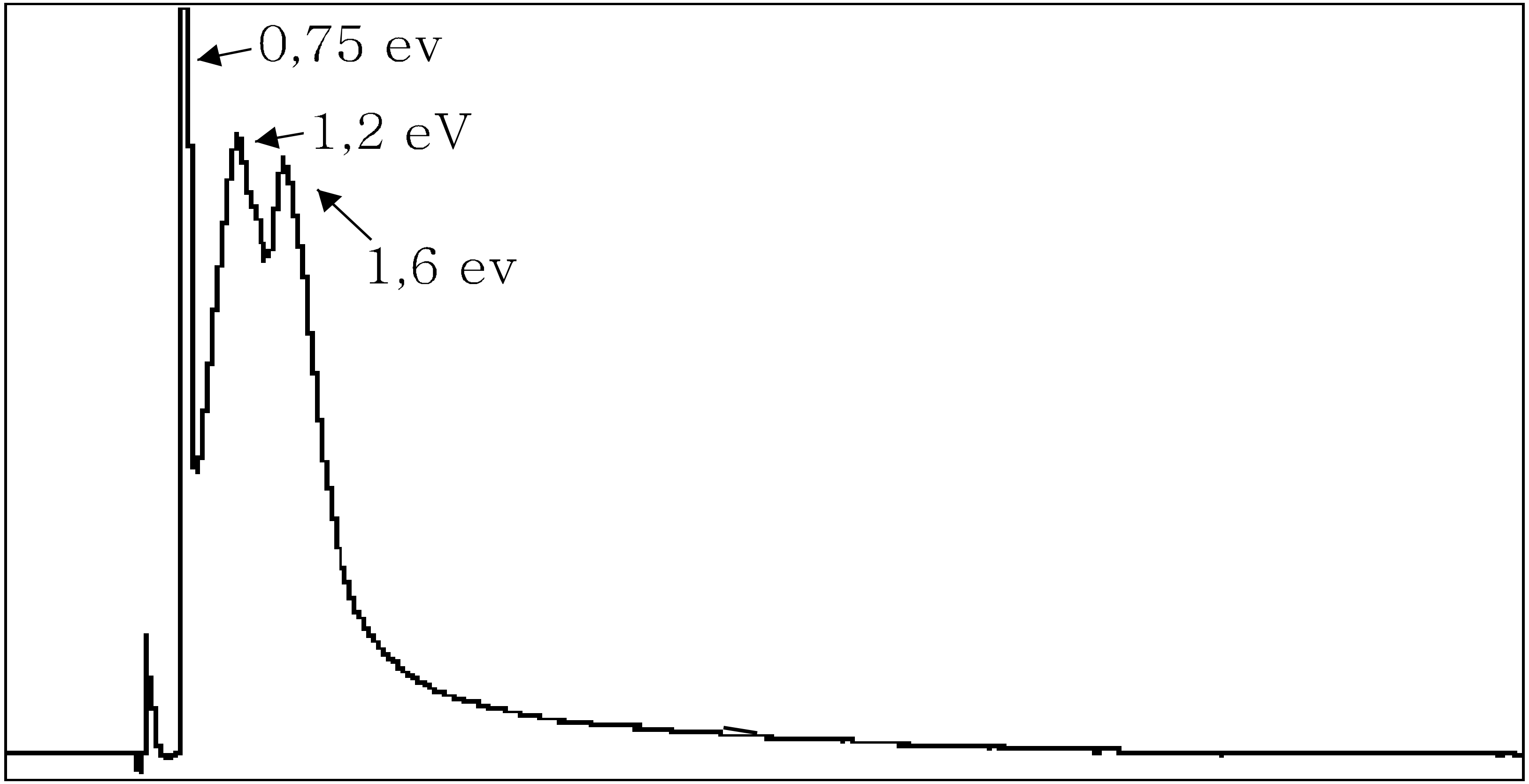
The experimental curves for the crystalline silicone (bottom) and its approximating curve (top)





Extraction curve for monocrystaline silicon indicating the binding energies that correspond to separate peaks of the curve

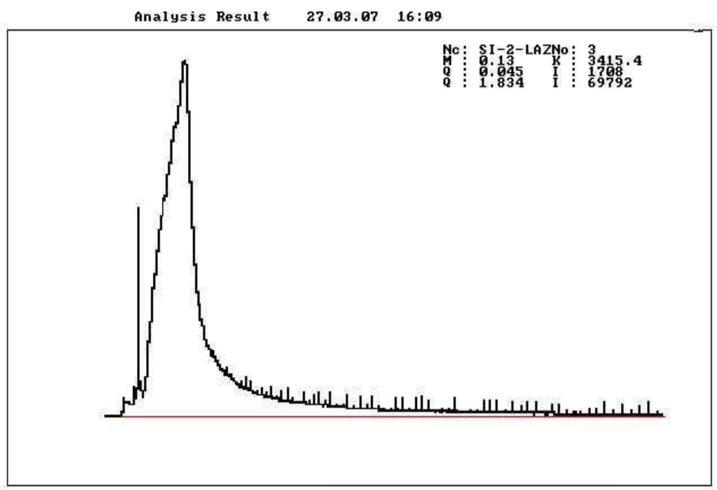
q(t)

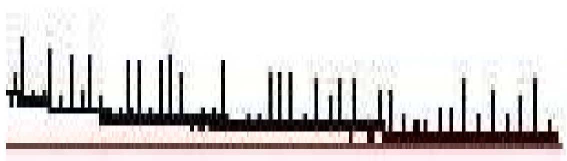


t

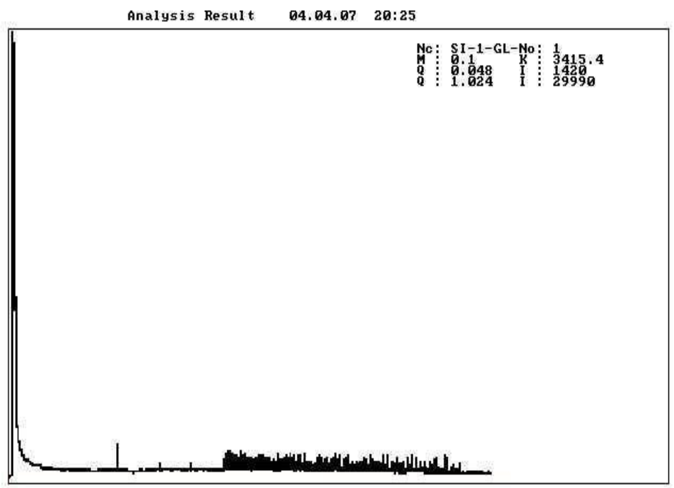
Diffusion of hydrogen from mono crystalline silicon plates

After laser treatment

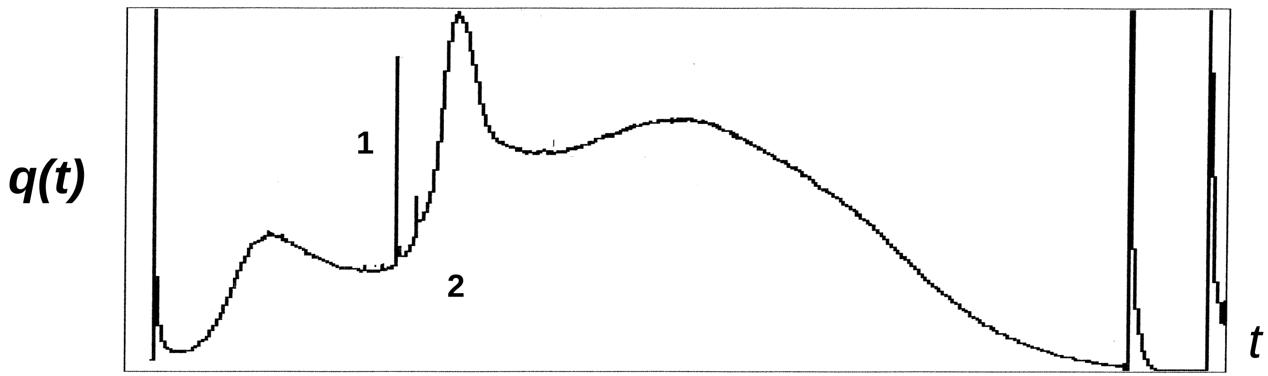




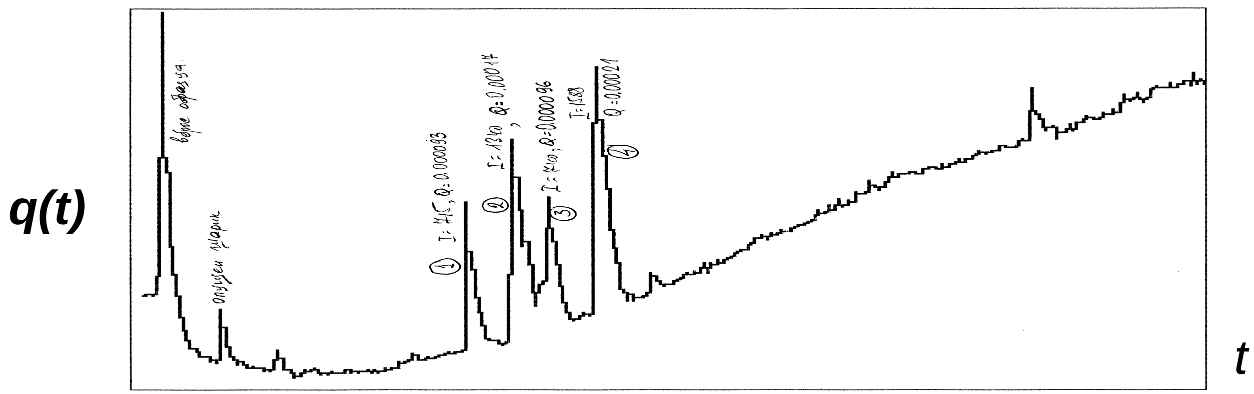
Before laser treatment



Explosive character of hydrogen emission from the traps-defects

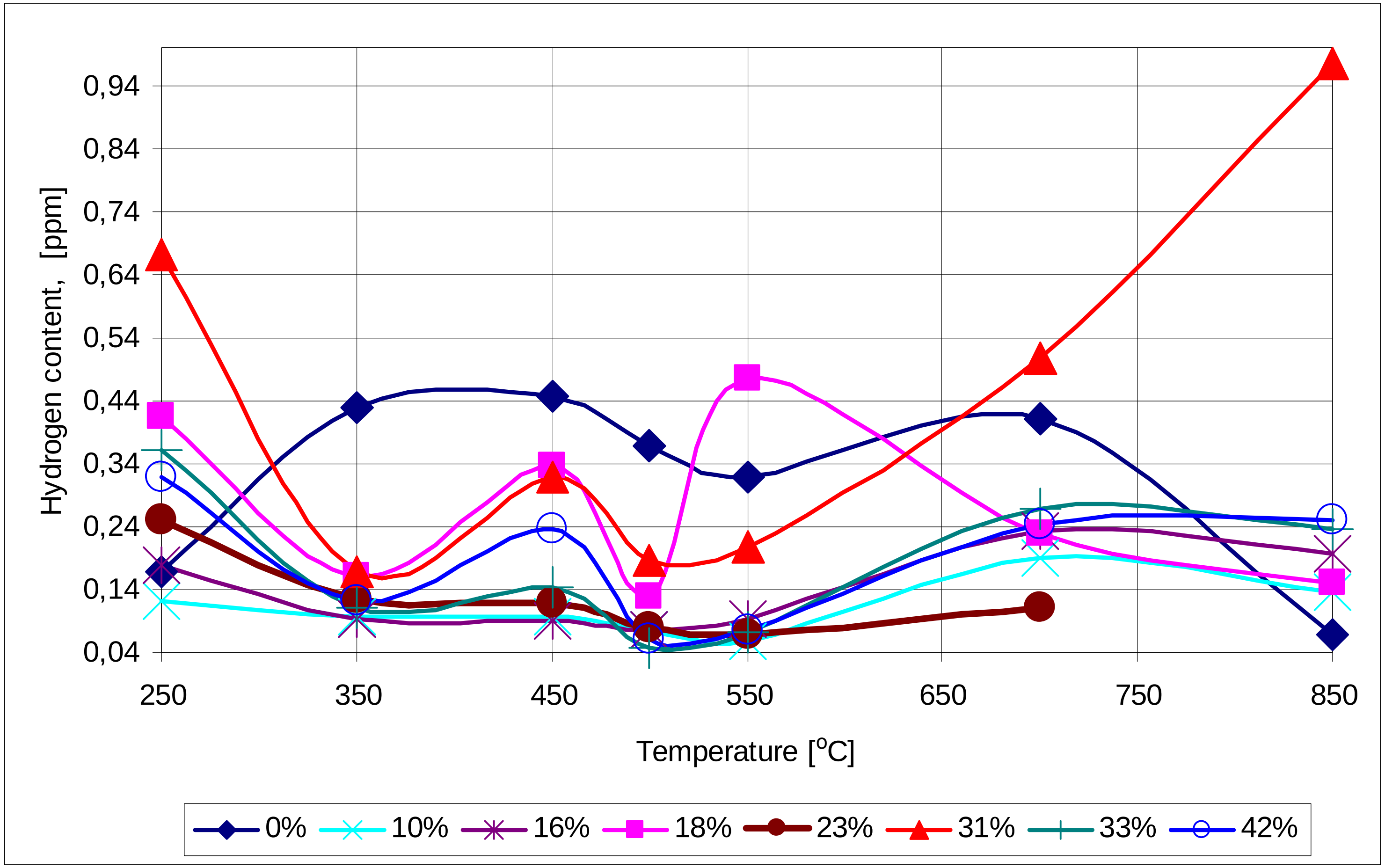


Experimental extraction curve for the aluminum alloy AD-31



Fragment of experimental extraction curve for the aluminum alloy A8

Nanosteels



Hydrogen content, [ppm]
0,94
0,84
0,74
0,64
0,54
0,44
0,34
0,24
0,14
0,04
250
350
450
550
650
750
850
Temperature [°C]
0%
10%
16%
18%
23%
31%
33%
42%

CONCLUSIONS

- We have the equipment that makes it possible to obtain information on the structure of hydrogen bonds within the material according to the hydrogen extraction curve at heating a specimen in vacuum. The accuracy of determination of the extraction curve makes it possible to obtain information on both the hydrogen binding energy in the metal and on the concentration of mechanical flaws.
- The experiments we conducted have confirmed that the fatigue phenomena and the destruction of structural materials are accompanied by increased concentration of bound hydrogen in the destruction zone.
- It was first time detected that the mechanical loads result in a substantial redistribution of hydrogen according to the binding energies inside metals.
- This approach to investigation of the properties of materials does not require preliminary saturation of the specimens. The natural hydrogen in the metals contains the information on the past history of the material, which, once the methods have been developed, will make it possible to obtain more detailed information from the measured extraction curves.
- The metrological setup consisting of a hydrogen analyzer and the calibration standards enables implementing the principle of a single measurement utility for analysis of various metals and alloys and obtaining additional information on the volume and structure of the internal and surface mechanical defects.