Tensile and Microhardness Measurements

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1. TENSILE MEASUREMENTS

Generally tensile tests are of two types with respect to the deformation regime: stress-strain test at constant strain rate and creep rupture experiment at constant loading.

1.1 Stress-Strain Test at Constant Strain Rate

By stress-strain test one can measure:

- the modulus of elasticity (E);
- the yield point (σ_y ; ε_y);
- the elongation at break (ε_b);
- the ultimate stress (tensile strength) (σ_b);
- the stress and strain at the minimum of the curve plotting neck formation (σ_{min} ,ε_{min});
- the breaking work (It can be graphically calculated from the area under the curve.).

$\sigma = P/S \quad \varepsilon = (\Delta l/l_0).100$

$E = \sigma/\varepsilon$

l_0 - length
S - cross-section area
P - loading
Δl - elongation

Mechanical behavior is influenced by complex factors. So we can summarize the following parameters:

· materials specific parameters:
- relative molecular mass
- branching degree
- crosslinking degree
- chain mobility

· morphological parameters:
- degree of crystallinity
- molecular orientations

· environmental parameters:
- strain rate
- type and level of loaded stress
- temperature

· thermal-mechanical pre-history

· material defects

In practice the influence of time and temperature is the most important.

If the time of deformation is very short, that means the strain rate is very high compared with the duration of the molecular dislocation processes, then the polymeric materials behavior is brittle and stiff.

If the dislocation mechanism has enough time to achieve an equilibrium value for the stresses, the polymeric materials respond tough and soft.

Similar dependences are resulting from the influence of the temperature. At low temperature they show a decided brittle behavior and high tensile strength. At high temperature the tensile strength is considerably lower but the toughness and elongation at break are very high (Fig.2).

That means that at one and the same application at different temperatures or deformation rates can result in brittle or tough behavior of the materials.

1.2 CREEP RUPTURE EXPERIMENT AT CONSTANT LOADING

The creep is a process of a time-dependent increase of material deformation at constant loading or stress.

Fig. 3 shows the schematic diagram of an apparatus used for creep rupture measurements.

Typical creep-test curves are shown in Fig. 4. They have three characteristic sectors: initial, settled and critical.

From these curves one can determine the following mechanical characteristics:

- time-to-break (τ_b);
- deformation at break(ε_b);
- deformation time-to-break (t_K). It is the time for reaching the critical creep sector;
- compliance (I) - It is a unit characterizing the ability of the polymers to develop a reversible deformation under applied stress.

When the condition $\sigma(t)$=const is fulfilled the compliance could be determined from the relation between deformation and stress.

$$I=\varepsilon(t)/\sigma$$

i.e. at the area of linearity of mechanical behavior the compliance is a inverse proportion to the elastic modulus.

Usually simultaneously with creep deformation some microdestructive processes take place in the material. They could be a transformation of the type of supermolecular structure, and appearance and growing of microcracks, even breaking of some tying molecules.

By now it is not clear which one of the processes - creep or destruction is primary. If the microdestructive processes are faster than the deformation, a breaking of the samples occurs. On the opposite, if the deformation is more facilitated then the creep takes place.

The plastification of the polymer accelerates the creep and the orientation delays it.

The creep rapidly increases if some vibration with not big amplitude is added. This is the so called vibrocreep. This phenomenon is connected with heating the material as a result from dissipation of the fluctuation energy.

Fig. 5 presents our attempt to compare both types of tensile experiments: the creep test and the test at constant strain rate.

2. MICROHARDNESS MEASUREMENTS

Hardness is a characteristic of the material which defines its local resistance against penetration, sawing, drilling, abrasion. Usually it is measured by static penetration of the material loaded with harder body having standard geometric shape, called « indentor».

2.1. HARDNESS TESTS

According to the geometry of the indentor the hardness test can be:

Brinel . In this test a steel ball is forced against the flat surface of the specimen. Usually it is preferred for measuring the macrohardness of large pieces in which large indentation is acceptable.

Knoop. The rhombic-based pyramidal diamond with included angles of 172^0 and 130^0 between opposite edges is used as indentor. This test is very sensitive to material anisotropy because of the twofold symmetry of the indentation.

Rockwell. In this test the depth of indentation is read from a dial. No microscope is necessary. Usually it is used in production and quality control, where absolute hardness is not important.

Scleroscopy. Here the rebound of a diamond tipped weight dropped from a fixed height is measured. This method is used for specimens that cannot be removed or cannot tolerate large indentation.

Scratch - hardness Test. In this test a corner of a diamond cube is

drawn across the sample surface under a constant force applied to the body diagonal of the cube, creating a V-shaped groove. Its width is measured microscopically.

Vickers. The test uses a square pyramid of diamond with included angles between nonadjacent faces of the pyramid of 136^0.

The Vickers macrohardness tests use a load P>30 N while the Vickers microhardness (MHV) tests use the load P<1.5 N. In contrast to the macrohardness Vickers microhardness is a unit dependent on loading.

And The Vickers microhardness is given by:

$$MHV=k.P/d^2$$

where d is the diagonal of the indentation, and k=1.8544 is a constant connected with indentor geometry.

2.2. METHODS OF MICROHARDNESS MEASUREMENTS

2.2.1. Measuring and Investigation of Mayer's Straight Lines.

The dependence between P and d is known as Mayer's power law:

$$P = a.d^n$$

where a and n are constants for the material. Constant a depends on the strength properties and constant n depends on the plastic features of the investigated material. In logarithmic scale the dependencies are known as Mayer's lines (Fig .6).

$$\log P= \log a + n.\log d$$

The slope of this line n shows the MHV changes inside the sample.

2.2.2. Standard Vickers measurement (MHV=f(P), MHV=f(d) (Fig. 7)

MHV is physico-mecanical characteristic considered as indicative of local plastic resistance of the material against the indentor penetration and it is connected with local irreversible deformation.

2.2.3. Indentation Measurements Under the Load.

2.2.4. Structural Microhardness (MHS) (Fig.8)

This material characteristic is defined analogically to the unit Vickers microhardness:

$$MHS = k.P/D^2$$

where D is the indentation diagonal under a load.

It is evident that thus defined MHS can be considered as measure of the local total material resistance against the penetration and is connected with the total deformation, including plastic, elastic and viscoelastic components.

2.2.5 Penetration Curves Test

The dependence $\Delta h = f(t)$ at constant load P=const is an object of investigation, where Δh is the penetration of the pyramid and t is the time measured since the loading moment. Different penetration curves are shown at Fig. 9.

2.2.6 Relaxation of the Indentation After Load Removal

In rubber-like elastic material only instant recovery takes place. In soft plastic material no recovery occurs. In viscoelastic material after instant elastic recovery a time dependent viscoelastic relaxation begins. The next picture illustrates different kinds of an impression recovery (Fig. 10).

2.2.7. Infinitesimal Hardness Behavior (IHV)

This graphical method is offered by Oesterle (Fig. 11). In order to determine the IHV value one constructs a regression curve of penetration depth (h) vs. load P . This curve is extended by interpolation to the point P=0, h=0 , because when there is no loading. no impression exists. By this procedure h value in the interpolated vicinity could be obtained. A curve P/h vs. P can now be drown both through the real measured points and through the points from the interpolated sector and extrapolated up to cutting the P/h ordinate at IHV value.

$$IHV = \lim P/h \text{ when } P \rightarrow 0$$

The method is suitable for microhardness measuring of thin coatings and foil materials with pronounced plastic deformation, i.e. with clear impression.

2.3. SCIENTIFIC APPLICATION OF MICROHARDNESS INVESTIGATION

The Vickers microhardness test is the mostly used microhardness measurement.

2.3.1. Optimization of the Parameters of the Measurement Ensuring the Reliability and Comparability of the Results.

These parameters are:

- indentation time and time under definite load;
- the minimum sample thickness when the influence of the pad is eliminated;
- the minimum distance between indentations;
- the temperature and humidity;
- the time interval between the removing of the indentor and the microscopic measurement of the size of the diagonals. That parameter is connected with the indentation relaxation;
- the sample orientation;
- the included angle between the light beam and the investigated surface which influences the measurement accuracy.

2.3.2. Investigation of the Deformation Processes Caused by the Indentor Penetration.

- *From mechanical point of view;*
- *From the view point of structural changes.*

2.3.3. Investigation of the Connection Between the MHV and the Structure of the Semicrystalline Plastics

The MHV is sensitive to:

- the degree of crystallinity;
- the lamella thickness and their perfection;
- the hardness of the crystalline phases;
- polymorphic changes in polymers;
- Curie transition and so on.

2.3.4. Investigation of the Connection Between the MHV and other Mechanical Characteristics

- the yield stress σ_y;
- modulus of elasticity E;
- sharpness of the maximum in the stress-strain dependencies at neck formation;
- anisotropy;
- similarity between temperature dependencies of the MHV and dynamic elastic modulus, respectively tg(δ), i.e. correlation with relaxation spectra and so on.

Recently the microhardness has been approved as a reliable technique for both mechanical and microstructural investigation, i.e. it occurs as a bridge between macro- and microcharacteristics of the materials.

2.3.5. Submicrohardness Investigation

This is microhardness investigation in the area of very small loading ($P<0,10$ N).

2.3.6. Microhardness Measurements on thin Materials

Examples for our microhardness investigation

· Structural changes at neck formation in HDPE and LLDPE.
· Structural changes of Gamma-irradiated PEO and determination of doses of «radiation annealing» and «radiation melting».
· Structural changes during sintering of Teflon and UHMWPE.
· Changes in wood-polymer composition exposed in HCl atmophere.
· Composition UHMWPE / Fe.
· Composition Teflon / basalt powder.
· Polymer coating based on UHMWPE and metal oxides.

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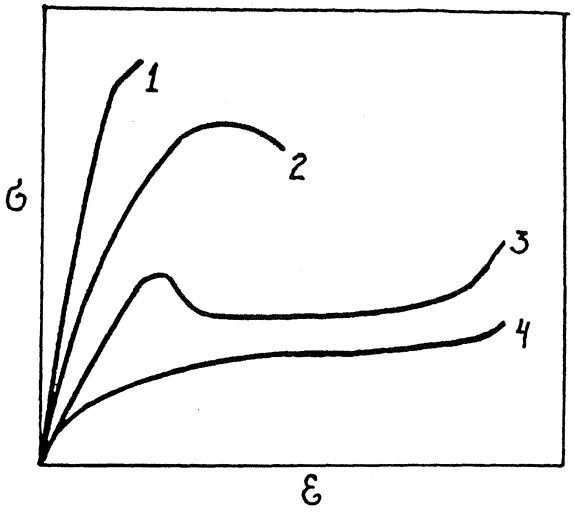


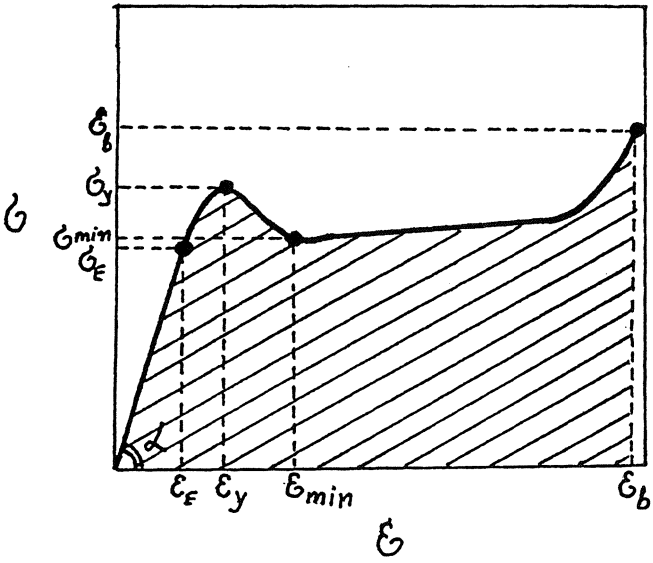
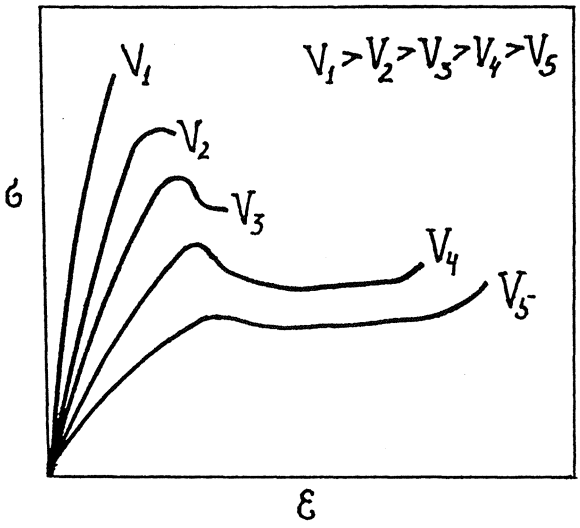


Fig. 1


V1
V1>V2>V3>V4>V5
V2
V3
V4
V5
σ
ε

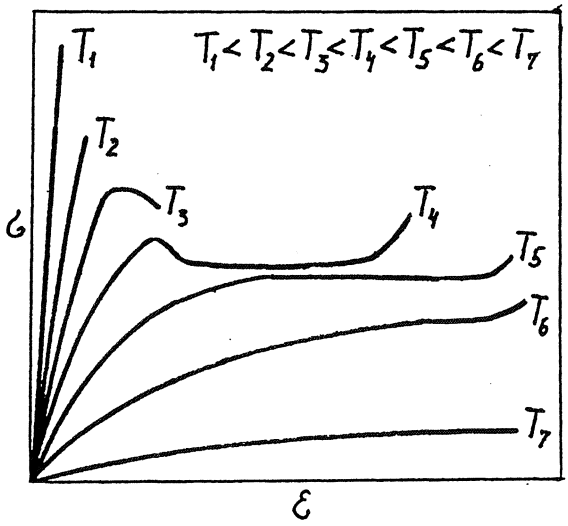

T1
T1<T2<T3<T4<T5<T6<T7
T2
T3
T4
T5
T6
T7
σ
ε

Fig. 2

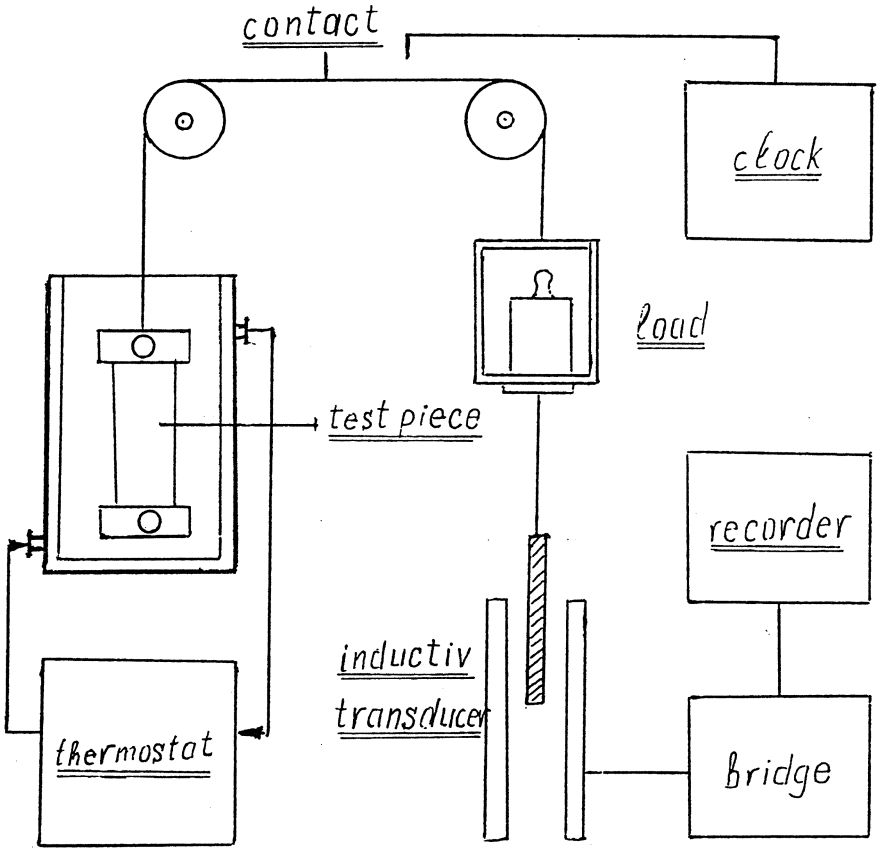

contact
clock
load
test piece
recorder
inductiv
transducer
thermostat
bridge

Fig. 3

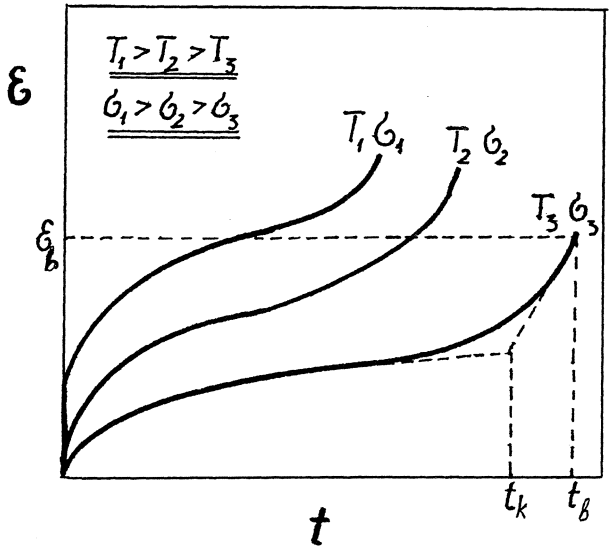

ε
T1 > T2 > T3
σ1 > σ2 > σ3
T1 σ1
T2 σ2
T3 σ3
εb
tk
tb
t

Fig. 4

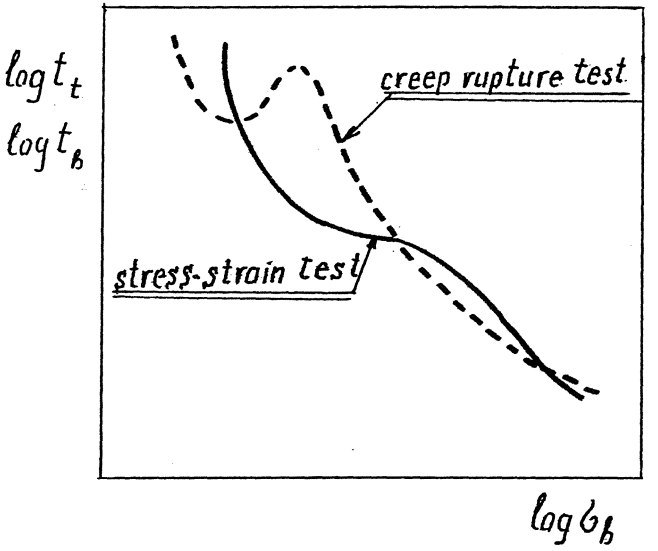

log tt
log tb
creep rupture test
stress-strain test
log σb

Fig. 5

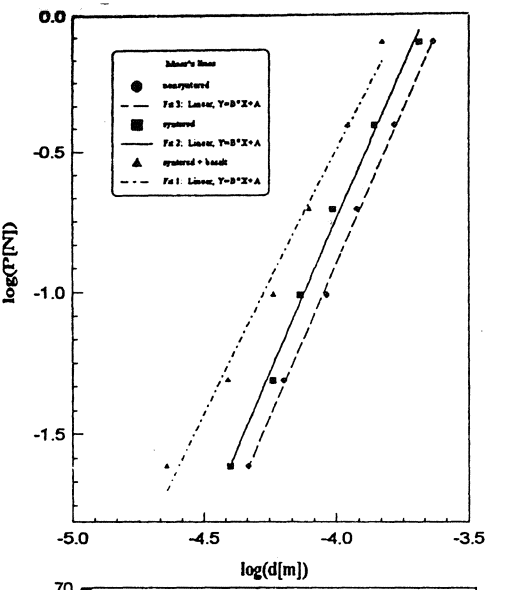

0.0
-0.5
-1.0
-1.5
log(P[N])
-5.0
-4.5
-4.0
-3.5
log(d[m])

Fig. 6

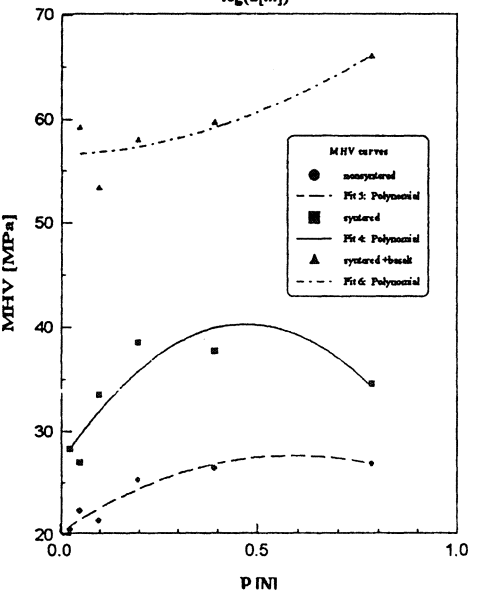

70
60
50
40
30
20
MHV [MPa]
0.0
0.5
1.0
P [N]
MHV curves
nonsystered
systered
systered +basalt

Fig. 7

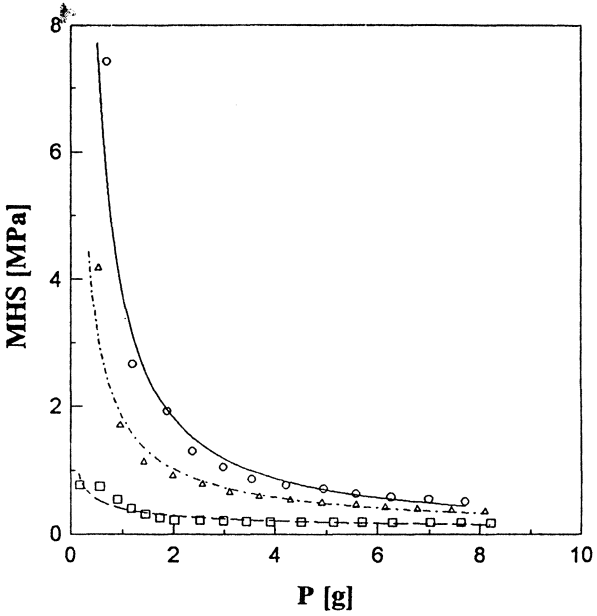

MHS [MPa]
P [g]
0
2
4
6
8
10

Fig. 8

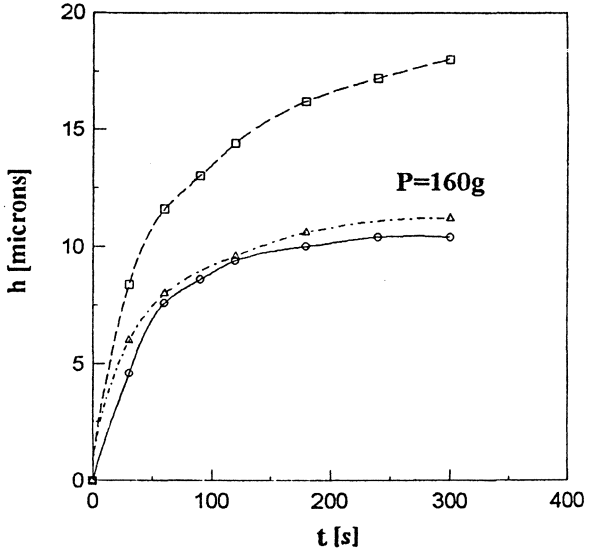

h [microns]
t [s]
P=160g
0
5
10
15
20
0
100
200
300
400

Fig. 9

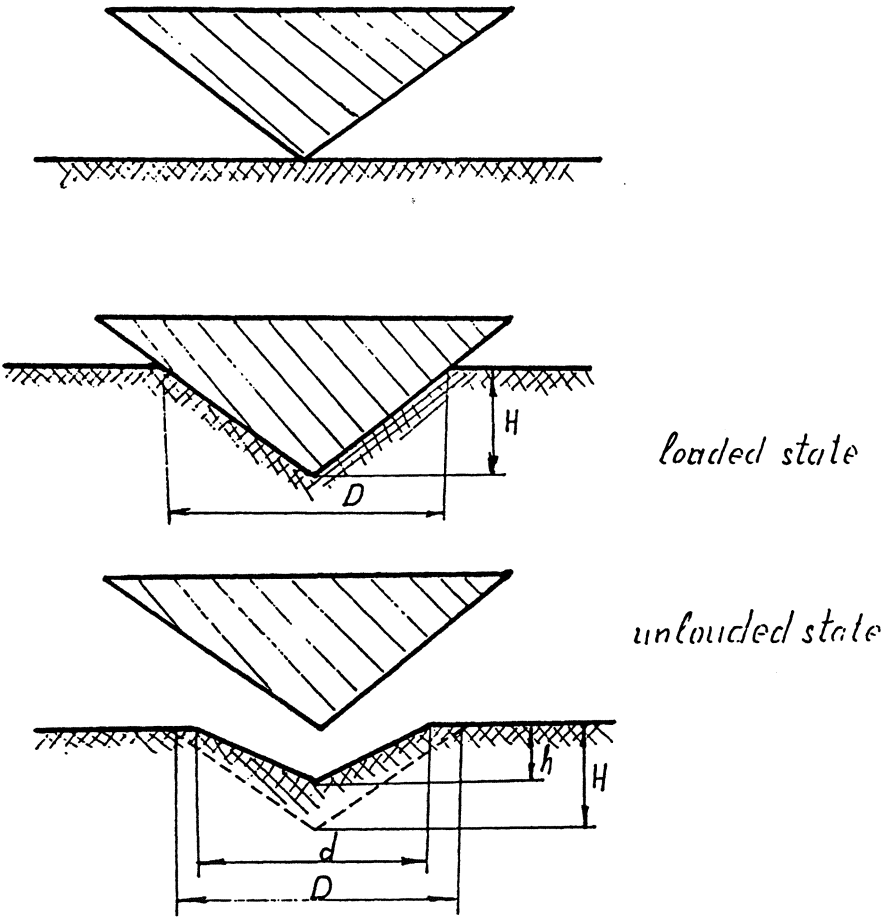


Fig. 10

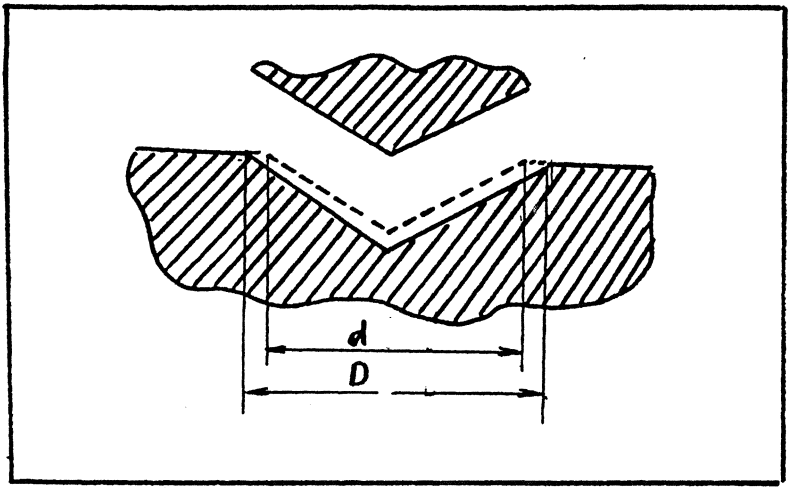
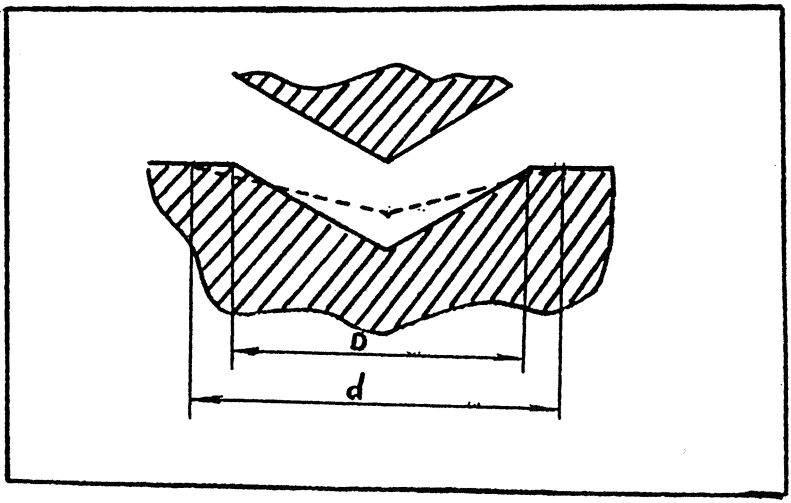


Fig. 9



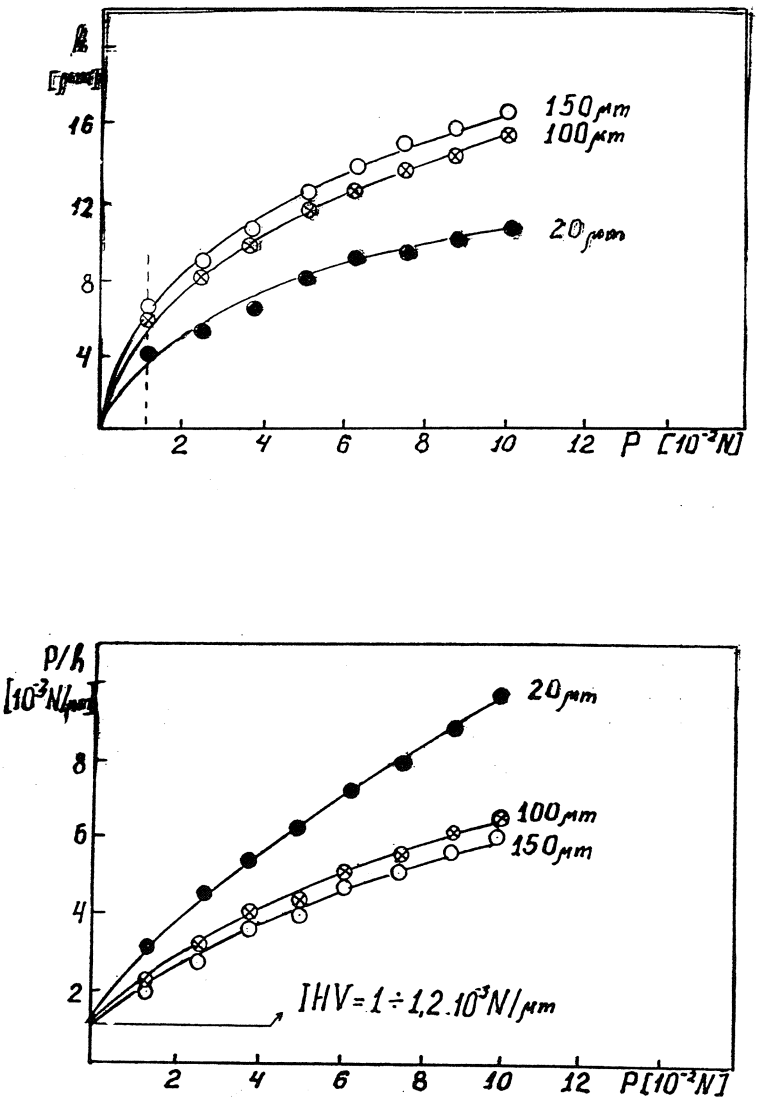

h
[μm]
16
12
8
4
2
4
6
8
10
12
P [10⁻²N]
150 μm
100 μm
20 μm
P/h
[10⁻²N/μm]
8
6
4
2
20 μm
100 μm
150 μm
IHV = 1 ÷ 1,2 .10⁻²N/μm
2
4
6
8
10
12
P[10⁻²N]

Fig. 11