Hardness Evaluation of Polytetrafluoroethylene Products

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Abstract. Influence of the strain rate on process of hardness measurement of teflon (PTFE) products by impact indentation method is considered. Possibility of hardness measurement at given value of intrusion depth is shown. Expression for coefficient of dynamics, taking account of influence of strain rate on results of hardness measurement is presented.

The goal of this paper is to consider the possibility of application of the dynamic indentation test method for the investigation of polytetrafluoroethelene materials (or PTFE), service properties of which depend on chemical composition and regime of heat treatment. It is known that among different test methods the most suitable ones for the evaluation of the properties of polymeric materials are indentation methods. In accordance with loading velocity indentation methods can be static or dynamic ones. Dynamic methods has number of advantages, the main of them are high test productivity and wide range of using. Moreover devices, based on these methods are small-sized and very convenient for using, because of the impact loading makes possible to produce rather high contact forces at small displacements. The last statement allows to consider impact dynamic devices as non-destructive ones and because of they can use for testing of finishing products.
Taking into account that the working pressure in PTFE products must to be no more than yield stress, the main parameters to be tested are hardness and yield stress of the material.
Hardness of PTFE in accordance with ISO 2039/1 is determined as the ratio of the maximum load $P_{\max}$ acting on the indenter by the square of impression A within 30 sec. of loading.

$$H = \frac{P_{\max}}{A} \quad (1)$$

The impression on PTFE surface is very difficult to be optically distinguished and for its measurements necessarily to create a special coating on the surface, to observe the impression in the reflection light. The standard ISO 2039/1-87 assumes also possibility to determine the square of impression projection using impression depth α with the help of displacement sensor. In this case hardness is calculated by follows:

$$H = \frac{P_{max}}{\pi D \alpha_{max}}, \quad (2)$$

where D is the diameter of the indenter tip, α_{max} is maximum of the indentation depth.
Disadvantage of equation (2) consists in dependence of measurement result on the indentation depth, because P_{max} and α_{max} are changed in non-proportional manner. In fig 1 the behaviour of changing of the static hardness H_s against P_{max} for the samples from PTFE is presented. The measurements carried out on Vickers hardness meter using the

indenter with tip diameter equal to 5 mm. at the loading ranged from 50 to 400 N. Simultaneously the registration of the indenter displacements was conducted. As can see in fig.1 at the same load we received different values of hardnesses.

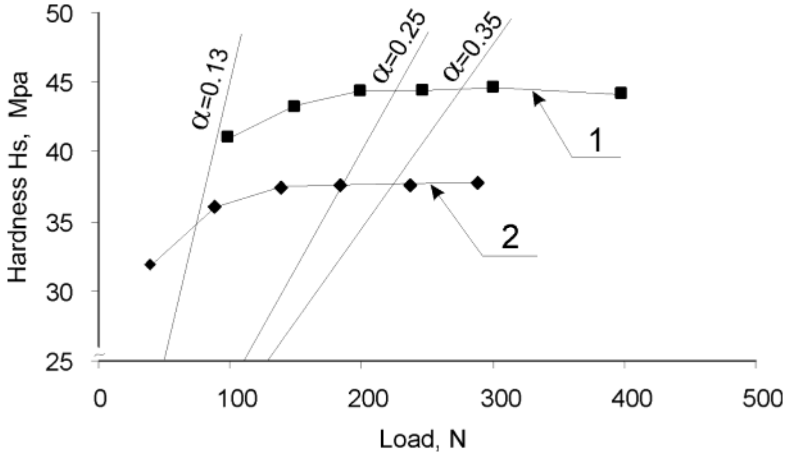

Fig. 1. Dependence of the static hardness versus load. 1- nonhardened and 2 – hardened PTFE.

For the removal of this ambiguity it was adopted to determine hardness at given constant value of the indentation depth equal to 0,25mm. According to this proposal the loadings should be equal to 49,1; 132; 358; 961N. Indentation depth should be in a range from 0,13 to 0,35 mm and the loading corresponding to the depth of 0,25 have to be determined by means of recalculation [1]. Straight lines in fig.1 corresponding indentation depths α=0,13, 0,25 and 0,35 mm are shown.

The process dynamic indentation is different from static one. During dynamic indentation additional resistance to indenter intrusion is occurred, which depends on impact velocity and chemical composition of polymeric material. Hard polymeric materials are deformed in plasticity manner when contact pressure is more than its yield stress. As it was experimental established, residual impression can be seen on tested surface of PTFE materials in the research temperature range from –10 up to +80 C°. In this case elastic-plastic properties are more pronounced than viscous-elastic ones. The last is confirmed by a small change of impression diameter measured immediately at the impact and after some minutes. However at temperature more than 80 C° change in impression diameter is increased up to 7%.

In fig. 2 dependence of the dynamic hardness, calculating from formula (2) versus different loading values is shown Variation of loading is carried out by the change of initial impact velocity V .The testing was made with the help installation IMPULSE - 1R which was designed in the Institute of Applied Physic of the National Academy of Science of Belarus and NPO Energomash, Khimki (Russia). As can see character of smooth increasing of dynamic and static hardness against loading is the same in considered range of contact forces up to 60 N, but values of dynamic hardness always higher than static one. In order to explain this excess it can use the model of polymeric materials deforming at different velocities [2]. In accordance with the model it can write expression for pressure, which equal yield stress, as follows

$$\sigma_{\text{d}} = A + B\ln[a_{\text{T}}(\bar{\varepsilon}'_{\text{d}}/\bar{\varepsilon}'_{\text{cr}})], \tag{3}$$

where A, B and a_{T} are coefficients which depend on chemical composition and temperature, $\bar{\varepsilon}'_{\text{cr}}$ and $\bar{\varepsilon}'_{\text{d}}$ are mean strain rates at dynamic and static intrusion on given indentation depth.

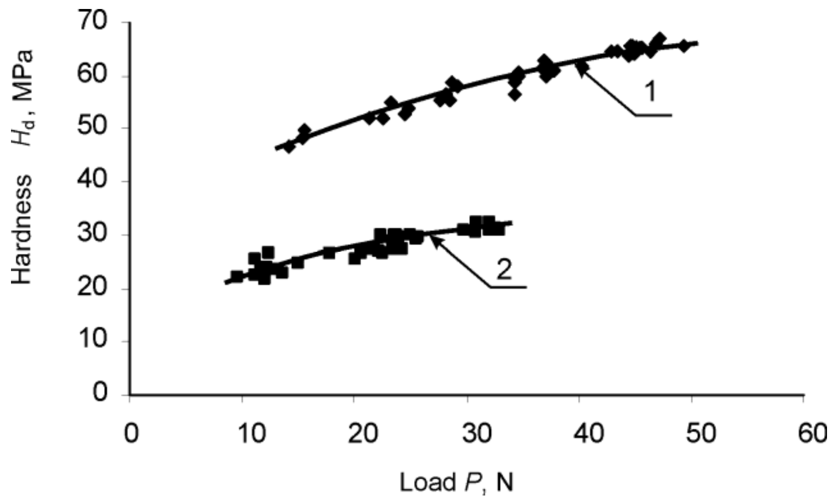

Fig. 2. Dependence of the dynamic hardness versus load. 1- nonhardened and 2 – hardened PTFE.

After some transforms we obtain

$$\sigma_{\text{d}} = \sigma_{\text{ст}} + B\ln(\bar{\varepsilon}'_{\text{d}}/\bar{\varepsilon}'_{\text{ст}}), \tag{4}$$

where $\sigma_{\text{ст}} = A + B\ln(a_{\text{T}})$ is pressure at static loading. As can see in the case, when $\bar{\varepsilon}'_{\text{ст}} = \bar{\varepsilon}'_{\text{d}}$ static and dynamic pressure will be equal $\sigma_{\text{d}} = \sigma_{\text{ст}}$.

Taking into account that hardness is pressure, measuring at certain strain, equation (4) can be wrote as follows

$$H_{\text{d}} = H_{\text{ст}} + B\ln(\bar{\varepsilon}'_{\text{d}}/\bar{\varepsilon}'_{\text{ст}}) \tag{5}$$

Since hardness value is function strain, it is necessary to determine strain value corresponding strain applied in above mention the static test at intrusion $\alpha = 0{,}25$ mm. Evaluating strain value as

$$\varepsilon = 0{,}2\frac{d}{D}, \tag{6}$$

where $d = 2\sqrt{D\alpha}$ is diameter of impression projection, we determine $\varepsilon = 0{,}087$.

With the help of equation (5) we can calculate intrusion depth at given strain and diameter of the indenter tip

$$\alpha = 6{,}25D\varepsilon^2 \tag{7}$$

In installation IMPULSE - 1R diameter D of the indenter tip is 1mm.and formula (6) show that α should be no less than 50 μm. In fig 3 typical diagrams: contact force versus displacement, which were obtained during impact test of PTFE products are presented.

From the fig. 3 it can see follows the characteristic feature of PTFE. The dependence of the contact force versus penetration on the active stage of impact is invariable on initial velocities of the impact in the range from 0,5 to 1,5 m/s. This fact confirms that the properties of PTFE are very closely to the properties of elastic-plastic materials. As can be seen, the dependences of the contact force vs penetration are similar and by only the values of maximum forces and penetrations are characterized. It means that at the given strain rates the second term in the equation (5) will be in insignificant degree depend on the strain rate at the dynamic testing.

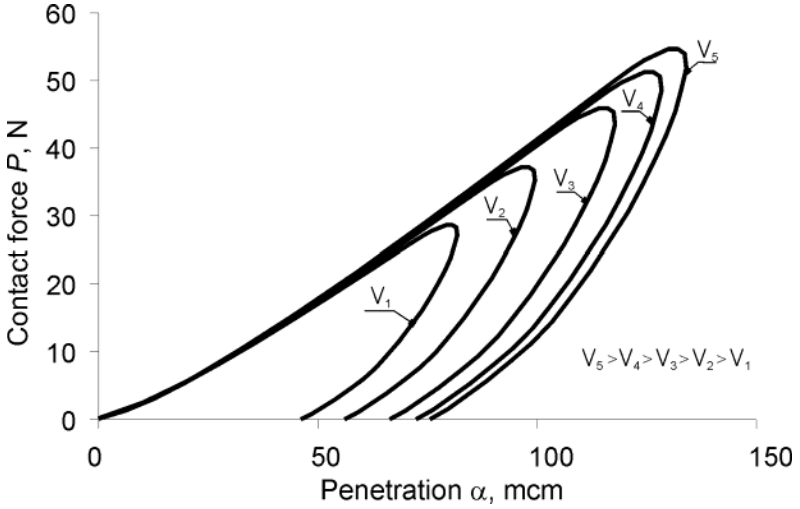

Fig. 3. Typical dependencies contact force-penetration at the dynamic testing of PTFE with different initial velocity: 1 – 0,5; 2 – 1,0; 3 – 1,2; 4 –1,35; 5 – 1,5 m/s.

Actually, the estimation of the values of the strain rate at the testing with the help of static and dynamic method showed that the value of logarithm in (5) changes in the limits from 7,5 to 10. At the static testing according to ISO 2039/1 load is applied during 1-2 seconds, after that the load is sustained by the 30 second. During this process the diameter of the impression increases due to creep, but the finishing value of the impression on the hardened PTFE is greater than initial impression but not more than 4 %. Taking into account that the deformation, which necessary to attain at the tests, is 0,087, the average rate of the static strain will be $\frac{\varepsilon}{t} = \frac{0,087}{1,5} \approx 0,06\ \text{s}^{-1}$. The average strain rate at the dynamic testing at the temperature in the range of -10 to + 80 ^{0}C will be varied in the limits of 100-600 s^{-1}.

In the Figure 4a the change of the values of logarithm $\ln(\bar{\varepsilon}'_d / \bar{\varepsilon}'_s)$ versus initial velocity of the indenter V_0 for one of the PTFE samples is shown.

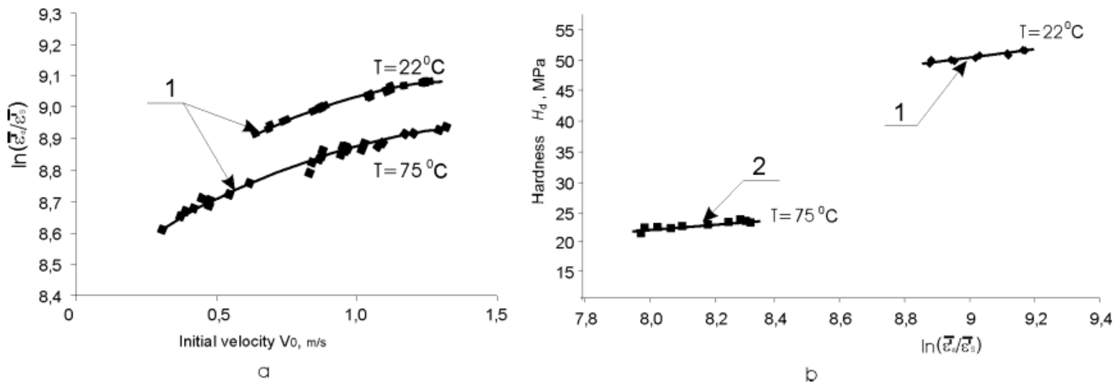

Fig. 4. a) Dependence of $\ln(\bar{\varepsilon}'_d / \bar{\varepsilon}'_s)$ in the equation (5) versus initial velocity of the indenter; b) dependence of the dynamic hardness against $\ln(\bar{\varepsilon}'_d / \bar{\varepsilon}'_s)$. 1- nonhardened and 2 – hardened samples.

The change of $\ln(\bar{\varepsilon}'_d / \bar{\varepsilon}'_s)$ is small, although slightly depends on the temperature, strain rate, and degree of crystallinity (i.e. structure and properties of PTFE). In the figure 4b the change of the dynamic hardness for the samples of PTFE with different degree of crystallinity at the different temperatures is shown. As it follows from the dependences, reflected in figure 4a, the difference in the value of logarithm $\ln(\bar{\varepsilon}_d^{'}/\bar{\varepsilon}_s^{'})$ is characteristic only for PTFE in the whole investigated temperature and strain rate ranges, and furthermore it depends on the degree of crystallinity (i.e. structure and of the properties of PTFE). Consequently, the real change in the hardness, caused by the change in the strain rate (in the investigated range of initial velocities from 0,5 to 1,5 m/s) insignificantly and does not exceed 5 %. It means that the value of $\ln(\bar{\varepsilon}_d^{'}/\bar{\varepsilon}_s^{'})$ can be accepted to be constant at a certain temperature.

The change of the hardness at different temperatures will be determined by the structural and temperature-sensitive coefficient **B.** In this case equation (5) will be exact not only for the average values of the strain rate, but also for its instantaneous values $\varepsilon^{'}(t)$ at the given moment of time. This gives an opportunity to determine the static hardness using the dynamic measurements.

In the Figure 5 the change of the hardness in the process of the indenter intrusion at the dynamic and static tests is shown. As can be seen from the figure the value of dynamic hardness (stress) differs and higher the static stress on the certain practically constant value. This is the result of the fact that the change of the logarithm in the given range of strain rate differs insignificantly.

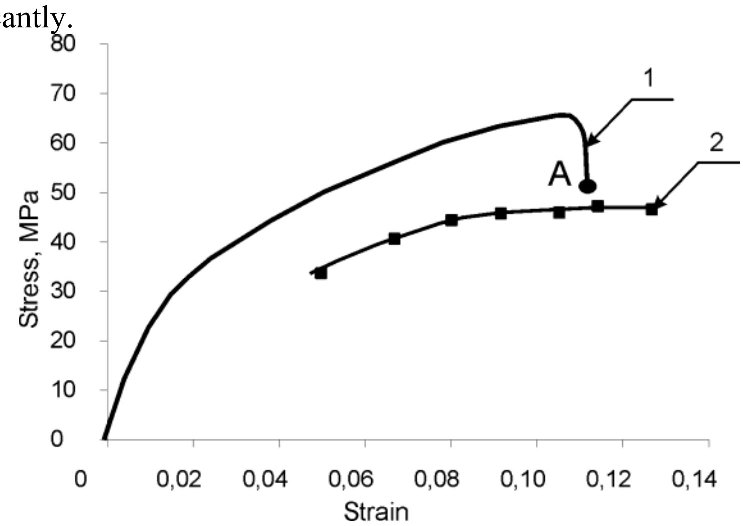

Fig. 5. Dependence of stress versus strain at the dynamic (1) and static (2) testing.

Furthermore, at the sharp reduction of the strain rate at the end of the impact until zero, static and dynamic hardness practically coincide (point A in the figure 5). The small exceeding of dynamic hardness in this case can be explained by the influence of the viscous properties of PTFE, which were not taken into consideration. In practice, as a rule, accepted to evaluate the properties of material by the static hardness value. Taking into account that the strain rate at the dynamic measurements practically does not changes, the value of logarithm $\ln(\bar{\varepsilon}_d^{'}/\bar{\varepsilon}_s^{'})$ can be considered as the constant, equal to 8,6. After division of equation for H_d by H_s we will obtain that $\frac{H_d}{H_s} = K_d$, where $K_d = 1 + B\ln(\bar{\varepsilon}_d^{'}/\bar{\varepsilon}_{cm}^{'})/H_s$ is dynamic coefficient for the given temperature. The existence of this coefficient speaks about the possibility of the determination of the static hardness from the results of its determination at the dynamic testing. In the figure 6 the experimental dependence, obtained at the testing of PTFE with different degree of crystallinity in the range of temperatures from -10 to +80 ^{0}C is shown.

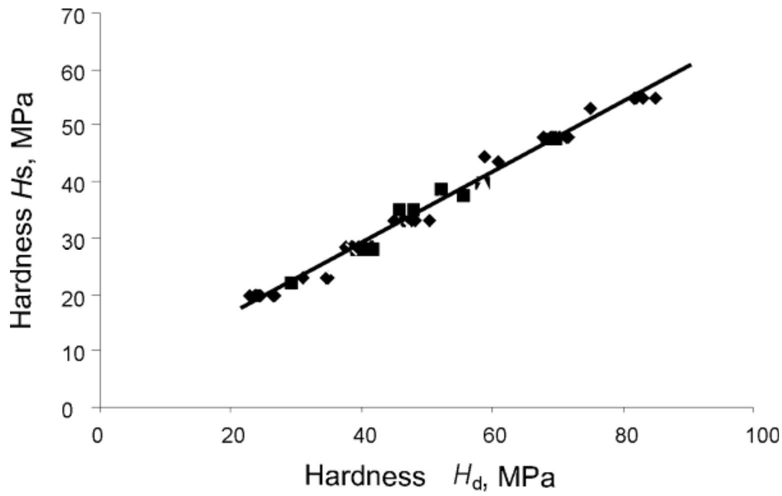

Fig. 6. Relation between the static and dynamic hardness for PTFE with different crystallinity.

Correlation coefficient of this dependence is 0,982 and presented dependence can be described by the equation (8).

$$H_s = 0{,}624 H_d + 4{,}511 \text{ (MPa)} \quad (8)$$

As practice showed, the error in the determination of static hardness from the results of dynamic measurements does not exceed 6%. In this connection it is possible to assert that at the condition $\varepsilon_s = \varepsilon_d$ the static hardness can be found with simple conversion with the help of equation (8).

References

[1] ISO 2039/1-2001 Plastics. Determination of hardness. Ball indentation method
[2] *Racke H.H., Fett T.* / Materialprufung, 1968. Bd. 10, N 7, P.226–230.