UDK 620.178.2: 537.226.86

Construction of Stress-Strain Curves for Brittle Materials by Indentation in a Wide Temperature Range

Yu. V. Milman*, I. V. Gridneva, A. A. Golubenko
Frantsevich Institute for Problems of Materials Science, National Academy of Sciences of Ukraine, 3, Krzhyzhanovsky St., Kyiv-142, 03680, Ukraine

Abstract:

A test method procedure for constructing stress-strain curves by indentation of brittle and low plastic materials under temperature ranging from 20 to 900 °C was developed recently by Yu. Milman, B. Galanov et al. According to this test method procedure stress-strain curves $\sigma - \varepsilon$ for Si, Ge, SiC, TiB$_2$ and WC/Co hard alloy were constructed in the above temperature region and mechanical parameters such as elastic point, $\sigma_e$, yield stress, $\sigma_y$, etc. were extracted by using the measurement results obtained by a set of trihedral pyramid indenters with different angles at the tip, $\gamma_1$, ranging from 45 to 85°.

Keywords: Indentation, Brittle materials, Stress-strain curve, Strain hardening, Elastic deformation.

Introduction

The possibilities to determine the mechanical properties of brittle materials by traditional methods are rather limited in view of their brittle fracture under stresses close to or even much lower than the yield stress.

However, since the development of a procedure of construction of stress-strain curves by indentation method by the authors of the present investigation, the possibility to obtain stress-strain curves and determine the strength and plasticity characteristics of brittle materials arose.

The major idea of the method consists in using pyramidal indenters sharpened to different vertex angles (the change of the vertex angle enables variation of the degree of deformation under the indenter from 2 to 30% of the total strain $\varepsilon_t$).

The use of each indenter enables obtaining of one point of the stress-strain curve. Each indenter enables determination of the average value of Meyer hardness HM, to which a specific strain $\varepsilon_i$ corresponds. The yield stress $\sigma$ is calculated from the value of HM using the Tabor relation $HM \approx 3\sigma$ [1].

Procedure and Experiment

In the investigation, nine trihedral indenters with angles between the face of pyramidal indenter and its axis $\gamma_1 = 45, 50, 55, 50, 65, 70, 80, 85^\circ$ were used. Hardness

* Corresponding author: milman@ipms.kiev.ua
tests were performed on PMT-3 microhardness testers at room temperature, and in tests in the
temperature range 20–900 ºC, a vacuum unit (hot hardness) was employed.
Measurement of hardness indentations and a study of deformation zones near
indentations were carried out on a MIM-10 metallographic microscope using a digital camera
with further computer data processing.
It should be noted that for brittle materials, not all of the nine indenters can be used,
since at large degrees of deformation, intensive brittle fracture of indentations occurs.
The total strain $\varepsilon_t$ depends on the plastic strain $\varepsilon_p$ and the elastic strain $\varepsilon_e$
$$\varepsilon_t = \varepsilon_p + \varepsilon_e \tag{1}$$
We used the following relations [2, 3],
$$\varepsilon_p = -\ln \sin \gamma_2, \tag{2}$$
where $\gamma_2$ is the angle between the face of the pyramidal hardness indentation and the axis of
loading.
The inequality $\gamma_2 > \gamma_1$ always holds.
$$\ctg \gamma_2 = \ctg \gamma_1 - 1.77 \frac{HM}{E_{ef}}, \tag{3}$$
where $E_{ef}$ is the effective modulus of the indenter–specimen contact pair, $HM$ is the Meyer
hardness:
$$\frac{1}{E_{ef}} = \frac{1 - v_1^2}{E_1} + \frac{1 - v_2^2}{E_2} \tag{4}$$
where $E_1$ and $v_1$ – Young modulus and Poisson’s ratio for a material of the specimen,
$E_2$ and $v_2$ – the same characteristics for material of the material of the indenter.
Elastic deformation $\varepsilon_e$ is determined from the equation:
$$\varepsilon_e = (1 - v_1 - 2 v_1^2) \frac{HM}{E_1}, \tag{5}$$
It is worthy to note that the shape of hardness indentations and the degree of plastic
(or elastic) deformation under the indenter for metals differ significantly from those for
ceramic materials. For metals, the angle $\gamma_2$ between the axis of the pyramidal indentation and
its face is slightly higher than the corresponding angle of the indenter $\gamma_1$. The value of elastic
strain is not larger than 0.5%, and plastic strain contributes to the total strain. At the same
time, in ceramic materials, angle $\gamma_2$ is much larger than angle $\gamma_1$, and the fraction of elastic
strain under the indenter is rather significant.
Depending on the material, one can determine the upper and the lower yield
strength, the limit of proportionality, the fracture stress and other characteristics on $\sigma$–$\varepsilon$
curves. Determination of strain hardening in each portion of the curve becomes possible.

Results and Discussion

Stress–strain curves for brittle (in standard mechanical tests) materials were
constructed in a wide temperature range. In Fig. 1, stress–strain curves for brittle single-
crystal silicon are presented.

The existence of a critical temperature $T_c = 300–400^\circ$C, above which stress–strain
curves have a shape characteristic of crystalline materials with strain hardening attaining
100%, was established. Below $T_c$, strain hardening is practically absent. Such a character of
stress-strain curves agrees well with the phenomenon of semiconductor→metal phase
transition revealed earlier by the authors [4]. Thus, it was shown that at $T > T_c$, the measured
hardness corresponds to the yield stress, whereas at $T < T_c$, the value of hardness corresponds
to the pressure of the phase transition and does not reflect the value of the yield stress. At \( T < T_{cr} \), the value of hardness is independent of the apex angle of the indenter \( \gamma_1 \), i.e., of the degree of total strain \( \varepsilon_t \) under the indenter.

![Stress–strain curves for single-crystal silicon at different temperatures under an indentation load \( P = 1.15 \) N.](image1)

In an analysis of the stress–strain curves obtained for silicon at 600 ºC and above, it was established that parabolic strain hardening is characteristic for them from the onset of plastic flow. That is why the \( \sigma-\varepsilon \) curves can be approximated by the Ludwik equation [5]

\[
\sigma = \sigma_s + N \varepsilon_p^n,
\]

where \( \sigma_s \) is the lower limit of proportionality, \( \varepsilon_p \) is the plastic strain, \( N \) is the strain hardening coefficient, and \( n \) is the strain hardening index.

In the case of the dislocation mechanism of deformation [6], the strain hardening coefficient is described by the formula

\[
N = \alpha G b^{1/2} l^{-1/2},
\]

where \( \alpha \) is the constant close to 1; \( G \) is the shear modulus, \( b \) is Burgers vector; \( l \) is the average length of the slip plane; \( n \approx 0.5 \). As the test temperature increases, \( N \) decreases as a result of decreases in \( \alpha \) and \( G \).

![Curve \( \sigma-\varepsilon_p \) in logarithmic coordinates for single-crystal Si.](image2)

In Fig. 2, a \( \sigma-\varepsilon_p \) curve typical of Si constructed in logarithmic coordinates at 600ºC (where the value of the plasticity appears to be sufficient for the determination of \( N \) and \( n \)) is
shown. Here points are located on the straight line, which gives grounds to use the Ludwik equation in the determination of \( N \) and \( n \), whose values at different temperatures are presented in Tab. I. Actually, as the test temperature is increased, \( N \) decreases, and the values of \( n \) are close to 0.5.

**Tab. I. Values of the strain hardening coefficient \( N \) and the strain hardening index \( n \) for single-crystal Si**

<table>
<thead>
<tr>
<th>Temperature, ºC</th>
<th>( n )</th>
<th>( N ), GPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>600</td>
<td>0.42</td>
<td>2.72</td>
</tr>
<tr>
<td>700</td>
<td>0.55</td>
<td>2.37</td>
</tr>
<tr>
<td>800</td>
<td>0.45</td>
<td>1.08</td>
</tr>
<tr>
<td>900</td>
<td>0.56</td>
<td>0.92</td>
</tr>
</tbody>
</table>

It is known [6] that if the strain hardening index is equal to 0.5, then this is the indicator of the dislocation mechanism of deformation.

In Fig. 3, a dependence of strain hardening \( \Theta \) and \( \sigma_p \) on the degree of plastic deformation for single-crystal Si is presented. As can be seen from Fig. 3, strain hardening \( \Theta = \frac{d\sigma}{d\varepsilon_p} \) decreases abruptly as the strain \( \varepsilon_p \) increases, and at \( \varepsilon_p \approx 12\% \), \( \Theta = \sigma_p \). This equality determines the condition of the deformation localization and thus the procedure used enables determination of the strain at which the localization of plastic flow becomes essential.

**Fig. 4** Stress-strain curves for single-crystal Ge at different temperatures under an indentation load \( P = 1.15 \) N.
Stress–strain curves for single-crystal germanium at temperatures from 20 to 800 °C are given in Fig. 4.

In the construction of stress–strain curves of such a brittle material as Ge, we managed to use only three indenters in the temperature range 20–300 °C, five in the range 400–500 °C, and six in the range 600–800 °C. The indenters with acute vertex angles $\gamma_1 = 45$, 50, 55° caused the fracture of indentations at low temperatures, whereas the indenters with obtuse vertex angles $\gamma_1 = 85$, 80° did not exert a plastic effect due to a large contribution of the elastic strain in the formation of indentations. For this reason, at some temperatures, stress-strain curves were constructed in the incomplete possible strain range from 2 to 30%.

At temperatures 20, 200, and 300 °C, hardness indentations form predominantly due to fracture processes (in the same temperature range, a phase transition is observed); that is why the curves were constructed for small values of strains at which fracture is not so essential.

Stress-strain curves for SiC- and TiB$_2$-base brittle ceramics were also constructed. For such materials, standard mechanical tests make it impossible to obtain stress-strain curves below the cold-brittleness temperatures, which for most ceramic materials are higher than 1000 °C.

In Fig. 5, stress-strain curves for TiB$_2$-based ceramics at temperatures 20, 400, 600, 800, and 900 °C are shown.

![Stress-strain curves for TiB$_2$-base ceramics at different temperatures and under an indentation load $P = 2.34$ N.](image)

In the construction of the stress-strain curves for this material, seven of nine indenters were used. Due to fracture processes, we failed to get a hardness indentation with the most acute indenter with $\gamma_1 = 45^\circ$ as at low temperatures ($<400$ °C) we failed to use a blunt indenter with $\gamma_1 = 85^\circ$ (due to a large contribution of the elastic strain in the formation of the hardness indentation).

At room temperature, at a strain $\varepsilon_t > 10\%$, brittle fracture processes are significant in the formation of hardness indentation; that is why the stress-strain curve at 20 °C shows essential scattering. Beginning from 400 °C and above, the stress-strain curves enables determination of strain hardening. In Tab. II, values of strain hardening coefficients $N$ and strain hardening indices $n$ are shown.

<table>
<thead>
<tr>
<th>Temperature, °C</th>
<th>$n$</th>
<th>$N$, GPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>400</td>
<td>0.42</td>
<td>6.94</td>
</tr>
<tr>
<td>600</td>
<td>0.50</td>
<td>6.67</td>
</tr>
<tr>
<td>800</td>
<td>0.51</td>
<td>5.26</td>
</tr>
<tr>
<td>900</td>
<td>0.52</td>
<td>5.13</td>
</tr>
</tbody>
</table>
As the test temperature rises, $N$ slightly decreases, and the value of $n$ (as in the case of single-crystal Si) is close to 0.5, which indicates the dislocation mechanism of deformation of this ceramics.

**Fig. 6** Typical hardness indentations of TiB$_2$ ceramics obtained using trihedral indenters with different vertex angles at different temperatures.

In Fig. 6, typical hardness indentations of TiB$_2$ ceramics obtained with triangular
indenters with different vertex angles at different temperatures are presented. At low temperatures and large strains, indentations are accompanied with cracks; as the test temperature increases, the TiB$_2$ ceramics becomes more plastic, and indentations form almost without fracture.

As in the case of TiB$_2$ ceramics, in the investigation of SiC ceramics, we managed to use seven of nine indenters for the same reasons. The constructed stress–strain curves are shown in Fig. 7. The scattering in values of $\sigma$ is caused by brittle fracture processes accompanying the formation of indentations, particularly in the cases of using indenters with acute vertex angles, i.e., at degrees of deformation $\varepsilon > 10\%$ (Fig. 7).

![Stress–strain curves for SiC ceramics at different temperatures under an indentation load $P = 2.34$ N.](image)

Fig. 7 Stress–strain curves for SiC ceramics at different temperatures under an indentation load $P = 2.34$ N.

In contrast to TiB$_2$ and SiC ceramics, the WC-Co alloy is a composite material, which determines specific features of its mechanical properties and the mechanism of deformation. For the WC-6%Co alloy, the Meyer hardness was tested in the temperature range 20-800 $^\circ$C, and stress-strain curves were constructed (see Fig. 8). In this case, it was possible to use all 9 indenters.

![Stress–strain curves of the WC-6%Co alloy at different temperatures.](image)

Fig. 8 Stress–strain curves of the WC-6%Co alloy at different temperatures.

As shown in [7], among ceramic materials, WC-Co alloys are characterized by the highest plasticity $\delta_H \approx 0.8$. That is why, for WC–6%Co, it was possible to obtain hardness indentations without fracture in the investigated strain range 2–33%.
At all temperatures, the stress-strain curves are parabolic in character, which enabled us to determine strain hardening at all temperatures.

**Tab. III.** Strain hardening coefficients $N$ and strain hardening indices $n$ for the WC-6%Co alloy at different temperatures

<table>
<thead>
<tr>
<th>Temperature, ºC</th>
<th>$n$</th>
<th>$N$, GPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>0.45</td>
<td>2.67</td>
</tr>
<tr>
<td>200</td>
<td>0.40</td>
<td>2.26</td>
</tr>
<tr>
<td>400</td>
<td>0.42</td>
<td>2.00</td>
</tr>
<tr>
<td>600</td>
<td>0.36</td>
<td>1.98</td>
</tr>
<tr>
<td>800</td>
<td>0.41</td>
<td>1.77</td>
</tr>
</tbody>
</table>

In the calculation of $N$ for the WC-6%Co hard alloy, it was taken into account that only the cobalt interlayer (10 vol. %) deforms and the length of the slip plane is equal to the thickness of the cobalt interlayer $\lambda \approx 0.3$ µm. In this case, the experimentally determined values of $N$ (see Tab. III) practically coincided with data calculated by formula (7). The values of the strain hardening coefficient $N$ decrease with temperature, whereas the values of the strain hardening index $n$ are close to 0.4.

**Conclusions**

On the basis of the developed procedure, for brittle materials, namely Si, Ge, SiC, TiB$_2$, and a WC-Co hard alloy, stress-strain curves in a wide temperature were constructed.

For single-crystal Si and Ge, critical temperatures $T_{cr}$ of about ~300-400 ºC for Si and equal to 300 ºC for Ge, below which the hardness is determined by the phase transition and above which it is determined by the yield stress, were established.

Parameters of strain hardening for Si, TiB$_2$, and the WC-Co alloy in the temperature range 200-900 ºC were first determined. It was established that for all these materials, the strain hardening index $n \approx 0.5$, which indicates the dislocation mechanism of deformation. At the same time, the strain hardening coefficient $N$ increases in the sequence Si→WC-Co→TiB$_2$. Moreover, for all investigated materials, $N$ decreases as the test temperature increases. The effective strain hardening coefficient $N_{ef}$ was calculated taking into account the fact that the WC-6%Co alloy is a composite material, in which WC is the non deformable phase, and the ductile Co content is 10 vol. %.

Thus, the developed procedure makes it possible to construct stress-strain curves of brittle materials in wide range of the temperatures and analyze their mechanism of deformation.

**Acknowledgement**

The authors are grateful to Dr. S. Chugunova and Mrs. I. Goncharova for experimental help and useful discussion of results.

**References**


---

**Садржај:** Тест процедура за конструисање кривих оптерећење-напрезање озубљавањем кртих и слабо пластичних материјала у температурном интервалу од 20 до 900 °C је скоро развјена од стране Милмана, Галанове и других. На основу ове тест процедури конструисане су криве оптерећење-напрезање $\sigma$-$\varepsilon$ за Si, Ge, SiC, TiB$_2$ и WC/Co тврду легуру у датом температурном интервалу и одређени су механички параметри као што је еластична тачка, $\sigma_e$, област флуидности $\sigma_s$, и друге вредности добијене измереним величинама из скупа триедарских пирамидних озубљивача са различитим углом шили 1, у интервалу од 45 до 85°.

**Кључне речи:** Озубљавање, крти материјали, крива оптерећење-напрезање, напон закаљења, еластична деформација.