Application of the Plasticity Characteristic Determined by the Indentation Technique for Evaluation of Mechanical Properties of Coatings: I. Specific Features of the Test Method Procedure

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Abstract: Specific features of the test method procedure capable for determining the plasticity characteristic $\delta_H$ by indentation of inhomogeneous coatings affected by residual stress was clarified. When the value of the plasticity characteristic for coating was found to be as great as $\delta_H > 0.5$ a simplified model was found to be reasonably adequate, while a modified model assumed compressibility of the deformation core beneath indentation. The advantage of the modified approach compared to the simplified one was grounded experimentally only if the elastic deformation for coating becomes greater than $\varepsilon_e \approx 3.5\%$, resulting in the decrease of plasticity characteristic $\delta_H < 0.5$. To overcome non accuracy caused by the effect of the scale factor on measurement results a comparison of different coatings was suggested using stabilized values of the plasticity characteristic $\delta_H$ determined under loads higher than critical, $P \geq P_c$, ensuring weak dependence of microhardness values on the indentation load.

Keywords: Coating; Mechanical properties; Plasticity characteristic.

1. Introduction

Mechanical properties of coatings are of great importance for predicting their behaviour under loading during exploitation. For a long time this problem was usually limited by application of microhardness measurement results because the coating thickness was too small (typically $10^{-1}$-$10^{-3}$mm). Effective development of the theory and practice of the indentation technique has given a wide scope for determining mechanical properties of coatings [1-24]. Besides microhardness, determination of a complete set of mechanical parameters, which is important for entire controlling of the coating resistance to failure, was enabled by the indentation technique. Among them characteristics relevant to evaluation of plasticity properties of coatings are the most helpful. It is notable that excluding the indentation technique no more mechanical tests are capable of evaluating the plasticity for a number of covalent crystals and ceramic materials because of their brittleness.

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Correlation of Meier hardness, \( H_M \), with material yield stress, which corresponds to the deformation averaged over the indentation contact area and determined in the direction of loading, was originally found by Tabor \([1, 2]\). Using load-displacement indentation employed usually for determining the hardness number through penetration depth it was shown clearly that two different components of deformation, elastic and plastic, contribute directly to the microhardness value, if pyramid indenters have been used for material testing \([6, 7]\). Furthermore, contribution of the elastic component tends to be quite small (smaller than about 10%) for metals whilst the opposite is true for high-strength and brittle materials, which have a high value of the covalent component of the atomic bond. During testing of materials such as covalent crystals, ceramics and ceramic coatings, the plastic component does not achieve even the half of the total deformation under indentation and varies substantially depending on the material nature \([6, 7, 17]\).

The dimensionless parameter, which defines the share of the plastic deformation, \( \varepsilon_p \), in the total deformation under indentation, \( \varepsilon = \varepsilon_e + \varepsilon_p \), has been suggested in \([10]\) as the plasticity characteristic, \( \delta_H \), quantifying the material ability to change its shape:

\[
\delta_H = \frac{\varepsilon_p}{\varepsilon} = 1 - \frac{\varepsilon_e}{\varepsilon} \quad (1)
\]

where \( \varepsilon_e \) is the elastic deformation.

The theory in \([10]\) states that for indenters of a pyramid shape the total deformation operating over the indentation is defined mainly by the angle \( \gamma_1 \) at the indenter tip, i.e. the angle between the indenter face and its axis. For the Vickers indenter it is as great as 68\%:

\[- \varepsilon = \ln \sin \gamma_1 \approx 7.6\% \quad (2)\]

According to the model \([10]\) the participation of the elastic deformation, \( \varepsilon_e \), is given by the equation:

\[- \varepsilon_e = (1 - \nu_1 - 2\nu_1^2) \frac{H_M}{E_1} \quad (3)\]

where \( E_1 \) and \( \nu_1 \) are Young’s modulus and Poisson ratio of the material, respectively.

Parameter \( \delta_H \) can be calculated by substituting Eqs. (2) and (3) in Eq. (1).

When Meier hardness in Eq. (3) is replaced by Vickers hardness \( H_V = H_M \sin \gamma_1 \), the following equation for calculating the plasticity characteristic can be determined:

\[
\delta_H = 1 - 14.3(1 - \nu_1 - 2\nu_1^2) \frac{H_V}{E_1} \quad (4)
\]

A modified theoretical approach \([13]\) was developed to improve the expression for determining the \( \delta_H \) parameter if the contribution of the elastic deformation, \( \varepsilon_e \), to the total deformation, \( \varepsilon \), is the greatest, such as for ceramics and covalent crystals. Compared to the simplified model \([10]\) it was suggested that the law of incompressibility should be applied for calculating the participation of plastic deformation, \( \varepsilon_p \), rather than for determining the total deformation, \( \varepsilon \). Thus, the share of plastic deformation, \( \varepsilon_p \), has been defined analogous to Eq. (2):

\[- \varepsilon_p = \ln \sin \gamma_2 \quad (5)\]

where \( \gamma_2 \) is the angle between the actual indentation face and its axis, \( \gamma_2 > \gamma_1 \).

The expression for determining the angle, \( \gamma_2 \), has been derived in the form:

\[
\cot \gamma_2 = \cot \gamma_1 - 1.77 \frac{H_M}{E} \quad (6)
\]
where $E^*$ is the effective modulus:

$$\frac{1}{{E^*}} = \frac{{1 - \nu_1^2}}{{E_1}} + \frac{{1 - \nu_2^2}}{{E_2}},$$

(7)

where indexes 1 and 2 correspond to parameters of the material and diamond indenter, respectively.

Finally, the elastic deformation, $\varepsilon_e$, is determined by Eq. (3) similar to the method suggested by the simplified theory [10] whereas the expression for calculating the participation plastic deformation, $\varepsilon_p$, has been derived in a modified form [13]:

$$-\varepsilon_p = \ln \left[ 1 + \left( \frac{\text{ctg} \gamma_1 - \frac{HM}{kE^*}}{kE^*} \right)^2 \right],$$

(8)

where $k$ is a coefficient, which both for the Vickers indenter and Berkovich pyramid is as high as 0.565.

The advantage of the modified model [13] is a good representation of the total deformation, $\varepsilon$, by means of summarising the shares of elastic, $\varepsilon_e$, and plastic deformation, $\varepsilon_p$, since they can be determined independently using Eqs. (3) and (8).

This paper aims to justify experimentally the applicability of modern approaches [10, 13] for determining the plasticity characteristic $\delta_H$ of coatings, which differ substantially from bulk materials by structural non-uniformity and also by the presence of an initial field of residual stresses. Specific features of the test method procedure, which are relevant to the subject matter, will be clarified also.

2. Experimental

A number of ceramic coatings made of carbides (TiC, ZrC, VC, NbC, Cr$_7$C$_3$), TiN-nitrides, silicides (NbSi$_2$, TaSi$_2$, WSi$_2$), iron borides (FeB, Fe$_2$B) and also galvanic Cr were employed in the present study. Ceramic coatings in the form of a single layer and those consisting of several layers were obtained by techniques of chemical vapour deposition (CVD), physical vapour deposition (PVD), and diffusion saturation method (DSM). Typical experimental conditions of coating application are listed in Table I.

The coating structure was examined by X-ray diffraction (XRD), optical microscopy, and scanning electron microscopy (SEM). Dimensions of the short and long axes of a grain, $D_{min}$ and $D_{max}$, arranged in parallel and perpendicular to the coating interface were used to define grain morphology (size and shape).

Tests with a standard Vickers pyramid were performed while indenting the cross-section of coatings. A rigidly fixed orientation of indentation arranged by the diagonals orthogonal to the coating interface was applied to avoid the effect of the residual stress acting in the coating on measurement results. The coatings were tested under indentation loads ranging from 0.2 N to 3.0 N. The final results were averaged over the data determined by measuring no less than 10 indentations under the each indentation load.

Elastic deformation, $\varepsilon_e$, and plasticity characteristic $\delta_H$, were determined using proper equations (1), (3), (4), (8) suggested by two theoretical models, i.e. either the original simplified model [10] or the modified one [13].

Values of Young’s modulus and Poisson’s ratio relevant to coating materials were adopted from a handbook [34]. In particular cases the values of Young’s modulus for coatings were determined by depth sensing tests using the nanoindentation technique.
Table I Conditions of coating application

<table>
<thead>
<tr>
<th>Specimen number</th>
<th>Coating composition*</th>
<th>Substrate</th>
<th>Method</th>
<th>Exposition conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>TiC</td>
<td>Carbon steel</td>
<td>CVD [26]</td>
<td>1050</td>
</tr>
<tr>
<td>2</td>
<td>ZrC</td>
<td>Carbon steel</td>
<td>CVD [25,27]</td>
<td>1050</td>
</tr>
<tr>
<td>3</td>
<td>VC</td>
<td>Carbon steel</td>
<td>DSM [28]</td>
<td>1050</td>
</tr>
<tr>
<td>4</td>
<td>NbC</td>
<td>Carbon steel</td>
<td>CVD [29]</td>
<td>1050</td>
</tr>
<tr>
<td>5-1</td>
<td>Cr$_7$C$_3$</td>
<td>Carbon steel</td>
<td>CVD [26]</td>
<td>1050</td>
</tr>
<tr>
<td>5-2</td>
<td>Cr$_7$C$_3$</td>
<td>Carbon steel</td>
<td>CVD ** [26]</td>
<td>900</td>
</tr>
<tr>
<td>6-1</td>
<td>Cr$_7$C$_3$</td>
<td>Carbon steel</td>
<td>CVD ** [26]</td>
<td>900</td>
</tr>
<tr>
<td>6-2</td>
<td>Cr$_7$C$_3$</td>
<td>Carbon steel</td>
<td>CVD ** [26]</td>
<td>900</td>
</tr>
<tr>
<td>7</td>
<td>δ-TiN</td>
<td>Ti-alloy</td>
<td>PVD [30]</td>
<td>400</td>
</tr>
<tr>
<td>8-1</td>
<td>FeB</td>
<td>Alloyed steel</td>
<td>DSM [31]</td>
<td>975</td>
</tr>
<tr>
<td>8-2</td>
<td>Fe$_2$B</td>
<td>Alloyed steel</td>
<td>DSM [31]</td>
<td>975</td>
</tr>
<tr>
<td>9-1</td>
<td>FeB</td>
<td>Carbon steel</td>
<td>DSM [32]</td>
<td>975</td>
</tr>
<tr>
<td>9-2</td>
<td>Fe$_2$B</td>
<td>Carbon steel</td>
<td>DSM [32]</td>
<td>975</td>
</tr>
<tr>
<td>10</td>
<td>NbSi$_2$</td>
<td>Nb-alloy</td>
<td>DSM [33]</td>
<td>1250</td>
</tr>
<tr>
<td>11</td>
<td>TaSi$_2$</td>
<td>Tantalum</td>
<td>DSM [33]</td>
<td>1250</td>
</tr>
<tr>
<td>12</td>
<td>WSi$_2$</td>
<td>Tungsten</td>
<td>DSM [33]</td>
<td>1250</td>
</tr>
<tr>
<td>13</td>
<td>Cr</td>
<td>Cast iron</td>
<td>Conventional galvanising process</td>
<td></td>
</tr>
</tbody>
</table>

Notes: *- composition is appointed in the direction from the specimen surface to the substrate; **the process was applied by twice to perform each type of carbide layer; CVD- Chemical Vapour Deposition technique; PVD- Physical Vapour Deposition technique; DSM- Diffusion Saturation Method

Nanoindentation experiments were performed using a Nano Indenter II tester. The continuously recorded load versus indenter displacement provided a coating material’s response to deformation. Then, load - displacement curves were used for calculation of Young’s modulus according to a proper method published in literature [6, 8, 15].

3. Results
3.1. Composition and structure of coatings

Coating layers with different grain morphology were pointed out and classified. Fig.1 shows a typical morphology of grains observed in coatings. Some coating layers consisted of globular grains (typical for layers of TiC, ZrC, VC) and others of somewhat elongated polyhedral crystallites (typical for layer of VC). Coatings of fibred grains (typical for layers of TiN, NbSi$_2$, TaSi$_2$, WSi$_2$) and those of columnar crystals (typical for layers of Cr$_7$C$_3$, FeB, Fe$_2$B) were pointed out additionally.

Furthermore, several groups of coatings were classified in respect to grain size. Among them there was the group of fine-grained coatings with the dimensions for both axes of grains of about $\leq 5 \mu m$ (typical for carbide layers). Another group unites coarse-grained coatings consisting of crystallites for which the dimensions for both axes are found in the order of magnitude as great as $10^1$-$10^2 \mu m$ (typical for layers of iron borides). The intermediate group refers to coatings (typical for layers of TiN-nitride and silicides) consisting of crystallites with substantially different dimensions of axes.
Fig. 1 (a, b) SEM images and (c-d) optical micrographs of coatings indicated in Table 1 by samples (a) 1, (b) 7, (c) 5, (d) 3, (e) 11, (f) 9 and made of (a) TiC, (b) TiN, (c) Cr$_2$C$_6$/Cr$_7$C$_3$, (d) Cr$_{23}$C$_6$/Cr$_7$C$_3$/VC, (e) TaSi$_2$, (f) FeB/Fe$_2$B. Grains of different morphology are shown in a coating cross-section: (a) globular grains; (b,e) fibred grains; (d) polyhedral crystallites; (c, f) columnar crystals.

One axis of these coatings was found to be big (to about $10^1$-$10^2$ $\mu$m) although another was small (about $\leq 5$ $\mu$m). The specific group unites coatings consisting of submicro-
and nano-scaled grains (typical for layers of TiC, ZrC, TiN, VC) for which the dimension of one grain axis at least was found to be smaller than 1 \( \mu m \).

Table II shows the composition and structural parameters of representative coatings listed in Table I.

### Table II Structural parameters and composition of coatings.

<table>
<thead>
<tr>
<th>Specimen number</th>
<th>Coating composition*</th>
<th>Thickness, ( \mu m )</th>
<th>Grain morphology</th>
<th>Shape</th>
<th>Dimensions, ( \mu m )</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>short axis</td>
<td>long axis</td>
</tr>
<tr>
<td>1</td>
<td>TiC</td>
<td>20</td>
<td>Globular</td>
<td>0.5</td>
<td>0.8</td>
</tr>
<tr>
<td>2</td>
<td>ZrC</td>
<td>20</td>
<td>Globular</td>
<td>&lt; 0.5</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>VC</td>
<td>20</td>
<td>Polyhedral</td>
<td>&lt; 0.8</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>NbC</td>
<td>20</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>5-1</td>
<td>Cr(_{23})C(_6)</td>
<td>8</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>5-2</td>
<td>Cr(_7)C(_3)</td>
<td>27</td>
<td>Columnar</td>
<td>1.8</td>
<td>7.5</td>
</tr>
<tr>
<td>6-1</td>
<td>Cr(_7)C(_3)</td>
<td>15</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>6-2</td>
<td>TiC</td>
<td>15</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>7</td>
<td>( \delta )-TiN</td>
<td>16</td>
<td>Fibred</td>
<td>&lt; 0.5</td>
<td>16</td>
</tr>
<tr>
<td>8-1</td>
<td>FeB</td>
<td>40</td>
<td>Columnar</td>
<td>20</td>
<td>40</td>
</tr>
<tr>
<td>8-2</td>
<td>Fe(_2)B</td>
<td>100</td>
<td>Columnar</td>
<td>33</td>
<td>100</td>
</tr>
<tr>
<td>9-1</td>
<td>FeB(^+)</td>
<td>40</td>
<td>Columnar</td>
<td>15</td>
<td>40</td>
</tr>
<tr>
<td>9-2</td>
<td>Fe(_2)B</td>
<td>330</td>
<td>Columnar</td>
<td>25</td>
<td>330</td>
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<tr>
<td>10</td>
<td>NbSi(_2)</td>
<td>100</td>
<td>Columnar</td>
<td>1.5</td>
<td>100</td>
</tr>
<tr>
<td>11</td>
<td>TaSi(_2)</td>
<td>80</td>
<td>Columnar</td>
<td>2.5</td>
<td>80</td>
</tr>
<tr>
<td>12</td>
<td>WSi(_2)</td>
<td>80</td>
<td>Columnar</td>
<td>4.8</td>
<td>46</td>
</tr>
<tr>
<td>13</td>
<td>Cr</td>
<td>370</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

Notes: * - composition is appointed in the direction from the specimen surface to the substrate; "**" - the phase is presented in the form of single crystals.

### 3.2 Effect of the scale factor on microhardness and the plasticity characteristic \( \delta_H \)

Microhardness measurement results were found to depend on the scale factor when indentation experiments were performed using small loads. While testing high hardness coatings a size dependence of an increasingly pronounced nature was found similar to that shown previously for covalent crystals and ceramics [7, 9, 17]. Apparently, the size dependence of the plasticity characteristic \( \delta_H \) on indentation load was found since the hardness value was included in the right side of Eqs. (3), (4), and (8).

Typical plots of coating Vickers hardness, \( HV \), and plasticity characteristic \( \delta_H \) vs. indentation load are shown in Fig. 2. It can be seen that the hardness number decreases with increasing indentation load, resulting in an increase of the plasticity characteristic \( \delta_H \). As the indentation load further increases to values higher than critical load \( P_c \), the values of parameters \( HV \) and \( \delta_H \) tend to be almost stabilised. It is possible that a weak decrease of hardness numbers occurs in fact when the indentation load increases up to high values [7, 9]. However, these changes are insignificant.

Indentation load marked by \( P_c \) truncates the region in which the size dependence of parameters \( HV \) and \( \delta_H \) is essential. It is notable that the intensity of the increase of hardness
numbers that occurs with decreasing the indentation load in the region \( P \leq P_c \) is different for each type of coating.

Some ideas on the physical nature of this phenomenon will be given later. However, one important aspect related to the specific feature of the test method procedure for high hardness ceramic coatings could be mentioned here.

Fig. 2 A summary of the data for mechanical parameters of coatings vs. indentation load \( P \). Closed and open symbols denote Vickers hardness, \( H_V \), and plasticity characteristic \( \delta_{Ht} \), respectively. Coating compositions are marked by symbols and shown on the plots.

This aspect relates to a comparison of mechanical properties for different coatings. To avoid responsibility as to inaccuracies, which can appear due to the effect of the scale factor, it is reasonable to compare different coatings using values of the plasticity characteristic \( \delta_{Ht} \).
determined under loads higher than critical, $P \geq P_c$, ensuring the most stable values of hardness.

Therefore, the critical load $P_c$ was determined carefully in further indentation experiments with coatings.

3.3. Comparison of the results determined by simplified and modified theoretical models

A comparison between values of the plasticity characteristic, $\delta_H$, determined by both theoretical approaches [10, 13] is important for understanding the limits of their applicability depending on the mechanical behaviour of coatings.

Data summarising values of elastic deformation, $\varepsilon$, and the plasticity characteristic $\delta_H$, which were calculated according to both theoretical models [10, 13] using Eqs. (4) or (1), (3), (8), are given for some representative coatings in Table III. The experimental values of Vickers hardness, $HV$, determined under load condition $P \geq P_c$ as well as Young’s modulus and Poisson’s ratio, which were used in the calculation procedure, are also listed in Table III.

Table III  Elastic deformations, $\varepsilon$, and plasticity characteristic $\delta_H$ determined for coatings in experiments with a Vickers indenter.

<table>
<thead>
<tr>
<th>Specimen number</th>
<th>Layer</th>
<th>HV, GPa</th>
<th>E, GPa</th>
<th>$\nu$</th>
<th>$\varepsilon_{\text{e},%}$</th>
<th>$\delta_H^*$</th>
<th>$\delta_H^{**}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>TiC</td>
<td>35.9</td>
<td>465</td>
<td>0.17</td>
<td>6.43</td>
<td>0.15</td>
<td>0.24</td>
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<tr>
<td>1’</td>
<td>TiC</td>
<td>35.9</td>
<td>445***</td>
<td>0.17</td>
<td>6.72</td>
<td>0.11</td>
<td>0.22</td>
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<td>2</td>
<td>ZrC</td>
<td>29.7</td>
<td>355</td>
<td>0.191</td>
<td>6.64</td>
<td>0.12</td>
<td>0.23</td>
</tr>
<tr>
<td>3</td>
<td>VC</td>
<td>21.4</td>
<td>430</td>
<td>0.32</td>
<td>2.55</td>
<td>0.66</td>
<td>0.61</td>
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<tr>
<td>3’</td>
<td>VC</td>
<td>21.4</td>
<td>413***</td>
<td>0.32</td>
<td>2.66</td>
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<td>0.59</td>
</tr>
<tr>
<td>4</td>
<td>NbC</td>
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<td>550</td>
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<td>2.81</td>
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<td>Cr$_2$C$_3$</td>
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<td>2.60</td>
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<td>Cr$_2$C$_3$</td>
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<td>303***</td>
<td>0.26</td>
<td>3.37</td>
<td>0.56</td>
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<td>6-1</td>
<td>Cr$_2$C$_3$</td>
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<td>320***</td>
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<td>0.48</td>
<td>0.47</td>
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<td>TiC</td>
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<td>0.17</td>
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<td>0.45</td>
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<td>TiN</td>
<td>18.7</td>
<td>440</td>
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<td>0.62</td>
<td>0.60</td>
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<td>16.8</td>
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<td>0.25</td>
<td>3.24</td>
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<td>0.56</td>
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<td>0.48</td>
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<td>0.57</td>
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<td>3.95</td>
<td>0.48</td>
<td>0.49</td>
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<td>400</td>
<td>0.24</td>
<td>2.76</td>
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<td>0.62</td>
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<td>530</td>
<td>0.24</td>
<td>2.19</td>
<td>0.71</td>
<td>0.69</td>
</tr>
<tr>
<td>13</td>
<td>Cr</td>
<td>9.6</td>
<td>279</td>
<td>0.31</td>
<td>1.85</td>
<td>0.76</td>
<td>0.74</td>
</tr>
</tbody>
</table>

Notes: $^*$ - this parameter was calculated according to the simplified model [10] by Eq. (4);
$^{**}$ - this parameter was calculated according to the modified model [13] by Eqs. (1), (3), (8);
$^{***}$ - Young’s modulus was determined experimentally by the nanoindentation technique;
$^{****}$ - coating was alloyed strongly by chromium.

Fig. 3 shows values of the plasticity characteristic $\delta_H$ determined for a number of different coatings. It can be seen that both models are quite similar when the elastic
deformation of coatings was small $\varepsilon_e < 3.5\%$ and, so, their plasticity characteristic was great $\delta_H > 0.5$. When the elastic deformation further increased, resulting in $\delta_H < 0.5$, the opposite is true, i.e. the modified approach [13] gives $\delta_H$ values greater than those determined by the simplified model [10].

![Graph showing the relationship between elastic deformation and plasticity characteristic.](image)

**Fig. 3** A summary of the data for the plasticity characteristic $\delta_H$ of coatings vs. elastic deformation, $\varepsilon_e$. Numbers 1 and 2 denote the values of plasticity characteristic $\delta_H$ determined according to the simplified [10] theoretical model and the modified [13] one, respectively. Symbols mark coating compositions that are shown on the plot.

It is notable that the transition point plotted in Fig. 3 at $\varepsilon_e = 3.5\%$ corresponds to the relation $HV/E \approx 0.05$, which seems to be of engineering interest since it gives the guideline for calculating the $\delta_H$ characteristic using an adequate model. It can be assumed that for $HV/E < 0.05$ the simplified model [10] is reasonably adequate for determining values of the plasticity characteristic $\delta_H$ for coatings similar to those given by the modified approach [13]. However, for $HV/E > 0.05$ use of the modified model [13] is preferable compared to the simplified one [10].

4. Discussion

4.1. Physical nature of the effect of the scale factor on measurement results

It was demonstrated previously that the Meier equation could be used for describing the effect of the scale factor on microhardness measurement results [6, 9]:

$$P = p \left( \frac{d}{d_0} \right)^n$$

(9)
where \( n \) is a constant; \( d_o \) is the unitary indentation diagonal that was introduced for retaining the dimensional balance between left and right members; \( p \) is the particular load under which the indentation diagonal \( d = d_o \).

For a Vickers indenter the Meier hardness is determined by equation:

\[
HM = \frac{2P}{d^2}
\]  

(10)

So, taking into account Eqs. (9) and (10) we obtain:

\[
HM = \alpha P^{\frac{1}{n}}
\]

(11)

where \( \alpha = \frac{2P^2}{d_o^2} \).

It could be seen from Eq. (11) that the similarity law is satisfied if \( n = 2 \). But, default of the similarity law takes place when the power index \( n \) becomes smaller than 2 and is dependent on the material’s nature [6, 9].

Analogously with the method published originally in [6, 9] the power index \( n \) was determined experimentally in tests with coatings examined in the present study. In agreement with the data obtained originally for single crystals and bulk polycrystalline materials [7, 9] the results determined for coatings indicate that the power index \( n \) in Meier’s equation (9) becomes actually less than 2 and it decreases proportionally as the value of the \( HV/E \) ratio increases, as shown in Fig. 4.

**Fig. 4** Correlation of the power index \( n \) in Meier’s equation (9) vs. ratio of Vickers hardness to Young’s modulus, HV/E, (A) for coatings of different compositions as well as (B) for single crystals and bulk polycrystalline material such as (1) Cu, (2) W, (3) ZrN, (4) NbC, (5) ZrC, (6) Al2O3, (7) Ge, (8) SiC, (9) Si, (10) C (diamond). Coating compositions are marked by symbols and shown on the plot. The data for single crystals and bulk polycrystalline material are adopted using data published in [7, 9].

That is why the intensity of the increase of the hardness number determined in the region \( P \leq P_c \) increases as the power index \( n \) decreases, as seen in Fig.2. It is notable that for the same \( HV/E \) ratio the value of the power index \( n \) in Meier’s equation (9) found for coatings is greater than that indicated for single crystals and bulk materials [9]. The reason for this
could be attributed to strain hardening caused by the small grain size of coatings. Therefore, the dependence HM(P), which was determined for coatings employed in the present study, is more weak compared to that recorded for single crystals and bulk materials.

Unfortunately, no good correlation is observed if the data related to critical load $P_c$ is plotted as a function of the HV/E ratio. Two different lines form, which indicate only that for fine-grained coatings (typically for TiN-coating and also for carbide coatings) the critical load $P_c$ has quite low values ranging from 0.5 to 0.7 N whereas for course-grained coatings somewhat higher values varying between 1.2 and 1.4 N are obtained.

**Fig. 5** Correlation of Vickers hardness for coatings consisting of (a) fine grains and (b) coarse grains vs. the ratio of indentation depth to the dimension of grain short axis, $h/D_{\text{min}}$. Coating compositions are marked by symbols and shown on the plot. The dotted line truncates the region where the effect of the scale factor is essential.

It was pointed out previously that the size dependence of microhardness measurement results on indentation load occurs because plastic deformation $\varepsilon_p$ decreases essentially with the decreasing indentation load in the region of very small values [9]. From the physical standpoint it was suggested that contribution of the $\varepsilon_p$ component was ensured by the indentation size resulting in a certain volume of the deformed material, allowing easy plasticity deformation [9].
The experimental results indicate that the critical load condition could be attributed to coating grain structure rather than to its composition. Correlation appears by plotting hardness numbers vs. the ratio of the indentation depth to dimension of grain short axis, \( h/D_{\text{min}} \), as shown in Fig. 5. It could be seen that critical load \( P_c \) is achieved only if the indentation depth increases to \( h > 1.5D_{\text{min}} \). Under conditions \( h > 1.5D_{\text{min}} \), which occurred under loads \( P \geq P_c \), the length of dislocation slip planes was assumed to be controlled by grain size and, if so, hardness and, as a result, the plasticity characteristic \( \delta_H \) become almost independent on the indentation load, as shown in Fig. 5a. In a particular case referring to coarse-grained coatings for which \( h < D_{\text{min}} \) (typical for coatings made of \( \text{Fe}_2\text{B}, \text{WSi}_2 \)) dislocation movement cannot be blocked essentially by grain boundaries similar to that observed for single crystals. Under this condition an average extension of dislocation slipping increases continuously with increasing of the indentation load. Therefore, as shown in Fig. 5b for coarse-grained coatings (typically for \( \text{Fe}_2\text{B}, \text{WSi}_2 \)) the hardness number was found to decrease weekly as the indentation load increases even in the region of over critical loads \( P \geq P_c \) similar to that found previously for single crystals and bulk materials [7].

Thus, the results demonstrate that specific features of size dependence are defined both by the crystal nature of the coating material and by its grain structure.

4.2. Plasticity characteristic \( \delta_H \) of coatings

Carbide coatings based on metals of group IV-A (Ti and Zr) have the smallest values of plasticity characteristic \( \delta_H \), as shown in Table III and Fig. 4. In contrast with that carbide coatings based on metals belonging to groups’ V-A and VI-A (V, Nb Cr,) have considerably larger values of the plasticity characteristic, \( \delta_H \approx 0.6 \). Close values of parameter \( \delta_H \) are found for TiN-coating and layers made of iron borides (FeB, Fe2B). Silicide coatings demonstrate different mechanical behaviour depending on their phase composition. The values of plasticity characteristic \( \delta_H \), which are similar to those found for carbide coatings based on metals of groups’ V-A and VI-A, were found only for TaSi2-coating. Compared to TaSi2-coating the value of plasticity characteristic \( \delta_H \) determined for NbSi2-coating is somewhat smaller although the opposite is true for WSi2-coating. Among the coatings investigated in the present study metallic coating made of galvanic chromium has the greatest value of plasticity characteristic, \( \delta_H \approx 0.74 \). Generally, with the growth of \( \delta_H \) characteristic coating materials could be ordered in the following manner: carbides based on metals of group IV-A, iron borides, carbides based on metals of groups’ V-A and VI-A, titanium nitride, silicides of refractory metals, galvanic chromium.

5. Conclusions

Effective application of modern theoretical approaches for determining the plasticity characteristic \( \delta_H \) of coatings by the indentation technique was tested experimentally.

(1) Allowing for small thickness of coatings, which are usually affected by residual stress, specific test method procedures were developed. In order to avoid inaccuracies, which can occur due to the influence of the scale factor, a comparison of different coatings was proposed, using values of the plasticity characteristic \( \delta_H \) determined under loads higher than critical, \( P \geq P_c \), ensuring almost stable values of hardness.

(2) Critical load \( P_c \), which provides stable values of hardness for fine-grained coatings, is achieved when dimensions of the indentation depth, \( h \), was ensured by the relation \( h/D_{\text{min}} > 1.5 \) (where \( D_{\text{min}} \) is the short axis of the grain). In a particular case of coarse-grained coatings for which \( h < D_{\text{min}} \) the hardness number was found to decrease weekly as the
indentation load increases even in the region of over critical loads $P \geq P_c$, similar to that found previously for single crystals and bulk materials.

(3) Meier’s relation $P = const \times d^n$ for which $n < 2$ was shown to be satisfied for coatings similar to those found for single crystals and bulk materials. However, the value of power index $n$ for coatings is greater compared to that indicated for single crystals and bulk materials. That is why deviation from the similarity law for which $n=2$ is more week for coatings than that found for single crystals and bulk materials.

(4) The plasticity characteristic $\delta_H$ determined by the simplified model and by the modified one was found to be of a quite similar value when elastic deformation does not exceed the value of $\varepsilon_e \approx 3.5\%$, resulting in the plasticity characteristic $\delta_H > 0.5$. If only the elastic deformation becomes greater than $\varepsilon_e \approx 3.5\%$, corresponding to $HV/E$ ratio $> 0.05$, the modified model was found to be preferable.

(5) It was found that with the growth of $\delta_H$ coatings could be ordered in the following manner: carbides based on metals of group IV-A, iron borides, carbides based on metals of groups V-A and VI-A, titanium nitride TiN, silicides, galvanic chromium.

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References


Резюме: Рассмотрены особенности определения характеристики пластичности δH при индентировании неоднородных покрытий, находящихся под воздействием остаточных напряжений. Установлено, что результаты расчетов, проводимых как в приближении упрощенной модели, так и согласно ее модифицированному варианту, учитывающего сжимаемость ядра деформации, показывают хорошую сходимость в тех случаях, когда характеристика пластичности покрытий составляет δH > 0,5. Экспериментально обосновано, что преимущества модифицированной модели по сравнению с упрощенным приближением проявляются при испытании покрытий, для которых упругая деформация оказывается больше, чем εe ≈ 3,5%, что приводит к уменьшению характеристики пластичности до значений δH < 0,5. Для предотвращения неточностей, связанных с влиянием масштабного фактора на результаты измерений, предложено сравнивать различные покрытия, используя стабильные значения характеристики пластичности δH, полученные при нагрузках выше критической, при которых значения микротвердости слабо зависят от нагрузки.

Ключевые слова: Покрытие; механические свойства; характеристика пластичности.
Садржај: Размотрена су специфична својства одређивања карактеристике пластичности $\delta_H$ при индентирању нехомогености превлака које се налазе под утицајем заосталих напрезања. Утврђено је да резултати прорачуна који се врше како априксимацијом упрощеног модела, тако и у складу са његовом модификованим варијантом, која узима у обзир спољашњост језгра деформације, показују добру конвергенцију у оним случајевима када је карактеристика пластичности превлака $\delta_H > 0,5$. Експериментално је потврђено да се предности модификованих модела, у поређењу са упрошћеном априксимацијом, испољавају при испитивању превлака код којих је еластична деформација већа од εc ≈ 3,5%, што доводи до снижења карактеристика пластичности до вредности $\delta_H > 0,5$. Ради спречавања непрецизности повезаних са утицајем величине фактора на резултате мерења, предложено је поређење различитих превлака применом стабилних вредности карактеристике пластичности $\delta_H$ добијених при оптерећењима изнад критичног, при којима вредности микротврдоће слабо зависе од оптерећења.

Кључне речи: Превлака, механичка својства, карактеристика пластичности