Analysis of the indentation size effect on the hardness measurements of materials

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Abstract. The influence of the indentation size effect on the hardness measurements of materials using indentation method was studied. A method for determining and comparing the hardness of two different materials with studying the indentation size effect was developed. To identify the influence of the indentation size effect on the Vickers hardness of materials with different levels of hardness two-dimensional parameters $\alpha$ and $\gamma$ were proposed.

1. Introduction

Recently, interest in the indentation size effect (ISE) and its influence on the mechanical properties of materials have received much scientific and practical attention [1–4]. This is due to the rapid development of nanomaterials and nanotechnology, as well as a new multi-level approach to study the deformation and strength of solids. Studies showed that the indentation method is widely used in order to determine the mechanical properties of materials at different indentation size levels. As levels of the size factor, macro-, meso-, micro-, nano levels are usually used [5–6].

Studies the influence of the ISE on the hardness values of materials showed that with decreasing indentation load $F$ and consistently geometrical parameters of indents, hardness values increase, especially in the low loads [7]. Geometric parameters of the indent can be used as criteria for classification of indentation scale levels. If the indent depth is chosen for such a criterion, then the dimensional levels can be classified into: nano-level at a depth of less than 0.1 µm, micro-level from 0.1 to 1 µm, meso level from 1 to 10 µm and macro-level more than 10 µm.

In ISO 6507-1: 2005, three ranges of hardness scales are given when the pyramid is used as indenter. Table 1 shows the Vickers hardness test method, for the three different ranges of test force for metallic materials.

| Indentation load $F$, N | Hardness       | Designation                 |
|-------------------------|----------------|-----------------------------|
| $F \geq 49.03$          | $\geq HV 5$   | Vickers hardness test       |
| $1.961 \leq F < 49.03$  | HV 0.2 to $< HV 5$ | Low-force Vickers hardness test |
| $0.098 \leq F < 1.961$  | HV 0.01 to $< HV 0.2$ | Vickers microhardness test |
In another standard ISO 14577-1: 2002, in order to distinguish between macro-, micro-, nano-ranges of indentation, either the load \( F \) or the indent depth \( t \) were selected as the differentiation criterion (Table 2).

**Table 2.** Application ranges for instrumental indentation (ISO 14577-1: 2002).

| Macro range | Micro range | Nano range |
|-------------|-------------|------------|
| 2 < \( F \) ≤ 30 \( \times \) 10\(^4\) H | \( F \leq 2 \) H; \( t > 0.2 \) мкм | \( t \leq 0.2 \) мкм |

For the nano-range, the depth \( t \) is 0.2 μm (200 nm) or less, for the macro range, such a depth is not indicated, but only the limits of the indentation load \( F \) are given. Therefore, when the pyramid is used, it would be logical to introduce the indentation mesoscale as a transition from the micro to the macro range, indicating the maximum indents depths (Table 3).

**Table 3.** The proposed classification of the dimensional levels of indentation.

| Indentation depth \( t \), μm | Indentation diagonals \( d \), μm | Indentation volumes \( V \), \( \mu m^3 \) | Dimensional indentation level |
|-----------------------------|-----------------------------|-----------------------------|-----------------------------|
| 0.001 < \( t \) ≤ 0.2 | 0.007 < \( d \) ≤ 1.4 | 2.80 \( \times \) 10\(^6\) < \( V \) ≤ 6.53 \( \times \) 10\(^2\) | Nanoscale |
| 0.2 < \( t \) ≤ 2.0 | 1.4 < \( d \) ≤ 14 | 6.53 \( \times \) 10\(^2\) < \( V \) ≤ 653.10 | Micro scale |
| 2.0 < \( t \) ≤ 10 | 14 < \( d \) ≤ 70 | 6.53 < \( V \) ≤ 8.17 \( \times \) 10\(^3\) | Meso scale |
| \( t \) > 10 | \( d \) > 70 | \( V \) > 8.17 \( \times \) 10\(^3\) | Macro scale |

Table 3 additionally shows the indents diagonal \( d \) and volume \( V \), which are also dimensional parameters.

In the work [8], it was found that the deformable volume of the material during indentation process depends on the indentation volume \( V \). When using the Vickers pyramid, the indentation volume \( V \) depends on the indentation depth \( t \) or diagonal \( d \):

\[
V = 8.167t^3 = 0.0238d^3.
\]  

(1)

It should be noted that for comparing the hardness \( H V \) of two or more different materials, it is necessary to carry out indentation not with the same indentation load \( F \), but with the same indentation depths \( t \) or diagonals \( d \).

Therefore, to ensure the same indentation volume, and, consequently, the same deformable volumes of compared materials, it is enough to withstand the same depth \( t \) or diagonal \( d \).

The easiest way is to achieve this condition by using the kinetic indentation of materials with registration of the indentation diagrams in the coordinates “load - displacement of the indenter”.

Having the indentation diagrams of the compared materials, one can calculate their hardness at the same indentation depth \( t \).

Using a micro-hardness tester, which allows to measure the indentation diagonal \( d \), so it is not necessary to repeatedly perform the indentation of the pyramid to reach the specified value of the indentation diagonal \( d \). To reach this value, it is enough to first reveal a softer material, perform an indentation on two materials under the same load \( F_1 \), and then for a harder material, perform another indentation under the load \( F_2 = 3F_1 \). The relationship between the indentation load \( F \) and the indentation diagonal \( d \) is accurately approximated by Meyer's power equation:

\[
F = \alpha d^n,
\]  

(2)

where \( \alpha \) and \( n \) are constant coefficients for each material.

According to the results of two indentations for a more solid material, the coefficients \( \alpha \) and \( n \) are calculated using the following formulas:
\[ n = \frac{\ln(F_2 / F_1)}{\ln(d_2 / d_1)}, \]  
(3)

\[ \alpha = \frac{F_2}{d_2^2}. \]  
(4)

where \( d_1 \) and \( d_2 \) are the indentation diagonal corresponding to the loads \( F_1 \) and \( F_2 \). Knowing \( \alpha \) and \( n \), it is possible to calculate the required indentation load \( F' \) for a harder material, at which the indentation diagonal will be equal to the diagonal of softer material \( d_s \), corresponding to the load \( F_1 \):

\[ F' = \alpha d_s^2. \]  
(5)

Then the hardness of the harder material \( H_V \) is calculated depending on the indentation load \( F' \) and \( d_s \) according to the formulas:

\[ H_V = 1.854 \frac{F'}{d_s^2}, \]  
(6)

where \( F \) measured in kgf, \( d_s \) in mm.

2. Materials and experimental process

For practical testing, two steel samples with a large hardness difference \( H_V \) were selected: sample I 112HV10 and sample II 809HV10. Tests were performed by the existing method with the same indentation loads \( F \) at three different dimensional indentation levels, and also by the proposed method, then the values of \( n, a, F', H_V(F') \) for solid samples were determined. Tests were performed on an automated Instron Tukon 2500 instrument.

Table 4 presents the results of determining the hardness \( H_V \) by the existing method and the hardness \( H_V(F') \) according to the proposed method at three different dimensional indentation levels.

| \( F_1 \), kgf | Macro | Mezo | Micro |
|---|---|---|---|
| Sample I | Sample II | Sample I | Sample II | Sample I | Sample II |
| HV, kgf/mm² | 112 | 810 | 120 | 850 | 161 | 988 |
| \( d_1 \), µm | 407 | 152 | 124 | 47 | 7.59 | 3.06 |
| \( F_2 = 3F_1 \), kgf | - | 30 | - | 3 | - | 0.015 |
| HV², kgf/mm² | - | 805 | - | 823 | - | 936 |
| \( d_2 \), µm | - | 263 | - | 82 | - | 5.45 |
| \( n \) | - | 2.003 | - | 1.975 | - | 1.9033 |
| \( a \) | - | 435.5 | - | 419.12 | - | 305.1 |
| \( F' \), kgf | - | 71.95 | - | 6.789 | - | 0.0282 |
| \( H_V(F') \), kgf/mm² | - | 806 | - | 818 | - | 907 |
| \( \Delta \% \) | 0.5 | 3.8 | 8.9 |

From table 4 it can be seen that when determining the hardness of steel by the existing method at a given constant indentation load \( F \), the values of \( H_V \) are greater than the values of \( H_V(F') \) determined by the proposed method. The difference in hardness value varies in the range from 0.5 to 8.9%.
depending on the size level of indentation. At the macro– and meso indentation levels, the influence of the ISE is not so drastically reduced (from 0.5 to 3.8%). A large difference in hardness values determined by the existing and proposed methods is observed during the transition to the micro level, which is explained by the strong influence of the ISE.

In addition, in this work, experiments were performed to identify the influence of the ISE on the Vickers hardness of different materials. Steel samples with different levels of hardness were selected, a sample from mild steel Armco iron and samples of different non-ferrous metals.

For each sample, the values of Vickers hardness were determined at indentation loads from 0.01 to 10 kgf (micro, meso and macro) on an automated InstronTukon 2500 instrument. To identify the influence of the indentation size effect, 2 coefficients $\gamma$ and $\alpha$ were proposed:

$$\gamma = \frac{HV}{HV_{\text{max}}}$$  \hspace{1cm} (7)

$$\alpha = \frac{F_{\text{max}}}{F}$$  \hspace{1cm} (8)

The values of the coefficients $\gamma$, $\alpha$ for all loads for all samples were determined, and graphs of the dependence of $\gamma$ on $\alpha$ for each material were plotted (figure 1 and figure 2).

Figure 1. Plot of $\gamma$ vs. $\alpha$ for steel samples.

Figure 2. Plot of $\gamma$ vs. $\alpha$ for non-ferrous metals.
The experimental results show the influence of the ISE on the hardness values of materials during indentation at different scale levels. From figure 1 and 2 it can be seen that the ISE is more noticeable especially in the micro range. At the same time, the graphs $\gamma$ vs. $\alpha$ are different for all materials, for example, for samples with hardness from 100 to 200 HB, the coefficient $\gamma$ clearly increases with decreasing load, i.e. transition from macro to micro indentation. For samples with a hardness level of 450 to 800 HV, the coefficient $\gamma$ slightly changes with decreasing load, that is, for soft materials, the value of $\gamma$ increases greatly with decreasing the indentation load and, therefore, the influence of the ISE will be more noticeable. For non-ferrous metals, there is a small change in the coefficient $\gamma$ during the transition from one dimensional level to another. The analysis of the obtained results indicates that the dependence of $\gamma$ on $\alpha$ is close to linear. The graphs $\gamma$-$\alpha$ make it possible to determine the hardness value at any indentation load, for example, at a certain load value $F$ and, based on $F_{\text{max}}$, we can determine the value $\alpha$ and therefore $\gamma$. Knowing the value of hardness at maximum load $F_{\text{max}}$, it is possible to obtain the value of hardness at any other load $F$.

3. Conclusion

The following conclusion can be made by the results of experiments. Studies and experiments showed that, determination of hardness should be performed with equal indent diagonals $d$ or depths $t$. Determination and comparison of the hardness of samples showed that the excess of hardness for a more solid sample, determined by the existing method with the same indentation load, can reach 9% compared to the hardness determined by the proposed method. When the indentation transitions from the micro to the nano range, this excess can significantly increase due to the stronger influence of the ISE. In the indentation process, the effect of the ISE is more pronounced in the soft material comparing with the hard material.

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