Determinaton of the mechanical properties of carbide composites by spherical indentation

Sergejev, F.; Petrov, M. & Kübarsepp, J.

Abstract: Carbide composites are materials based on the carbide phases of the refractory or high melting temperature metals. The hard carbide phase (WC, TiC) is usually embedded into soft matrix – pure metals (Fe, Co, Mo, Ni and others) or their combinations. The hard phase supports high hardness and strength of the carbide composites, while matrix is needed for toughness and plastic properties. The ordinary tensile testing is not applicable for yield stress determination in case of carbide composites. The ball indentation depth sensing technique was used to determine yield stress of hard materials. In present work the first results of the determination of the yield stress of the WC-Co and TiC-Fe/Ni carbide composites using spherical indentation are presented.

Key words: carbide composites; yield stress; spherical indentation.

1. INTRODUCTION

The use of indentation techniques for mechanical properties (hardness, fracture toughness) evaluation of brittle materials (metal matrix composites, ceramics, hardmetal, cermets etc.) is very popular because of ease of tests conduction, no need for precise and expensive specimen preparation, standard tools (indenters) and equipment are used of specified geometry, a lot of measurements can be conducted on relatively small testpieces and so on. Those techniques are improved and supplemented by new analytical procedures to gain better understanding of abilities, restrictions and accuracy of indentation methods [1,2]. The method for measuring hardness and elastic modulus by instrumented indentation techniques has been adopted and widely used for the characterization of mechanical behavior of materials at small scales. Its attractiveness stems largely from the fact that mechanical properties can be determined directly from indentation load and displacement dependences (curves) without the need to image the hardness impression [3]. The indentation size effect is investigated for application at nano-scale [4] also. Instrumented indentation tests results have shown steep dependence on the load. The materials indentation hardness and indenter penetration depth are in linear dependences from indentation load [1-4]. The stress measured by instrumented indentation technique is the actual response of the material to the indentation, hard and stiff indenter penetration into the materials surface. The stress field generated by the indentation process is heterogeneous and leads to plastic deformation and damage in the vicinity of the tip. Using Hertz’s theory, the spatial dependence of the stress components during indentation can be estimated, by considering the elastic contact of a spherical indenter with a semi-infinite half space. Numerous studies are conducted to investigate the elastic-plastic indentation stress field employing the spherical indenters (Hertz's theory) as most reliable one, supported by finite-element (FE) methods [5,6]. Only few of them are describing indentation behavior of hard materials [7].
2. EXPERIMENTAL PART

2.1 Materials tested
One TiC-based cermet with nickel-molybden binder (TiC-Ni/Mo), and conventional hardmetal (WC-Co) are tested. The main mechanical properties along with composition and microstructural parameters can be found from Table 1. All materials were produced in Powder Metallurgy Laboratory at Tallinn University of Technology (TUT). The testpieces were produced through conventional press and vacuum sinter powder metallurgy according to ASTM B406. Then specimens were prepared to following dimensions (width × height × length) - ((15.0±0.3) × (5.0±0.3) × 35 mm³). Finally specimens were ground and polished on cloth with 1 μm diamond paste to a surface roughness of about $R_a=0.2$ μm on two sides (measured along 8 mm of the specimen by the Surtronic 3+ apparatus, using CR filter), see Figs. 1 and 2. Opposite ground faces were parallel within 0.03 mm. In order to remove surface contaminants, the samples were cleaned in alcohol and dried by compressed air.

![Fig. 1. SEM micrograph of tested H15 carbide composite.](image)

![Fig. 2. SEM micrographs of tested T30A carbide composite.](image)

2.2 Spherical indentation procedure
The indentation tests were performed using Zwick ZHU/Z2.5 apparatus. The difference in the materials response related to indenter shape is obvious if compared with indentation curves made with Berkovich indenter at nanoindentation [1] and micro-macro indentations with Vickers pyramid, see Fig. 3.

![Fig. 3 Spherical indentation curves for studied H15 and T30A carbide composites.](image)

The yield stress determination is related with elastic stresses and materials response to elastic strains and is best characterized

<table>
<thead>
<tr>
<th>Grade</th>
<th>Carbidewt%</th>
<th>Binder wt%</th>
<th>Average carbide grain size $d_x, \mu m$</th>
<th>Transverse rupture strength $R_{TZ}, MPa$</th>
<th>Vickers hardness HV, MPa</th>
<th>Elastic modulus $E, GPa$</th>
<th>Fracture toughness $K_{IC}, MPa.m1/2$</th>
<th>Poisson ratio ν</th>
</tr>
</thead>
<tbody>
<tr>
<td>H15</td>
<td>WC, 85</td>
<td>Co, 15</td>
<td>1.98</td>
<td>2900</td>
<td>1170</td>
<td>560</td>
<td>15.2</td>
<td>0.23</td>
</tr>
<tr>
<td>T30A</td>
<td>TiC, 70</td>
<td>Ni/Mo, 30</td>
<td>2.00</td>
<td>1600</td>
<td>1280</td>
<td>395</td>
<td>16.9</td>
<td>0.31</td>
</tr>
</tbody>
</table>
by spherical indentation. The comparative load-displacement spherical indentation curves for studied materials are shown in Fig. 4.

![Indentation Curves](image)

**Fig. 4.** Vickers indentation curves for studied H15 and T30A carbide composites.

To determine mechanical properties more precisely the spherical indenter of relatively high elastic modulus $E=640$ GPa and Vickers hardness of about 1750 MPa was used. Used spherical indenter is made of hardmetal (WC-6 wt.%Co), and is commercially available from RedHill precision balls and roller producer.

2.3 Additional measurements

The indentations cavities (indents) parameters were inspected by Mahr profilometer. The cross-section profiles of the residual indents for studied carbide composites are shown in Figs. 5 and 6.

![Cross-section Dimensions](image)

**Fig. 5.** Indent cross-section dimensions of T30A cermet for 300 N indentation load.

The vertical (indent depth) axis is in micrometers, and the horizontal (indent width) axis is in millimeters.

The residual radius $R_r$ designated in the Figs. 5 and 6 as $R$ can also be found using equation:

$$\frac{1}{R^*} = \frac{1}{R} - \frac{1}{R_r},$$  \hspace{1cm} (1)

where $R^*$ - is the modified radius, can be found from simple relation (2); $R$ – is the indenter radius.

![Cross-section Dimensions](image)

**Fig. 6.** Indent cross-section dimensions of H15 hardmetal for 300 N indentation load.

The modified radius can be found from Hertz equation for contact displacement:

$$\frac{\mu_1}{h} = \frac{a^2}{R},$$  \hspace{1cm} (2)

where $a$ - is the projected contact radius.

3. RESULTS AND DISCUSSION

The results of indentation hardness measurements performed on a range of brittle solids demonstrate that the indentation pressure is indenter shape insensitive over a wide range of geometries. Observations (and numerical calculations) indicate that the plastic zone exhibits spherical symmetry, regardless of indenter geometry and that identical plastic zone boundaries develop for spherical and Vickers pyramidal indentations of equal volume [9]. In current study we have used the spherical indenter as the Hertz analytical model for stress assessment analysis is simple and in case of carbide composites the plastic deformations are very small, or can be neglected. The elastic straining response is desired and the appropriate stress conditions at relatively
shallow indentations can be achieved only by spherical indentation, see Fig. 7.

![Fig. 7. Schematic of indenter contact with a sample surface](image)

The main mechanical properties can be measured by indentation are yield strength or yield point (in case of elastic materials with low plastic deformations the proof stress determined as the yield at 0.2 % strain), Young’s modulus or modulus of elasticity and Poisson’s ratio.

The reduced Young’s modulus can be determined by equation derived by Hertz:

\[ E^* = \frac{3P}{4\sqrt{R^* h_c^*}} \],

where \( P \) - is the load applied on the indenter; \( R^* \) - is the modified radius; \( h_c \) - is the indenter elastic displacement.

The materials modulus of elasticity (\( E \)) can be then calculated from:

\[ \frac{1}{E^*} = \frac{1}{E_i} + \frac{1}{E} \],

where \( E_i \) - is the Young’s modulus of indenter.

The materials Poisson ratio is then

\[ E = \frac{E^*}{1 - \nu_i^*} \],

where \( \nu_i \) - is the Poisson’s ratio of indenter.

The most efficient way to extract effective stress-strain dependences form indentation data is to use the simple relations proposed by T. F. Juliano et al [10] for true indentation stress

\[ \sigma = \frac{P}{\pi a^2} \left( \frac{h}{h_c} - 1 \right), \]

where \( h_c \) - is the contact depth and \( h \) – is the total indenter penetration depth.

and modulus of elasticity

\[ E = \frac{1 - \nu^2}{\frac{4}{3P} \left( \frac{2(h-h_c)}{a^2+h_c^2} \right)^{2/3}} \],

where \( \nu \) - is the Poisson’s ratio of material.

The effective stress-strain curves based on calculations according to equations (6) and (7) from indentation data are shown in Figs. 8 and 9.

The offset yield point (proof stress, \( \sigma_{0.2} \)) for WC-15 wt.%Co (Grade H15) hardmetal is equal to approx. 2920 MPa.

![Fig. 8. Indentation effective stress-strain curve for H15 hardmetal](image)

The offset yield point (proof stress, \( \sigma_{0.2} \)) for TiC-30 wt.%Ni/Mo (Grade T30A) cermet is equal to approx. 1800 MPa.

![Fig. 9. Indentation effective stress-strain curve for T30A cermet](image)
curves and extrapolation of the effective stress and effective strain dependences, as the effective stress-strain graphs (see Figs. 8 and 9) are limited by the indentation stress. The yielding of the materials starts at the stress levels equal to 700 MPa and 850 MPa for H15 and T30A, respectively, and is apparent due to the measurable residual indentation depth seen in the Fig. 4. However, the 0.2 % offset stress is located far beyond the limits of the indentation test results, the indentation force is limited by the testing apparatus, and can not be visualized on the effective stress-strain curves.

The higher proof stress for WC-Co hardmetal can be addressed to the higher strain absorption ability of the tungsten carbide grains compared with titanium carbide phase. As shown by previous studies the elastic strain energy storing in the composite may relax either by origin and propagation of microcracks, or by a local plastic strain. The resistance of a composite to brittle fracture depends, therefore, on the ability of its carbide phase to undergo local plastic strain (to absorb fracture energy by local plastic strain), was proved by XRD studies [11]. Although the offset yield point results obtained in current study are in good agreement with proof stresses ($R_{0.2}$) for similar carbide composites published previously [11, 12].

4. CONCLUSIONS

The spherical instrumented indentation technique can be used as express and reliable technique to measure main mechanical properties of carbide composites (WC-based hardmetals and TiC-based cermets). Obtained load-displacement ($P-h$) curves can be transformed into effective stress-strain ($\sigma_{eff}, \varepsilon_{eff}$) curves similar to true stress-strain curves from compression testing.

The properties measured from stress-strain curves, such as, modulus of elasticity, Poisson’s ratio and yield strength $\sigma_y$ (or offset yield point $\sigma_{0.2}$) can be obtained even for very hard materials, if spherical indenter of higher than tested materials modulus of elasticity is used. The current study neglects the effect of microstructure evolution, surface roughness, residual stress, and many other factors that may affect the indentation response, that should be studied in future works.

5. ACKNOWLEDGEMENTS

This work was supported by the Estonian Ministry of Education and Research (project SF 014062s08) and the Estonian Science Foundation (grant No. 7889). Authors are grateful to Dr. Jüri Pirso from Powder Metallurgy Laboratory of Tallinn University of Technology and PhD Maksim Antonov from Department of Materials Engineering (TUT) for help in testing and personal communications.

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