EFFECT OF LIGHT ABSORPTION ON INDENTATION MODULUS OF DENTAL FILLING COMPOSITES

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Keywords: nanoindentation, dental filling composites, light absorption

1. Introduction

In many branches such as biomechanics, orofacial surgery and material engineering FEM analyses are used for different problem solving1. These analyses are used for the dental fillings lifetime improvement in order to prevent fracture of filling, loss of filling and creating of secondary caries on the tooth-filling interface. These failures are caused by the stress as an effect of the light activated polymerization process and occlusal loading. Several studies deal with optimization analyses and try to decrease the polymerization residual stresses in filling by different shaping of the cavity and/or by the manner how the cavity is filled2. These analyses require knowledge of exact mechanical properties of all materials used for FEM model including the dental filling composites. This work is focused on effect of polymerization light absorption on elastic modulus evaluating at a different depth of the material. As the most suitable method for local mechanical properties determination the nanoindentation method was chosen.

2. Materials and methods

Cylindrical specimens were made from dental filling composites (Filtek™ Supreme XT, Filtek™ Silorane and Charisma) with different chemical composition. The material was pushed into the epoxy mould, Fig. 1, covered by a transparent plastic film and pressed by a glass slide. Polymerization of the material was activated by the use of light curing unit (Translux Power Blue™, Heraeus Kulzer GmbH, Germany) for time period according to instructions for use. Each specimen was irradiated only from one side in order to simulate real conditions in tooth. These specimens were halved (diamond cut-off wheel, Ø150 × 0,4 × Ø12,7 mm, low speed, Struers) in order to evaluate effect of light absorption to elastic modulus at different depth of the material. The surface of the cut face was finished mechanically in grinding machine with abrasive papers of subsequently decreasing abrasiveness up to 4000-grit and then was polished. Thus prepared specimens were stick on magnetic metal thin plate in order to prevent movement of specimen during indentation.

Nanodma load controlled experiment was performed on Hysitron’s TriboLab® system (Hysitron, Inc., Minneapolis, USA) at the CTU in Prague. During the test a sinusoidal force was applied to a sample and the resultant amplitude and displacement were measured. Harmonic loading with dynamic load amplitude 0.02 mN was specified for a harmonic frequency 5–150 Hz. During the nanodma experiment was applied static load with maximum force \( P_{\text{max}} = 2 \text{ mN} \). A diamond tip \( E_i = 1141 \text{ GPa} \) and \( v_i = 0.07 \) was used for experimental measurements in room temperature conditions. The indentation matrix 3×7 (about separation of 15×15 µm) was placed in every 0.5 mm of depth to a value of 3.5 mm under the irradiated surface. The procedure of dynamic data analysis was adopted from Asif et.al.1. An automatically calculated data of the damping coefficient \( C_S \) and the contact stiffness \( K_S \) of the specimen are than used to determine the viscoelastic properties. The reduced storage modulus \( E_{r'} \), the reduced loss modulus \( E_{r''} \) of the specimen, and their ratio \( \tan \delta \) can be calculated by equations (1), (2) and (3), respectively:

\[
E_{r'} = \frac{K_S}{2A_C} \quad E_{r''} = \frac{\alpha C_S}{2\sqrt{A_C}} \quad \tan \delta = \frac{\alpha C_S}{K_S}
\]

where \( A_C \) is the contact area based on tip area function related to contact depth at quasistatic loading, and \( \alpha \) is the frequency [rad/s]. The storage and loss modulus of the sample \( E_i' \) and \( E_i'' \), respectively, are related to the reduced storage and loss modulus according to equations (4) and (5):

\[
\frac{1}{E_i'} = \frac{\left(1 - v^2\right)}{E_i'} + \frac{\left(1 - v^2\right)}{E_i''} \quad (4)
\]

\[
\frac{1}{E_i''} = \frac{\left(1 - v^2\right)}{E_i'} + \frac{\left(1 - v^2\right)}{E_i''} \quad (5)
\]

where the subscripts \( i \) and \( s \) refer to the indenter and sample materials, respectively, and \( v \) is the Poisson’s ratio. Poisson’s ratios of the material were determined from the measured ultrasonic velocities and the densities of the materials. Experi-

Fig. 1. A halved epoxy mould with composite specimen
mental measurement was carried out and analysed according to ultrasonic technique description\(^1\). Poisson’s ratios were set as \(\nu_s = 0.285\) for Filtek\textsuperscript{TM} Supreme XT, \(\nu_s = 0.28\) for Filtek\textsuperscript{TM} Silorane, and \(\nu_s = 0.3\) for Charisma.

### 3. Results

The values of reduced storage modulus and loss modulus are desired results of nanoDMA analyses. The typical example of average values of moduli of material Charisma is presented in Fig. 2. For all materials the ratio of storage and loss modulus didn’t reach over ten percent and the range of variation coefficient was around 10 ± 5 %.

![Fig. 2: Reduced storage and loss moduli of Charisma measured one day (left) and one month (right) after manufacturing](image)

**Fig. 2.** Reduced storage and loss moduli of Charisma measured one day (left) and one month (right) after manufacturing.

The mean values of complex moduli \(E\) [GPa] measured one day after specimens manufacturing on the upper layers are: \(E = 14.5\) for Filtek\textsuperscript{TM} Supreme XT, \(E = 11.8\) for Filtek\textsuperscript{TM} Silorane; \(E = 6.9\) for Charisma. The decrease of values of complex moduli in dependence on depth \(h\) [mm] is presented in Fig. 3.

![Fig. 3: The percentage expressed decrease of complex indentation modulus \(E\) in dependence on depth \(h\)](image)

**Fig. 3.** The percentage expressed decrease of complex indentation modulus \(E\) in dependence on depth \(h\).

### 4. Discussion

Good repeatability of measurements was observed for low frequency of 50 Hz and in range 50–100 Hz. For frequencies higher than 100 Hz the standard deviation was increasing. The ratio of storage and loss modulus increase in time. It means that the elastic behaviour of the material is more significant whereas the viscous component is suppressed.

### 5. Conclusion

Dynamic indentation method nanoDMA proper for viscoelastic properties testing was successfully applied on dental filling composites. The polymerization depth that is guaranteed by producer was in accord with obtained results. The smaller decrease of modulus caused by light absorption was observed in depth to 1.5 mm and more significant decrease in range of depth 1.5–3.5 mm.

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The intent of this study was to evaluate elastic modulus of three dental filling composites. NanoDMA was focused on mechanical properties of upper irradiated layers of the specimens and on deeper layers where the intensity of polymerization radiation decreased. The nanoindentation testing was performed using a Hysitron’s TriboLab\textsuperscript{®}. Poisson’s ratios of composites were obtained by ultrasonic measurement of mechanical properties of the same materials. First observation showed decrease in modulus due to light absorption.
THE EFFECT OF SURFACE ROUGHNESS ON NANOINDENTATION

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Keywords: nanoindentation, surface roughness, WC-C coating, nanohardness, indentation modulus

1. Introduction

Nanoindentation measurement is strongly affected by surface roughness. The reason is that the depth of penetration is determined from the zero point of initial contact of the indenter with the surface. Natural roughness of real surfaces causes uncertainty in the determination of the zero point and subsequently, of contact area with the surface asperities at shallow indentation depths which significantly increases the data scatter. The analytical models usually involve elastic or completely plastic contact and often take into consideration the statistical variation in asperity height in real surfaces.1

ISO 14577-1 standard requires that the surface roughness, Rs, is below 5 % of indentation depth, h (ref.2). In the case of thin coatings, an additional requirement of ISO 14577-4 is that the h is below 10 % of the coating thickness, d, to avoid substrate effects.2 However, real coatings often exhibit roughness, which does not satisfy this requirement. Therefore, the aim of this work is to investigate the effect of substrate and coating roughness on nanoindentation data.

2. Experimental procedure

The material of the substrate was a rod of tool steel 12 050. Seven samples with the diameter of 55 mm and thickness of around 4 mm were machined from the rod, tempered from 860 °C into oil and annealed at 200 °C. Their surfaces were ground with a diamond wheel with the grain size of around 100 µm. Two samples in the as-ground (A-G) state were used as the representatives of the highest surface roughness, and the remaining samples were polished up to 1 µm. Two of them represented the lowest surface roughness. Subsequently, one sample was ground on 80/63 diamond disc and the last one on the polishing cloth with 15 µm diamond spray. “Macro-roughness”, Rm, was measured using contact profilometry (model SJ 201, Mitutoyo) on ten lines with the length of 10 mm each. The distance between the lines was 1 mm. The value of Rm was determined as an average of these ten measurements. “Micro-roughness” was measured using an atomic force microscope (model Dimension Icon, Veeco). Two areas with the size of 50 µm × 50 µm were imaged in a tapping mode for each sample. Rv was determined as an average value from ten 10 µm × 10 µm areas arbitrarily distributed on both images.

Nanohardness, Hν, and indentation modulus, Eν, of the substrates were investigated using nanoindenter (model NHT, CSM, Switzerland) in sinusoidal loading mode up to 10 mN, 100 mN and partial unloading (Continuous Multi-Cycle – CMC) mode up to 400 mN. At least 20 indents were made in automatic mode on each sample and corresponding depth profiles were generated. The extreme curves were excluded from further analysis. The values of hardness and indentation modulus were determined as an average from the stable part of each depth profile.

PECVD WC-C coating with the thickness of around 500 nm has been deposited on all substrates simultaneously. The deposition conditions were as follows: total pressure 3 Pa, current density 1 mA cm–2 and bias voltage –5 kV. The roughness range is 3–300 nm in the very local areas (10 µm x 10 µm) free of any deposition artifacts. Coating roughness is often higher than the substrate roughness both in nano- and micro-range, especially at low Rv. The ratio of Rv of the coating and substrate is in Fig. 1. The roughness of the coating is dominant on well polished substrates and this effect is greatly suppressed when roughness of the substrate exceeds 130 nm. The same is valid for AFM measurements from Tab. II. This data suggests that large “coating grains” grew on flat surfaces due to limited number of nucleation sites. Vice versa, rough surfaces provide increased nucleation site density and the resulting coating consists of smaller grains, therefore, Rv is determined by the roughness of the substrate.

3. Results and discussion

Tab. I summarizes roughness data from the contact profilometer on the substrates and coatings. The range of Rs in the substrates is 0.02–0.6 µm whereas it is 0.1–0.6 µm in the coatings. Tab. II shows similar data for Rv obtained from AFM. The roughness range is 3–300 nm in the very local areas (10 µm x 10 µm) free of any deposition artifacts. Coating roughness is often higher than the substrate roughness both in nano- and micro-range, especially at low Rv. The ratio of Rv of the coating and substrate is in Fig. 1. The roughness of the coating is dominant on well polished substrates and this effect is greatly suppressed when roughness of the substrate exceeds 130 nm. The same is valid for AFM measurements from Tab. II. This data suggests that large “coating grains” grew on flat surfaces due to limited number of nucleation sites.

Table I

Roughness data on the substrates and corresponding coatings obtained by contact profilometry

<table>
<thead>
<tr>
<th>Substrate</th>
<th>1 µm</th>
<th>1 µm</th>
<th>15 µm</th>
<th>80/63</th>
<th>80SiC</th>
<th>A-G</th>
<th>A-G</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rs [µm]</td>
<td>0.024</td>
<td>0.024</td>
<td>0.135</td>
<td>0.412</td>
<td>0.45</td>
<td>0.502</td>
<td>0.573</td>
</tr>
<tr>
<td>Rv [µm]</td>
<td>0.098</td>
<td>0.099</td>
<td>0.175</td>
<td>0.662</td>
<td>0.622</td>
<td>0.555</td>
<td>0.623</td>
</tr>
</tbody>
</table>
data scatter, which increases with $R_a$. Hardness of the coating determined at the same conditions exhibits the same behavior regardless of the macro- or nano-roughness level (Fig. 3 and Fig. 4, respectively). The main effect of $R_a$ increase is the substantial increase of data scatter, especially in the case of nano-roughness measured using AFM. Indentation modulus exhibited the same behavior both in the substrate and in the coating.

4. Conclusions

Surface roughness of the examined WC-C coating increases on substrates with smaller roughness, probably due to unconstrained growth at the limited number of nucleation sites. Hardness (and indentation modulus) of steel substrates and WC-C coatings does not depend on $R_a$, it only substantially increases the scatter of the measurements.

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Current study revealed that surface roughness of WC-C coating increases with steel substrate roughness reduction and nanohardness (and indentation modulus) of substrates and coatings does not depend on $R_a$ it only substantially increases the scatter of the measurements.
MEASUREMENT OF PARAMETERS DETERMINING MECHANICAL PROPERTIES OF GRAINS WITH PARTICULAR ORIENTATION IN NON-ORIENTED ELECTROTECHNICAL STEELS

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Keywords: spherical indentation, crystallographic texture

1. Introduction

The non-oriented (NO) steels are mainly used as core materials for rotating equipments. Important parameters that determine good magnetic properties of electrical steels are crystallographic texture, grain size and chemical composition. These properties of NO steels are provided by (100) [0vw] so called “rotating” cube texture. However, this texture state is not achieved in the practice so far. It is important to notice that maximal attention in the field of the research and development of NO steels is concentrated on achievement of the cube texture component intensity in the plane of the steel sheet.

2. Experimental material and procedure

As experimental material was used NO electrical steel (FC) that was taken from industrial line after cold rolling with 74 % of deformation and subsequent recrystallization annealing in laboratory conditions (800 °C/10 min). The chemical conception of the experimental material is presented in Table I.

The study was carried out with nanoindentation testing method. To determine the hardness a triangular Berkovich indenter has been used. The sample were measured with the Quasi Continuous Stiffness Measurement (QCSM) module at a maximum force of 100mN. The QCSM module allows measuring the contact stiffness depth – dependent at one and the same position of the sample. In addition, the sensitivity of the measurements will be raised in the small force range, so that stiffness values may be readily determined even for very small forces and depths. In order to determine stress-strain curves a spherical indenter with radius 21 μm has been used. The measurements have been carried out using a new method where the stress-strain curve of metals is determined with a neural network from indentation measurement with spherical indenter.

3. Results and discussion

The microstructure of the investigated steel obtained after laboratory heat treatment is presented in Fig. 1. This sample was subjected to annealing at 950 °C/5 min. As one can see, the material is characterized by columnar microstructure with average grain size about 200 μm.

Fig. 1. Microstructure of FC material

The micro and nano-indentation were carried out in the FC material passed the laboratory annealing. It means that investigated grains in material characterized by single crystals oriented in space with low defects (dislocation) density. Inverse Pole Figure (IPF) map of the cross section-plane obtained from the sample in Fig. 1b is presented in Fig. 2. This map is used for defined of grains crystallographic orientation in investigated material. We have chosen one grain from each orientation; G (011), D(111), C(001) in which was performed nanoindentation measurements.

Fig. 2. IPF map representing the columnar grains in cross – section of FC steel

Hardness results

Five measurements were carried out in every grain (C, D, G). As one can see, there is the significant difference in hardness values and modulus values between the grain orientations Fig. 3.

Measurement of stress – strain curves

The 9 measurements in a 3 × 3 array in a distance of 50 μm have been carried out in each grain. Fig. 4a, b, c show the impressions.
It can be seen that not all indents are rotationally symmetric. This is the influence of the crystallographic orientation. The measurement curves have been averaged and only the average curve has been analyzed Fig. 5. As one can see between the individual grain orientations is a significant difference and each of these grain orientations have different intensity of deformation.

4. Conclusions

On the basis of differences in deformation behaviour of grains with particular crystallographic orientation it can be suggested, that for a given deformation stress the highest value of strain will be reached in D(111) orientated grains.

This fact is important in optimizing the degree of temper rolling for strain induced grain growth in non-oriented electrotechnical steel. For the investigated steel the recommended deformation is over 6 %.

This work was carried out within the frame of the project “Technology of preparation of electrotechnical steels possessing high permeability for high affectivity electromotors”, ITMS 26220220037 financed through European Regional Development Fund. Special thanks are due to Dr. Tomás Chudoba for nanoindentation measurements and Franziska Kairat from ASMEC Advanced Surface Mechanics GmbH.

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The aim of the present work is to determine the difference in mechanical properties between the grains possessing three predefined orientations (with (100); (110) and (111) planes perpendicular to the loading) by the spherical indentation method. By means of spherical indentation technique was measured a significant differences in deformation behavior between grain orientations in polycrystalline material, which is more difficult to determine by another method (such as microhardness test or uniaxial tensile test).
DIAGNOSTICS OF DEFECTS IN THE FORGED BLADES USED IN POWER INDUSTRY

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Keywords: Blades, Cr-martensitic steel, power plant, microstructure, fractography analysis

1. Introduction

The purpose of this paper is to determine the causes of cracks on forged blades, designed for turbine wheels of low pressure parts in 1,000 MW turbines. Visual inspection identified cracks in the middle of shovel blades (Fig. 1, No. 1), in the transition area of the lock (Fig. 1, No. 2) and in the area of upper groove lock (Fig. 1, No. 3) developed during the final machining operations. The blades were manufactured by die forging of stainless steel X12CrNiMoV12-3 (the numerical designation 1.4938). The initial semiproduct was rod KR 110 mm, pre-forging was done at temperatures from 950 to 1050 °C and then finally forged at temperatures form 900 to 1100 °C. At the end of forging the straightening of blades was performed. The blades were then heat treated 1020 °C / oil + 620 °C / water and annealed to achieve a stress relief at 580 °C in order to meet regulatory requirements for yield strength min. 800 MPa, ultimate strength of 1000 MPa, elongation min. 14 % and the notch toughness of 55 J in longitudinal direction.

2. Experimental

Cracks, which were found in three areas of blades, were first identified using a magnetic method (see Fig. 1). Consequently, microstructural analysis of undamaged blades and blades with cracks was carried on a light microscope (Zeiss Neophot 32) and TEM (JEOL JEM2000FX and EDS microanalyzer LINK AN10000).

Samples for microstructural analysis were taken from areas around the cracks and the leaf blades lock in the transverse and longitudinal direction. Samples for light microscopy were metallographically prepared by standard and chemically etched (Vilallia Bain). The structures were taken at magnifications of 100, 200 and 500 times. The microstructure of the basic material had the typical structure of Cr tempered martensitic steel (see Fig. 2) and showed characteristics of needle-like structure of martensitic phase. Dispersivity and orderliness of carbide phases did not show significant roughness nor orientation or localization of carbides in the areas of original austenite grains. Cracks were filled with corrosion products with different morphologies. At the crack tip and in the region of the secondary propagation, cracks had intergranular course, were homogeneous and black colored. In the region of crack root they were wider, grayish and non-integral, which is typical for cracking of wustit phase (FeO). Observed cracks had the character of cracks resulting from the heat.

Thin foils for TEM were prepared by standard1 and subsequently electropolished in a 6% HClO4 in methanol at −35 °C and current 150 mA.

The analysis confirmed the martensitic structure with needle-like morphology and increased density of dislocations. In the fracture surfaces of interfaces within the martensite grains the large amount of M23C6 type carbide particles was observed, which precipitated during steel processing. Within intact blades the occurrence of carbide particles was lower1.

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Fig. 1. Cracks identified by magnetic method in three areas

Fig. 2. Crack in the region of blade lock and martensitic structure of matrix
Control of chemical composition of the material and the original cast blades was conducted on quantometer ARL 3460 OE. The results showed compliance with the chemical composition of all blades. Samples for microchemical analysis that was completed in Cameba MICRO microanalyzer were taken from corrosively attacked cracks surface. The results confirmed the presence of iron oxides. Oxides also showed a reduced concentration of Cr. Their formation therefore had to happen at temperatures of 1000–1100 °C at high temperature oxidation where highly porous wustida was created.

Fig. 3. a) Carbide particles M23C6 in dark field; b) microdiffraction from the carbide indicated in Fig. 3a, zone [021] of carbide M23C6

Fractographic analysis of samples with forging cracks was performed, images were obtained by scanning electron microscope JSM 840A and JSM 5510 LV. Samples were taken from the leaf blade with the original parts surfaces after forging (A) and the blade lock (B). Two zones were observed within the sample A that differed by the degrees of coloration, which identified different layer micromorphology. Zone I contained corrosion products and Zone II contained high temperature oxides. The fracture surface showed a mixed fracture with a significant proportion of intercrystalline decohesion (Fig. 4). Transcrystalline failure could not be unambiguously determined due to the layer of corrosion products, which was unremovable. The shape of the technological fracture beginning of B sample suggested that the fracture itself branched repeatedly along the grain boundaries and locally also the spread across the grain boundaries was observed. On the fracture surface signs of decohesion were found and locally also a transcrystalline quasi-brittle fracture was observed. The analysis of samples A and B implied that cracks were formed during forging, increased C content in the crack tip showed the influence of oil quenching.

Fig. 4. Occurrence of micromorphological characters of intercrystalline fracture of sample A (left zone I, right zone II)

Finally the mechanical properties were inspected. All measurements met the requirements.

3. Discussion and conclusions

No significant deviations from the material requirements, which could lead to cracks in the forged blades, were found by the analyses. Observed cracks were typical for those resulting from the heat during the two processes.Forging initiated the formation of intercrystalline cracks and austenitization annealing lead to the growth of thicker oxides. The cracking occurred due to improper forging procedure, where most likely an interval of forging temperatures was not kept. Susceptibility to fracture may be attributed to the high incidence of M23C6 carbides, which precipitated during steel processing. For blades with no cracks carbide phase occurred in much smaller quantities. Modified Cr martensitic steels showed significant carbide precipitation in supercooled austenite in the range of 700–800 °C while delays were tens of minutes. From the nature of the defects it can be assumed that the failure was caused by the finishing of forging process including straightening of blades within the forging temperatures interval.

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The subject of this article is to determine the causes of cracks and their character within the forged blades of turbine wheels of low-pressure parts in power plant. From the Cr martensitic stainless steel forged samples of blades were prepared. Visual inspection discovered surface cracks that were caused by drop forging. Causes of forging cracks in terms of microstructure and chemical composition were analyzed. Also fracture surfaces of samples of damaged blades were documented and fractography analysis was performed.
CONTACT STRENGTH AND CRACK FORMATION IN MONOLITHIC SiC AND MoSi₂

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Keywords: contact test, microstructure, cone cracks

1. Introduction

Conventional tensile tests for determination of strength of ceramics describe failure behaviour related to a simple stress state which is mostly uniaxial with insignificant gradients. Considering practical applications, mechanical loading leads to an inhomogeneous multi-axial stress state which can be simulated by line or point loading. The line or point loading is induced by two opposite rollers or spheres, respectively. Weibull analysis is commonly used for describing strength of brittle ceramics. It is defined by the characteristic strength \( \sigma_0 \) and the Weibull parameter \( m \) which is the measure of the scatter in strength values. The Fett’s theory which defines relationships between parameters of the Weibull analysis for the four-point bending test (\( \sigma_{0,bend}, m_{bend} \)) and single-cycle contact test using rollers (\( \sigma_{0,cont}, m_{cont} \)) suggests

\[
m_{bend} \approx 2 m_{cont,r}, \quad \sigma_{0,bend} \approx \sigma_{0,cont,r}
\]

The characteristic strength \( \sigma_{bend} \) and \( \sigma_{cont} \) results from experimental values of \( m_{bend} \) and \( m_{cont} \), respectively, defined as

\[
\sigma_{bend} = \frac{3P(S_i - S_j)}{2W^3}, \quad \sigma_{cont,r} = \frac{1.96P}{W}
\]

where \( S_i \) and \( S_j \) represent outer and inner spans, respectively. \( W \) and \( t \) are dimensions of a sample along directions parallel and perpendicular to a direction of the applied force \( P \) at failure, respectively. Finally, the stress \( \sigma_{cont,s} \) along with the Young’s modulus \( E \) have the forms

\[
\sigma_{cont,s} = \frac{1 - v_m^2}{3\pi} \left[ \frac{6P E_s^2}{R^2} \right]^{1/3} + \frac{1}{E} \left( \frac{1 - v_s^2}{E_s} + \frac{1 - v_m^2}{E_m} \right)
\]

where \( E_s, v_s \) and \( E_m, v_m \) are the Young’s modulus, the Poisson’s ratio for the spheres and a ceramic material, respectively.

The paper deals with the determination of contact strength of two monolithic ceramic materials using opposite rollers and spheres methods as well as with the verification of the Fett’s theory validity.

2. Experimental materials and tests

Two monolithic ceramics have been investigated: (1) silicon carbide (SiC) prepared at the Institute of Inorganic Chemistry, Bratislava, Slovakia; (2) MoSi₂ prepared at Cerwis, Erlangen, Germany.

Structure of the materials was studied using scanning electron microscopy on polished and etched samples. The bending test was applied at a load of \( S_i = 40 \text{ mm}, S_j = 20 \text{ mm} \). The contact mode by standard hardened steel rollers with the diameter \( D = 3 \text{ mm} \) was applied to specimens with the dimensions \( W \times t \times L = 3 \times 4 \times (10-15) \text{ mm} \). The load \( P \) of this test increased up to a value of the specimens. The contact mode by standard hardened steel spheres with the radius \( R = 2.5 \text{ mm} \) was applied to specimens with the dimensions \( W \times t \times L = 3 \times 4 \times 25 \text{ mm} \). In this case, a load increase was stopped at \( P = 4.9 \text{ kN} \), i.e. prior to failure of specimens with an aim to investigate a character of cracks. With regard to Eq.(3), material parameters of SiC, MoSi₂ and standard hardened steel (SHS) of spheres were used.

3. Results and discussion

Microstructure of SiC consists of fine submicron-sized equiaxed grains with a low aspect ratio, with inter-granular phase located at thin grain boundary films as well as at triple junctions. Three different phases are identified in MoSi₂, i.e. MoSi₂ grains with average size of 7 \( \mu \text{m} \), amorphous SiO₂, and low amount of MoSi. The SiO₂ particles are present at triple junctions of the MoSi₂ grains, and occasionally precipitates in the MoSi₂ grains. Pores with size of 25 \( \mu \text{m} \) are the only defects present. Table I presents the Weibull characteristic strength and bending strength of both materials. MoSi₂ with lower fracture toughness exhibits \( \sigma_{cont,s} / \sigma_{bend} = 8.3 \), and SiC exhibits its ratio approx. 7.6. In contrast to the contact test by rollers, a correlation between the bending mode and the contact mode by spheres can not be defined. Fig. 1 shows SEM micrographs of cross section views of (a) SiC and (b) MoSi₂ loaded by the contact test using spheres for \( R = 2.5 \text{ mm} \), up to \( P = 4.9 \text{ kN} \) and \( P = 1 \text{ kN} \), respectively. SiC exhibits multiple cone cracks with a perpendicular behaviour below a contact surface, followed by a linear behaviour. These cone cracks arise to a critical size during the loading and they are a reason of the failure and strength degradation. Multi-crack formation is connected with deformation of spheres and with an enlarged contact surface at the load increase.

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Fig. 1 shows SEM micrographs of cross section views of (a) SiC and (b) MoSi₂ loaded during contact test using spheres for $R = 2.5$ mm, up to $P = 4.9$ kN and $P = 1$ kN, respectively.

The paper deals with a determination of contact strength of monolithic ceramic materials (SiC, MoSi₂) using rollers or spheres. SiC exhibits multiple cone cracks and high contact strength during the test between spheres. In contrast to SiC, the MoSi₂ ceramics behaves as a quasi-plastic material and exhibits a median crack during the contact test by spheres, which is a reason of lower contact strength and higher Weibull modulus. The contact strength between rollers are similar to bending strength of both materials.

4. Conclusions

The Fett’s theory was proved for MoSi₂ but is not valid in the case of SiC due to presence of large processing defects. SiC exhibits multiple cone cracks and high contact strength during the test between spheres. In contrast to SiC, the MoSi₂ behaves as quasi-plastic materials and exhibits a median crack during the contact test by spheres, which is the reason of lower contact strength and higher Weibull modulus. The contact strength between rollers are similar to bending strength of both materials.

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VICKERS INDENTATION FRACTURE TOUGHNESS OF HVOF SPRAYED WC-BASED COATINGS

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1. Introduction

The high hardness and the resistance against the spreading of cracks are crucial properties of all kinds of materials, intended as wear resistant. While hardness is a well known and easily measured parameter, the measurement of fracture toughness still remains rather problematic in the case of coating materials. In the paper, fracture toughness of the HVOF sprayed WC-12%Co and WC-17%Co coating was evaluated by the Vickers indentation test in dependence on used load. The applicability of using different equations is discussed with respect to the size indentation effect of IFT.

2. Experimental

The WC-Co coatings were sprayed by the HVOF TAFA JP5000 spraying equipment onto a grid blasted steel surface, using a spraying procedure standard in ŠKODA VÝZKUM s.r.o. The thickness of the coatings varied from 350 to 400 µm. The cross sections of the samples, embedded in an epoxy resin, were mechanically grinded and polished using the Struers automatic polishing machine. The CSEM Scratch Tester, equipped with the Vickers indentor, was used to make the indentations in to the coatings cross sections. The 25, 50, 75, 100, 125, 150, 175 and 200 N loads were used, for each load 16-20 indents were made in dependence on their size. The space between the individual indents equaled at least 3 indents diagonal to avoid their mutual affecting.

The lengths of indents diagonals, as well as the lengths of cracks (Fig. 1), were measured by NICON EP IPOHT 200 microscope with software LUCIA. For the \( K_C \) calculations, the equations proposed by Chicot\(^2\) at condition of the Palmqvist, Intermediate and Radial-median cracking mode were used:

\[
K_C(r_{(\text{Palmqvist})}) = 0.0154 \left( \frac{E}{H_v} \right)^{1/3} \left( \frac{L}{a^{1.5}} \right) \tag{1}
\]

For Palmqvist cracks:

\[
K = 0.0089 \left( \frac{E}{H_v} \right)^{1/3} \left( \frac{L}{a^{1.75}} \right) \tag{2}
\]

For Intermediate cracks:

\[
K_{(r_{(\text{Inter})})} = (0.0074 - 0.0043q) \left( \frac{E}{H_v} \right) \frac{L}{a^{2.5}} e^{1.5r} \tag{3}
\]

where \( L \) is the used load [N], \( E \) is the elastic modulus, \( H_v \) is the Vickers hardness, and \( q \) is the coefficient, developed by Chicot\(^2\), involving the Mayers low of indentation size effect.

3. Results and discussion

The results of \( K_C \) calculations are summarized in the Fig. 2, 3 and 4. In the case of WC-12%Co the transition between he Palmqvist and Radial-median cracks according the condition of \( c/a \sim 2.5 \) is at indentation load 75 N, in the case of WC-17%Co at 125 N. It means that for lower loads, the Eq. (1) should be used, while for higher loads the Eq. (2) is

![Fig. 1. Schematic picture of the Vickers indent with cracks spreading in the anisotropic microstructure thermally sprayed coatings](image-url)
proper. From the experimental data it is clearly understood, that the $K_\text{IC}$ calculated according Eq. (1) and (2) are load dependent in the same manner as the Vickers microhardness. Incorporating the Mayer index to the $K_\text{IC}$ equation, the load dependance dissolved. The $K_\text{IC}$ value is constant across all range of used load and the unique average value can be determined, which is not possible to do for $K_\text{IC}$ determined according Eq. (1) and (2). On the other hand, the value of $K_\text{IC}$ (M-M) is very low due to high value of the q index.

Fig. 2. The load dependence of measured HV and $K_\text{IC}$ of WC-12% Co coating

Fig. 3. The load dependence of measured HV and $K_\text{IC}$ of WC-17% Co coating

Fig. 4. Comparison of $K_\text{IC}$ (M-M) results for WC-based coatings

Comparing the $K_\text{IC}$ values of the two WC-based coatings with different Co content, the lower fracture toughness of coating with lower Co content met the expectation. The unique average value of $K_\text{IC}$ (M-M) from all measurement without the respect to used load are $0.72 \pm 0.05$ for WC-12% Co and $1.53 \pm 0.15$ for WC-17%Co.

4. Conclusion

The study of WC-based HVOF coatings Vickers fracture toughness showed a strong load dependence of $K_\text{IC}$ values, calculated according to usually used equations. Distinction on the cracks on Palmqvist or Radial-median according to the rule of $c/a \sim 2.5$, or according to the slope of the $c - L$ dependence is useless regarding the selection of the proper $K_\text{IC}$ equation. The equation, proposed by Chicot for intermediate cracking mode is load independent in the all range of used load. It enables to determine one unique $K_\text{IC}$ value, comparable with measurement at different loads. The difference between the coatings with different Co content match the expectations – the $K_\text{IC}$ of WC-17%Co is higher compare to $K_\text{IC}$ of WC-12%Co. The used method enables to evaluate the fracture toughness of thermal sprayed coatings and represent the influence of their microstructural features. The unique load-independent $K_\text{IC}$ value is favourable for comparing the results measured at different load conditions.

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The Vickers indentation fracture toughness of HVOF sprayed WC-12%Co and WC-17%Co coatings was evaluated in dependence on used load. Three different equations for $K_\text{IC}$ value was used and their results compared. It was shown, that while usually used equations for both Radial-median and Palmqvist crack modes are strongly dependent on used load, the equation for intermediate cracking mode incorporating the Mayer index, is load-independent and enable the comparison of results.