DETERMINATION OF MECHANICAL PROPERTIES OF $MoSi_2$ COMPOSITES BY NANOINDENTATION

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Introduction

The $MoSi_2$ phase is a borderline ceramic–intermetallic compound with both covalent and metallic atomic bonds. Besides traditional use as heating elements, materials based on $MoSi_2$ appear to be promising candidates for wide variety of high-temperature structural applications, where high strength and temperature durability is required[1,2].

The main disadvantage of $MoSi_2$-based composites is low fracture toughness, low hardness in lower temperatures. Several ways of improving the performance of $MoSi_2$-based composites have been developed by incorporating SiC, ZrO_2 or HfO_2 particles into the matrix.

The aim of this work was to investigate mechanical properties of $MoSi_2$-based composites with reinforced 10 wt.% of ceramics particles: SiC, nanoSiC, Si_3N_4, ZrO_2, HfO_2 particles were used.

Depth sensing indentation technique[3] was used to determine mechanical properties in local scale represented by indentation depth of 100 nm and in global scale represented by the depth of 1000 nm.

2. Materials and experimental procedure

The materials were prepared by powder metallurgy technology using a high-energy milling process. The mechanical treatment was started from coarse-grained elemental powders (Mo and Si) with diameters of 100–500 μm and reinforced materials in order to prepare materials with 10 % of SiC, nanoSiC, ZrO_2, HfO_2 and Si_3N_4. The powders were milled using the steel ball milling set in argon environment. The milled powders were densified by hot pressing at 1550 °C in vacuum.

The experimental materials, incorporated with secondary particles were supplied in the form of bars. For investigation were samples mounted, grinded and polished.

The microstructures of investigated materials are shown in Fig. 1–6. Fig. 1 shows material of monolithic $MoSi_2$ in cross-polarized illumination. The average grain size of mono-

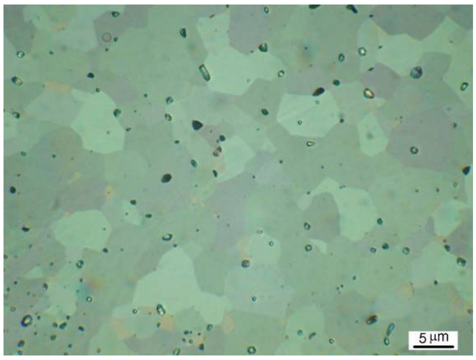

Fig. 1. **The microstructure of monolithic $MoSi_2$**

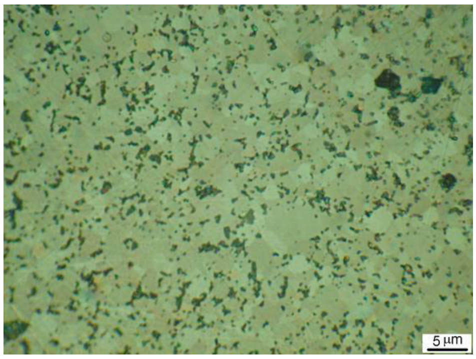

Fig. 2. **The microstructure of $MoSi_2$-based composites with incorporated 10 wt.% nanoSiC particles**

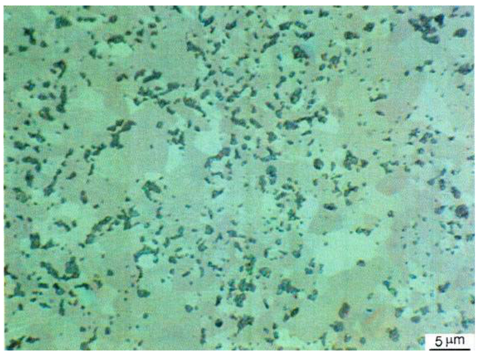

Fig. 3. **The microstructure of $MoSi_2$-based composites with incorporated 10 wt.% SiC particles**

lithic $MoSi_2$ is 5 µm. Incorporation of particles (SiC, ZrO_2, Si_3N_4) causes reduction of grain size growing, increasing ratio of pores, while HfO_2 particles cause opposite behaviour. The glassy SiO_2 phase was found in the microstructure in both cases too.

Depth sensing indentation tests were carried out using Nanoindenter XP with Berkovich tip. Over 50 indents were placed on each of investigated materials. The CSM (Continuous Stiffness Measurement) was used to show difference between properties measured in local and global scale. In the case of local properties the mean indent area is smaller then the area of one grain, while the global properties represent two and more grains.

The instrumented hardness (H_{IT}) and indentation modulus (E_{IT}) were evaluated in two indentation depths. The influence of incorporated particles on the properties of particular grains was investigated in the depth of 100 nm. The influence of grains boundaries and pores on properties was investigated in global scale of 1000 nm.

The goal of this contribution is to describe the effect of incorporated particles on the hardness and modulus for different scales.

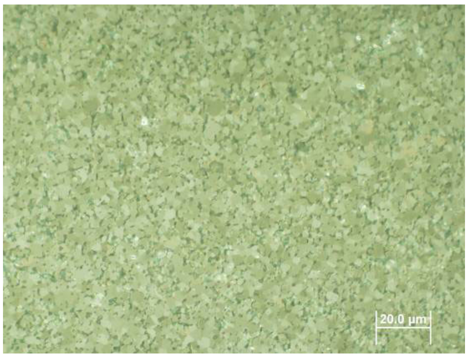

Fig. 4. **The microstructure of $MoSi_2$-based composites with incorporated 10 wt.% ZrO_2 particles**

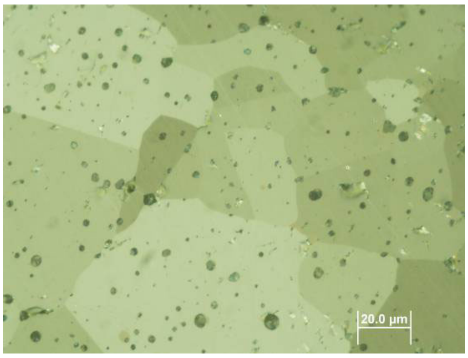

Fig. 5. **The microstructure of $MoSi_2$-based composites with incorporated 10 wt.% HfO_2 particles**

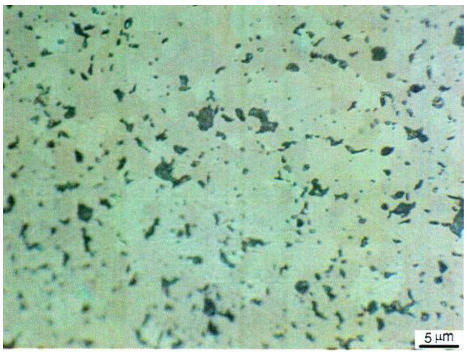

Fig. 6. **The microstructure of $MoSi_2$-based composites with incorporated 10 wt.% Si_3N_4 particles**

3. Results and discussion

The results of indentation experiments presented as: Hardness (H_{IT}) *vs*. indentation depth and modulus (E_{IT}) *vs*. indentation depth relations are shown in Fig. 7 and Fig. 8. The hardness and modulus values decrease with increasing indentation depth. The pores were observed in the microstructure of investigated materials. Therefore the hardness and modulus values decrease because of plastic zone under the tip closes to a phase intersecition. The presence of pores and their size distribution are documented in Fig. 1–6 and in the Table I. Hardness and modulus values were extracted from the depth of 100 nm to avoid the influence of pores on the improvement of mechanical properties by incorporation of particles. The modulus values and hardness values are summarized in the Table II and III. The hardness and modulus values were extracted from the depth of 1 µm in order to describe the effect of pores.

Both, hardness and modulus values in 100 nm depth are the highest for composites with 10 % Si_3N_4 and with 10 % nanoSiC particles. Similar results were measured for the maximum depth, too. The smallest difference between the average hardness and modulus values determined in local and global scale was measured in the material with ZrO_2 particles, but the scatter of the results was the highest. The grain size of

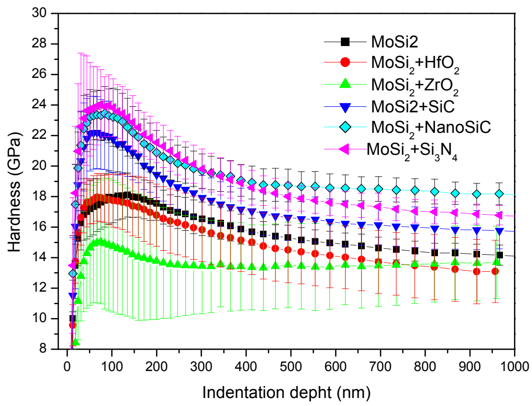

Fig. 7. **Hardness (H_{IT}) – indentation depth dependence of investigated materials**

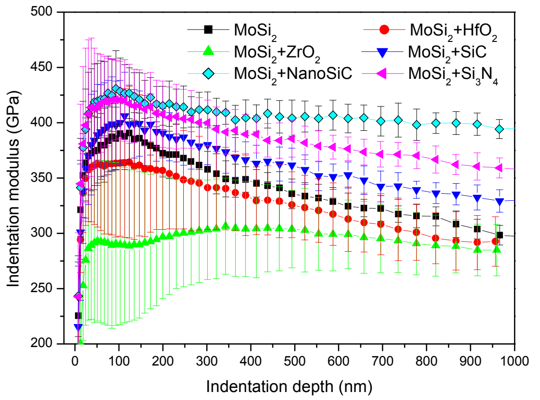

Fig. 8. **Indentation modulus (E_{IT}) – indentation depth dependence of investigated materials**

Table I
The size and area ratio of pores found in investigated materials

10 %	MoSi$_2$ + HfO$_2$	ZrO$_2$	nano SiC	SiC	Si$_3$N$_4$	monolith
Pore size, µm	1–2	2–4	–	1–2	–	0.1–1
Area ratio, %	10	5.6	–	10	–	0.7

Table II
Modulus of elasticity (GPa) determined in the depth 100 nm (E_{IT}100) and 1000 nm (E_{IT}1000)

10 %	MoSi$_2$ + HfO$_2$	ZrO$_2$	nano SiC	SiC	Si$_3$N$_4$	monolith
E_{IT}100	352	294	414	398	415	385
STD	65	86	41	31	33	24
% COV	18	29	10	7	8	6
E_{IT}1000	295	294	395	331	357	297
STD	30	22	12	10	9	9
% COV	10	8	3	3	3	3

this material is the smallest. The measured results in local scale are therefore strongly affected by both grain boundaries and pores.

Table III
Hardness (GPa) determined in the depth 100 nm (H_{IT}100) and 1000 nm (H_{IT}1000)

10 %	MoSi$_2$ + HfO$_2$	ZrO$_2$	nano SiC	SiC	Si$_3$N$_4$	monolith
H_{IT}100	18	15	23	22	24	18
STD	1.8	5	1.7	1.8	1.6	1.4
% COV	19	32	8	8	7	8
H_{IT}1000	12	13	18	16	16	14
STD	2	2	0.8	1	0.6	0.7
% COV	17	15	4	7	4	5

4. Conclusions

The depth sensing indentation method is usable to determine mechanical properties of composite materials; however it is very important to take in account the microstructure inhomogeneities, like pores, etc.

It is possible to determine the homogeneity of materials based on the scatter of measured results.

The highest hardness and modulus values were measured on materials with incorporated nanoSiC and Si$_3$N$_4$ particles.

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P. Zubko[a], B. Ballóková[b], L. Pešek[a], and O. Bláhová[c] *([a] Department of Material Science, Technical university of Košice, Košice, [b] Slovak Academy of Science, Institute of Materials Research, Košice, Slovakia, [c] New Technologies Research Center, West Bohemian University, Plzeň, Czech Republic):* **Determination of Mechanical Properties of MoSi$_2$ Composites by DSI**

The paper deals with the determination of mechanical properties of MoSi$_2$ composites with incorporated SiC, nanoSiC, Si$_3$N$_4$, HfO$_2$ and ZrO$_2$ particles. The hardness and modulus of the investigated composite materials were determined. The highest hardness and modulus values were obtained in the case of MoSi$_2$ with nanoSiC and Si$_3$N$_4$ particles. The great influence of pores was detected.