

INDENTATION TESTING OF MoSi₂ BASED COMPOSITES

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Abstract

The paper deals with the study of some of the basic mechanical properties of novel composite systems based on molybdenum disilicide (MoSi₂) with an addition of zirconia (ZrO₂) or hafnia (HfO₂) nanoparticles and their comparison to a monolithic MoSi₂ prepared by the same method. The measurements were carried out using the instrumented indentation technique where values of hardness and modulus of elasticity were simultaneously obtained for various indentation depths up to 600 nm in order to identify the possible load size effect or influence of the surface layers affected by machining. No such effects, which would significantly alter the results, have been observed. The results for all experimental materials were compared and related to their composition.

Keywords: *mechanical properties, hardness, modulus of elasticity, molybdenum disilicide*

INTRODUCTION

Molybdenum disilicide is one of the intermetallic compounds which offer the combination of a high melting point (2030°C), moderate density (6.23 g/cm³), high thermal conductivity and elevated oxidation resistance [1]. Mechanical properties of MoSi₂ could be improved by the addition of ceramics strengthening, for example: SiC and Si₃N₄ particles, nano SiC particles. They are thermodynamically compatible with MoSi₂, have a high elastic module and they are available as particles, fibers or whiskers, [2,3]. Composite materials can be considered as multiphase systems consisting of a matrix with particles dispersed within its bulk. Size, morphology, volume fraction and spatial distribution of secondary phases affect the final properties of dispersal strengthened material.

The aim of this work was to investigate the mechanical properties of MoSi₂ and MoSi₂-based composites reinforced with ceramics particles ZrO₂ and HfO₂.

Depth sensing indentation technique was used to determine the mechanical properties in local regions and statistically treated to obtain global properties.

EXPERIMENTAL MATERIALS AND METHODS

The experimental materials studied in the present work were prepared by the powder metallurgy technique of controlled reaction sintering in argon atmosphere.

The starting material was prepared in the Fraunhofer Institut für Fertigungstechnik und Angewandte Materialforschung in Dresden, Germany, according to the patent [4,5]. The procedure is based on High Energy Milling (HEM) of coarse grained (grain size between 100 and 500 μm) Mo and Si powders of high purity. After milling for several hours in an argon atmosphere in the planetary mill „Pulverisette 5“ (made by Fritsch, Germany), highly dispersed powder mixtures were obtained. During preparation of the composites, ceramic dispersoid particles (ZrO₂, HfO₂) were being added into the matrix at

various volume fractions. Finally, the milled mixtures were compacted by pressing to high density (over 95% of the theoretical density) and subsequently subjected to pressureless reaction sintering. The sintering was carried out in vacuum conditions at 1500°C.

In order to use the instrumented indentation technique, all specimens were ground and polished to obtain a mirror-like surface.

Instrumented indentation was carried out on a nano-indentation tester TTX-NHT (made by CSM Instruments, Switzerland). A Berkovich (three-sided) pyramid diamond tip was used in continuous multicycle (CMC) loading/unloading mode. In this mode a single indentation is loaded in a stepwise manner. At each force step, the tip is partially unloaded by a specific fraction of the load step. Each force step then provides two pieces of information: total depth of elastic/plastic penetration and measure the recovery from that load. This procedure enables one to calculate not only hardness but also various other variables (for instance indentation modulus of elasticity, elastic and plastic indentation depth, contact depth, energies of elastic and plastic deformation, etc.) as functions of depth of penetration of the indenter [6,7]. This method is often used for characterisation of coatings and thin layers or as a quick procedure how to map the depth profiles of measured characteristics in materials which might have mechanically heavily affected surfaces.

In this study the maximum load of the first cycle was 1 mN, the loading was increased up to 100 mN in 30 steps in a quadratic maximum load increment. After reaching the maximum load of each step, an unloading down to 90% of that value followed. The resulting load-penetration (P-h) curves were evaluated according to the analysis of Oliver and Pharr [8] and values of hardness (H) and elastic modulus (E) as functions of depth were found. Each specimen was subjected to at least 20 indentations and the results were statistically evaluated.

RESULTS AND DISCUSSION

The microstructure of the experimental materials was studied in greater detail elsewhere [9]. Microstructures can be observed in the Figs.1, 2, and 3, which show the polished surfaces of the experimental materials. They also illustrate the size and placing of the Berkovich indents after the testing. All indents correspond to the final load of 100 mN and were placed in a rectangular grid with 20 μm distance from each other.

In all materials the microstructure was well developed. The matrix grains had the size about 5 μm in the monolithic material (Fig.1). The material with an addition of ZrO_2 had heterogeneous microstructure, the secondary phase was uniformly distributed (Fig.2). Incorporation of particles caused a reduction of grain growth. This material tends to contain the highest volume fraction of the residual porosity, which reached about 5%, and the pore mean size was from 2 μm to 4 μm . Figure 2 illustrates very clearly the source of the scatter in resulting nanoindentation data as differences between various indented places can be appreciated.

Microstructure of $\text{MoSi}_2\text{-HfO}_2$ (Fig.3) consisted of the largest matrix grains (10 μm) with small pockets of HfO_2 glass, which, however, were generally larger than the ZrO_2 nanoparticles in the other composite. The microstructure of this material was virtually pore-free.

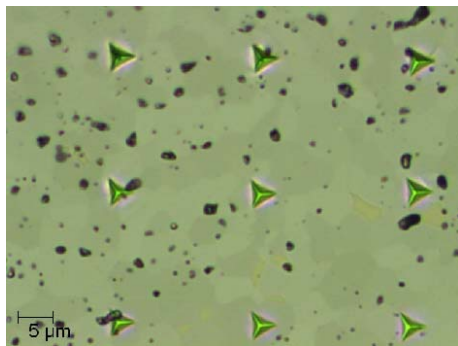


Fig.1. Polished surface of the monolithic MoSi_2 with nine Berkovich indents made by 100 mN load.

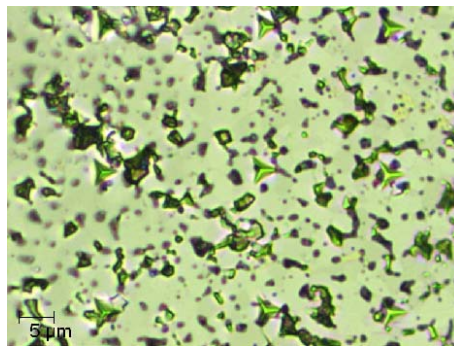


Fig.2. Polished surface of $\text{MoSi}_2\text{-ZrO}_2$ with Berkovich indents. Heterogeneity of the microstructure and its effect on the indentation can be appreciated.

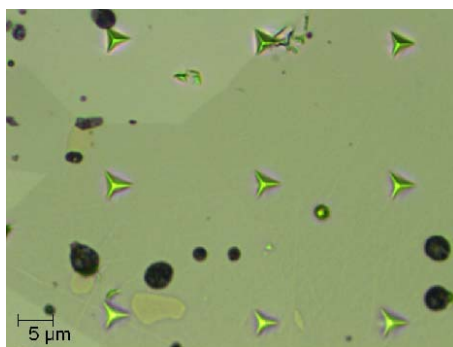


Fig.3. Polished surface of $\text{MoSi}_2\text{-HfO}_2$ with indentations.

Figure 4 summarizes the results of hardness measurements for all three materials. It shows the hardness values in relation to the depth of penetration. From the plot it seems that in all cases a not very strong load size effect is present. In general, the hardness values decrease slightly with increasing penetration (i.e. load). For a small penetration the scatter of all data is relatively large due to issues with finding a proper contact at very low loads. Also, at low loads, the results are more influenced by heterogeneity of the microstructure, as can be appreciated especially in the case of $\text{MoSi}_2\text{-ZrO}_2$ (Fig.2). At higher loads the stress field under the indenter is large enough to include large volumes of the microstructure where the differences average out. Here it can be seen that the composites have higher hardness than the monolithic MoSi_2 with $\text{MoSi}_2\text{-HfO}_2$ being the hardest material in the entire measured interval. The composite with ZrO_2 particles has, for the low loads, hardness similar to that of the monolith. This is probably due to the nature of its surface which could not be polished to the same quality as the other two materials. For the depths above 300 nm (corresponding to ~ 25 mN load) the $\text{MoSi}_2\text{-ZrO}_2$ is harder than the monolith, even considering the scatter of the data.

When compared with typical results of macro-indentation, the measured values are higher because the instrumented hardness is defined differently but are in quite good agreement with nanoindentation measurements on similar materials made by the slightly different technique of dynamic depth sensing indentation, the sinus mode [9]. Nevertheless,

the tendency corresponds to that found previously for analogous MoSi_2 based materials by the traditional Vickers method [10]. On the other hand, this tendency is at variance with the findings in [11], where an almost exactly opposite ranking was identified.

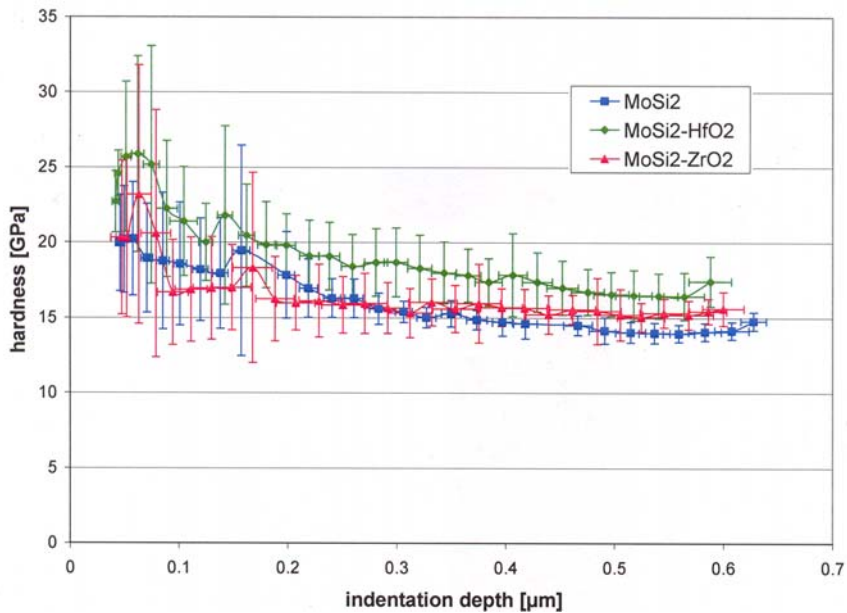


Fig.4. Depth profile of hardness of the experimental materials measured by instrumented indentation.

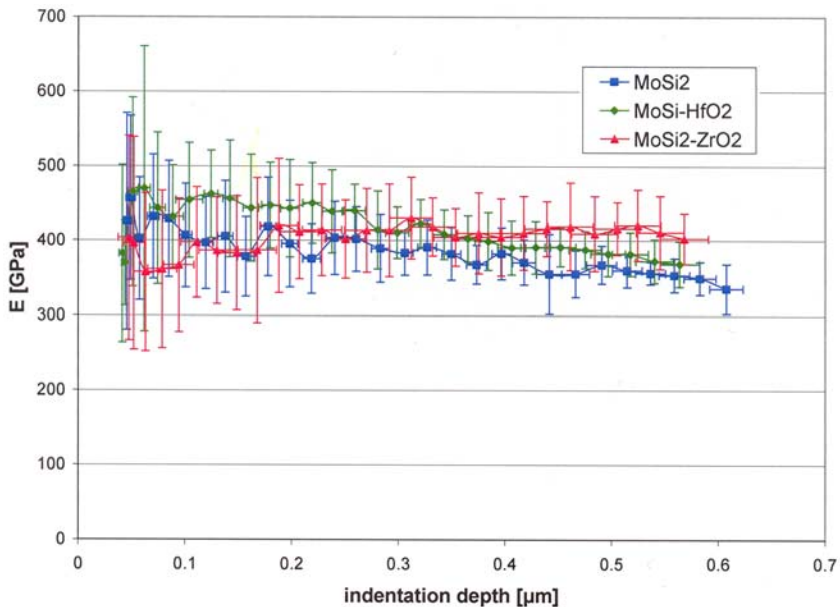


Fig.5. Modulus of elasticity of the three experimental materials measured by instrumented indentation as function of depth of penetration of the tip.

Figure 5 shows the values of modulus of elasticity for all experimental materials for loads up to 100 mN. The values are constant within the errors and correspond to the value from literature, 440 GPa [12]. There is a slight tendency, particularly for higher loads, suggesting that the $\text{MoSi}_2\text{-ZrO}_2$ composite is the stiffest material, while the monolith has the lowest elasticity of the three. This again is different from the results in [11]. These results can be a consequence of relatively large scatters of the data which are very close to each other. Then very little local differences may lead to different final tendencies.

CONCLUSIONS

Three MoSi_2 based materials were prepared by the same processing route: monolithic MoSi_2 and two composites $\text{MoSi}_2\text{-ZrO}_2$ and $\text{MoSi}_2\text{-HfO}_2$. All experimental materials were investigated by the progressive loading/unloading technique of instrumented indentation at loads from 1 up to 100 mN and indentation hardness and indentation were measured. It was found that all three materials had very similar values of measured mechanical properties with a little load size effect in hardness values. The composite $\text{MoSi}_2\text{-HfO}_2$ was the hardest material and monolithic MoSi_2 the softest one. The modulus of elasticity was in good agreement with the literature data and was very similar within the errors of measurement for all experimental materials.

Acknowledgement

The work was financed by the projects VEGA 2/0025/11.

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