INSTRUMENTED INDENTATION OF MoSi₂ BASED MATERIALS

M. Henžel*, J. Kovalčík, J. Dusza, A. Juhász*, J. Lendvai*

Institute of Materials Research, Slovak Academy of Sciences, Watsonova, 47, 043 53 Kosice, Slovak Republic
tel.: +421 556 3381 15, fax: +421 556 3371 08
*Department of General Physics, ELTE, Budapest, Hungary
e-mail: henzel@imrnov.saske.sk

As-recieved and as-deformed MoSi₂ have been studied using an instrumented hardness testing device. Micro-nanoindentation tests at loads from 10 to 2 000 mN were performed on the as recieved and pre-strained (at 1300ºC and 15 MPa, 24 hours) MoSi₂ using depth-sensing method. The Martens, universal and classical hardness values were calculated at different indentation loads. The indentation load size effect was calculated directly from loading curves. According to the results the pre-strain reduces the micro-nano hardness values of the investigated material, probably due to the activated slip systems during the high-temperature deformation.

Key words: molybdenum disilicide, instrumented indentation, pre-strain, Martens hardness, universal hardness, depth-sensing curves.

Molybdenum disilicide (MoSi₂) is a candidate material for high temperature structural applications as a furnace heating element and an electrical conductor in silicon intergrated circuit design or parts of engines [1]. MoSi₂ is known for a high melting point of 2030ºC, exhibits excellent high temperature oxidation resistance, and possesses many convenient properties such as high stiffness, high thermal conductivity, relatively low density, and high strength at elevated temperatures [2]. However, a major difficulty in the application of MoSi₂ as a structural material has been a lack of ductility and fracture toughness at temperature range 900–1400ºC. Toward this temperature range, with the onset of dislocation climb and diffusional creep processes, does MoSi₂ show significant plasticity in compression, bending and tension in both single crystal and polycrystalline materials [3, 4, 5]. Many approaches have been taken to reduce brittle-ductile transition temperature of MoSi₂ or to enhance the capability for plastic flow and obtain increasing of thougness at temperature range 900–1400ºC. The crystal structure of MoSi₂ is tetragonal ( C11b type), space group 14/mmm [6, 7]. The lattice parameters are a=0,3205 nm and c=0,7845 nm with c/a=2,45 (Fig. 1). MoSi₂ is also reported to have the hexagonal C40 structure above 1900ºC [8].

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In this material the dislocation-density limitation outgoes from a lack of overdue numbers of surface or internal dislocation sources. There is existing absence of knowledge concerning the relative mobilities of edge and screw dislocations, and information on the different dislocation types \(<100>\), \(<110>\), \(1/2<111>\) and \(1/2<331>\), their glide planes, and the operative slip systems as a function of temperature, strain rate and crystallographic orientation is only partially developed [9, 10].

Studies of the slip systems by means of hardness indentation for single crystal MoSi\(_2\) has found that primary and secondary slip systems were \{100\}<001> and \{110\}<001> respectively [9, 10, 2]. Berkowitz et al. [11] reported that \{110\} is the slip plane in single crystal MoSi\(_2\) deformed between 625 and 1125\(^\circ\)C under compressive load along three different directions. They concluded that the slip direction is \(<11\bar{1}>\). Umakoshi et al. [12] reported that slip occurs in \(<3\bar{3}\bar{1}>\) directions on both \{110\} and \{103\} planes.

The materials used in this investigation were monolithic MoSi\(_2\) prepared by Cesiwid, Erlangen, Germany. Samples for microstructure analysis were prepared using standard procedure and investigated using optical microscopy, as well as scanning and transmission electron microscopy (SEM and TEM). Microstructure of MoSi\(_2\) is shown in Fig. 2 [13]. Prestrain (Fig. 3) was performed by compressive creep test at the applied load of 15 MPa at 1400\(^\circ\)C for 24 hours. This procedure may cause increasing of dislocation density in tested material.
For hardness tests mirror polished samples have been used. The depth sensing tests were performed with Shimadzu DUH device with Vickers sharp indenter for all investigations [14]. Nominal peak loads of 10 to 2 000 mN were used and the dwell time at maximum load was 10 seconds in these experiments.
Total penetration depth $h$ (including elastic deformation) and relations for the Vickers geometry gives an apparent hardness called universal or Martens hardness. Universal hardness can be calculated from the equation as follows:

$$Hu = \frac{F}{26,43h^2},$$

where $F$ is applied load.

With a known Youngs modulus of the tested material, an analytic solution separates the contribution of elastic deformation, converting $Hu$ into the conventional hardness $Hv$, which is related to the plastic indent size [15]. $Hv$ is calculated from the equation as follows:

$$Hv = 4.Hu / \{1 + \sqrt{(1 - 12.Hu / E^*)}\}^2,$$

where $E^*$ is the effective contact stiffness, which can be determinate from the following equation:

$$E^* = \{(1 - v_i^2) / E_i + (1 - v_s^2) / E_s\}^{-1},$$

where $E_i$ is Youngs modulus of indenter
$v_i$ is Poissons ratio of indenter
$E_s$ is Youngs modulus of the tested material
$v_s$ is Poissons ratio of the tested material.

The next figures 5, 6 present the different F-h curves obtained for the MoSi$_2$ intermetallics under study. Values of universal hardness and conventional hardness are recorded in the same figure. Both universal and conventional hardness present obvious load size effect. The approximate value of hardness can be taken from steady state curve of universal and conventional hardness. Shape of the depth-sensing curve shows elastoplastic behaviour of MoSi$_2$. Values of conventional and universal hardness taken from
steady state curves are recorded in table 1. Both state as-received and as-deformed are presented.

Figures 7, 8 show the shapes of both states used in the study. Both shapes of the depth-sensing curve present elasto-plastic behaviour of MoSi_2. There is variety in shape of both curves. As-deformed state shows larger area of plastic deformation, material stiffness is decreasing. This effect is caused by high-temperature deformation in pre-strain material. The total indentation work also acquires higher values in pre-strained material. Looking at the table 1 it can be seen, that values of both universal and conventional hardness are decreasing in as-deformed state.

### Table 1

<table>
<thead>
<tr>
<th>Max. load [mN]</th>
<th>As-received state</th>
<th>As-deformed state</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Hu</td>
<td>Hv</td>
</tr>
<tr>
<td>500</td>
<td>7,5–8</td>
<td>10–11</td>
</tr>
<tr>
<td>50</td>
<td>10–11</td>
<td>13,5–15</td>
</tr>
</tbody>
</table>
Shapes of the depth-sensing curve obtained after instrumented indentation present elasto-plastic behaviour of MoSi$_2$.

Shapes of the load-depth curves are different for the as-received and as-deformed materials with larger area of plastic deformation (lower hardness) in the case of as-deformed material. This effect is probably caused by introduction of slip systems during high-temperature deformation in pre-strained material.

The total indentation work also acquires higher values in the pre-strained material. Values of both universal and conventional hardness are lower in the as-deformed state compared to the as-received material.

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АПАРАТНЕ ВИЗНАЧЕННЯ МІКРОТВЕРДОСТІ СПОЛУК НА ОСНОВI MoSi2

М. Гензель*, Й. Ковальчик, Й. Душа, А. Юхаш*, Й. Лендвай*

Інститут дослідження матеріалів, Словачька академія наук
ул. Ватсона, 47, 043 53 Коціє, Республіка Словаччина
тел.: +421 556 3381 15, факс: +421 556 3371 08
* Відділ загальної фізики, ЕЛТЕ, Будапешт, Угорщина
e-mail: henzel@imrnov.saske.sk

Сполуку MoSi2 одразу після отримання та деформації вивчали за допомогою пристрою для вимірювання мікротвердості. Тести на мікром'ятини при навантаженнях від 10 до 200 мН були проведені лише на щойно отриманих зразках (витриманих при температурі 1 300°С і тиску 15 МПа протягом 24 годин). Універсальну та класичну твердість Мартенса розраховано при різних навантаженнях. З кривих навантаження оцінено розмірні ефекти. З отриманих результатів вивчено деформації зразків при заданій температурі і тиску зменшують значення мікро/нано твердості досліджуваного матеріалу. Це відбувається, ймовірно, унаслідок процесів, що мають місце при високотемпературних деформаціях.

Ключові слова: дисилікат молібдену, апаратне визначення мікротвердості, деформація, твердість Мартенса, універсальна твердість, криві навантаження.

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