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INSTRUMENTED INDENTATION OF $MoSi_2$ BASED MATERIALS

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As-recieved and as-deformed $MoSi_2$ 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) $MoSi_2$ 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 ($MoSi_2$) 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]. $MoSi_2$ is known for a high melting point of 2030ºC, exhibits excellent high temperature oxidation resistance, and posseses 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 $MoSi_2$ 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 $MoSi_2$ 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 $MoSi_2$ or to enhance the capability for plastic flow and obtain increasing of thougness at temperature range 900–1400ºC. The crystal structure of $MoSi_2$ 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). $MoSi_2$ is also reported to have the hexagonal C40 structure above 1900ºC [8].

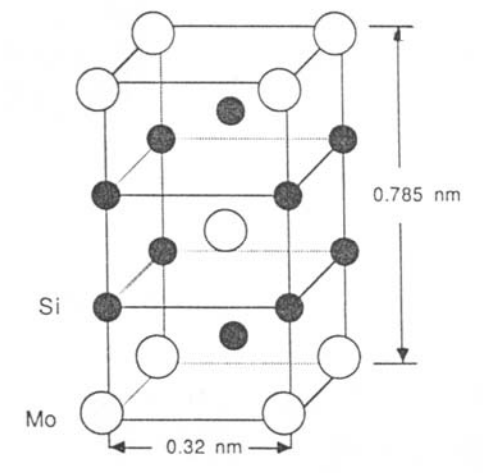

Fig. 1. Tetragonal unit cell of $MoSi_2$

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 knowlegde 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 1125ºC under compressive load along three different directions. They concluded that the slip direction is <1$\bar{1}$0>. Umakoshi et al. [12] reported that slip occurs in <3$\bar{3}$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 1 400°C for 24 hours. This procedure may cause increasing of dislocation density in tested material.

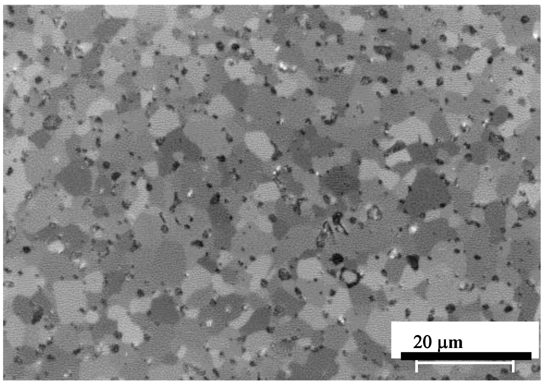

Fig. 2. Microstructure of $MoSi_2$

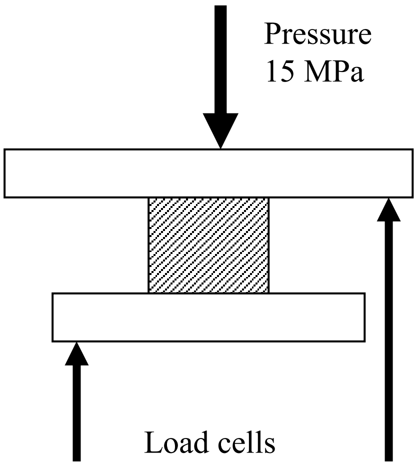

Fig. 3. Pre-strain of $MoSi_2$

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.

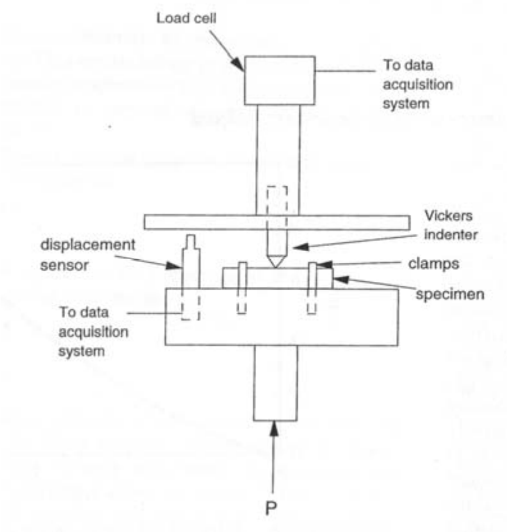

Fig. 4. Instrumented testing device

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:

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

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:

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

where E_i is Youngs modulus of indenter

ν_i is Poissons ratio of indenter,

E_s is Youngs modulus of the tested material,

ν_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 elasto-plastic behaviour of $MoSi_2$. Values of conventional and universal hardness taken from steady state curves are recorded in table 1. Both state as-recieved and as-deformed are presented.

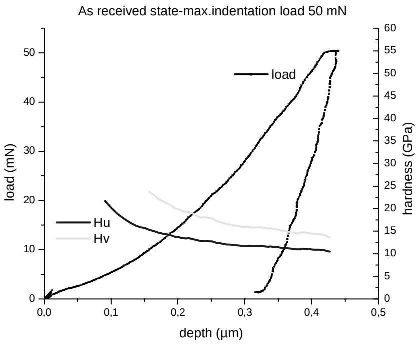

Fig. 5. Depth-sensing curve of as-received state with max. load 50 mN

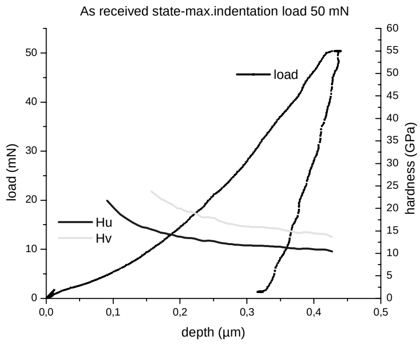

Fig. 6. Depth-sensing curve of as- deformed state with max. Load 500 mN

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

Hardness calculation from P-h curves

Max. load [mN]	**Hardness [GPa]**			
	As-recieved state		**As-deformated state**	
	Hu	**Hv**	**Hu**	**Hv**
500	7,5–8	10–11	5–5,9	5,2–7,5
50	10–11	13,5–15	8–10	10–12,5

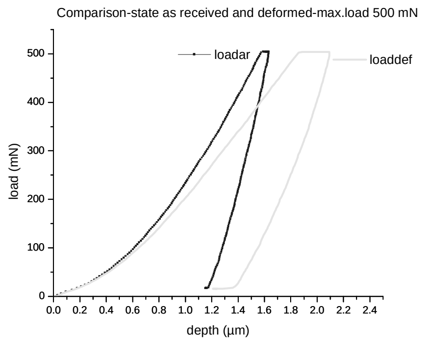

Fig. 7. Comparison of as-recieved state and as-deformed with max. load 500 mN

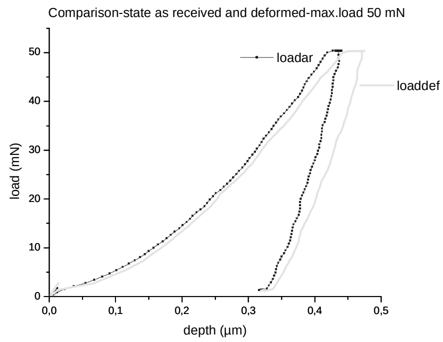

Fig. 8. Comparison of as-recieved state and as-deformed with max. load 50 Mn

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 ОСНОВІ $MoSi_2$

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Сполуку $MoSi_2$ одразу після отримання та деформації вивчали за допомогою пристрою для вимірювання мікротвердості. Тести на мікровм’ятини при навантаженнях від 10 до 200 мН були проведені лише на щойно отриманих зразках (витриманих при температурі 1 300^{0}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 робимо висновок, що витримка зразків при заданій температурі і тиску зменшує 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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