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VARIABLE THERMAL LOADING ANALYSIS OF (110) SINGLE CRYSTAL TUNGSTEN

ANALIZA SPREMENLJIVE TERMIČNE OBREMENITVE VOLFRAMOVEGA (100) MONOKRISTALA

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The temperature response of properties of single crystal tungsten (110) is still not well understood. Tungsten was chosen to illustrate the temperature dependence behaviour because of its isotropic elastic behaviour at low loads. All the mechanical properties are temperature dependent. The experiments were performed with tailor made Berkovich tip of radius 100 nm at 265 K, 373 K, 473 K and 623 K to study the behavior of tungsten single crystal at various temperatures. The phenomenon of material under the indenter, bouncing back at the end of unloading due to the accumulation of energy was observed. It was noted that the elastic recovery was lower at higher temperature. The experiments showed the onset of the first strain burst, the onset of plastic deformation in connection with periodic bursts, and the softening effects. Pile up, significant drop in hardness, change of elastic modulus and increase in displacement with increasing temperature were observed. Because of softening, the indentation depth is increased for the same loading conditions. Clear bursts were seen showing the nucleation of dislocations. At higher peak loads, the indentation contact in tungsten was not just elastic. This work attempted to explore the complete behaviour of metals at various temperatures, including the initial burst, the complete elastic recovery, the softening effect, the modulus and hardness.

Keywords: Nanoindentation, Mechanical Properties, Tungsten, Effect of Temperature

Vpliv temperature na lastnosti monokristala volframa (100) še ni popolnoma razjasnjen. Volfram je bil izbran za prikaz temperaturne odvisnosti zaradi elastičnega izotropnega vedenja pri majhnih obremenitvah. Vse mehanske lastnosti so odvisne od temperature. Preizkusi so bili opravljeni s prirejeno Berkovichovo konico s polmerom 100 nm pri temperaturah 265 K, 373 K, 473 K in 623 K, da bi ugotovili vedenje monokristala volframa pri različnih temperaturah. Opažen je bil pojav v materialu, da se vtis po razbremenitvi s konico sprosti zaradi velike nakopičene energije. Elastična poprava je bila manjša pri visokih temperaturah. Poskusi prikazujejo začetek vtisa in plastično deformacijo, ki je povezana s periodičnimi vtisi in učinke mehčanja. Kopičenje materiala, zmanjšanje trdote in elastičnega modula ter povečanje razmika pri povišanju temperature je bilo tudi opaženo. Zaradi mehčanja se povečuje globina vtisa pri enaki obremenitvi. Deformacije je spremljal nastanek dislokacij. Pri velikih obremenitvah kontakt pri vtisu ni bil popolnoma elastičen.

Ključne besede: nanovtis, mehanske lastnosti, volfram, vpliv temperature

1 INTRODUCTION

The ability to perform nanotest measurements at elevated temperatures opens up significant new possibilities. The behavior of the material is different when subjected to temperatures deviating from the room temperature and the temperature response of tungsten is still not clearly understood. Thorough study of the behaviour of the materials in different temperatures is very important for the design and applications of materials for different operating conditions.

Experiments were already performed on (100) tungsten [1]. It was found that the yielding under contacts can produce a 250 nm displacement extrusion. Nano-indentation experiments on tungsten revealed that the load displacement was not linear and an analytic technique was proposed for determining the contact area at peak load [2].

Experiments were performed on single crystal ionic materials with ultra sharp tips with $R < 10$ nm and special attention was given to the elastic response before the onset of plastic yield [3]. The load displacement curves exhibit periodic bursts in indenter penetration depth that was interpreted chiefly as consequence of the nucleation of dislocations. Plastic deformation in polycrystalline copper films clearly revealed the existence of a significantly higher density of dislocations around the nanoindentation. The characterisation of mechanical properties of thin films using spherical tipped indenters were investigated, also [4] and it was shown that the use of very small spherical tipped indenters provided a better solution of the contact problem. The role of substrate and interface adhesion on the force-displacement behavior of thin films indented with spherical tipped indenters was discussed, also [4].

The analytical formulation of the elastic limit predicting the location and slip character of a homogeneously nucleated defect in crystalline metals extends this formulation to the atomic scale in form of an energy-based local elastic stability criterion was investigated [5]. A fundamental framework for describing incipient plasticity that combines results of atomistic and finite-element modeling, theoretical concepts of structural stability at finite strain and experimental analysis were discussed, also [5]. Detailed interpretation of the experimentally observed sequence of displacement bursts was proposed to elucidate the role of secondary defect sources operating locally at stress levels considerably lower than the ideal strength required for homogeneous nucleation [6]. The advancements in making low dimensional structures from inorganic and organic compounds, determining the resulting, and necessarily local properties and assembling complex structures were explained[7].

The homogenous nucleation of dislocations was found in dislocation-free single crystals to be related to a sudden jump in the force-displacement curve. Experimental results of dislocation loop nucleation show good agreement with the continuum theory of dislocations[8]. It is found that the dislocations with a screw component are shown to glide across {111} planes and by a cross-slip mechanism giving rise to revolving terraces in the neighborhood of the nanoindentation trace with their edges parallel to compact n directions [9]. Molecular dynamics simulation showed that the burst and arrest of stacking faults were the key factors for the plastic deformation of nanocrystalline copper under nanoindentation [10]. High temperature nanotesting with micro materials measuring technology introduced the technique of high temperature nanoindentation [11]. The experiments were carried out on gold, soda-lime glass, fused silica and a polyimide. Results from fused silica show that its mechanical properties exhibit completely different temperature dependence from those of soda-lime glass, as expected since fused silica is an anomalous glass [12]. The small scale hardness and elastic modulus measurements on glass, gold, and single crystal silicon at room temperature and 473 K, show that the hardness and elastic modulus of soda lime glass and gold are lower at 473 K than at room temperature. In contrast, indentation testing of Si (100) at 473 K produced a similar hardness value to that obtained at room temperature, although the modulus was again reduced, from 140.3 GPa to 66.0 GPa. The 'pop out' event observed during unloading of a silicon indentation at room temperature, disappeared at 473 K [13].

The temperature response of properties of tungsten at high temperature is still not well understood, as relatively little research was focused on its high temperature behaviour [14] and the effect of temperature on dislocation nucleation process is not well understood. The behaviour of the material during loading and unloading was analyzed to understand the temperature effects on the reconstruction of the material during the removal of load. Emphases were placed on defects generation mechanisms during the elastic plastic contact of crystals and special attention was given to the elastic response before the onset of plastic yield. This work attempted to explore the complete behavior of tungsten at increased temperatures, including the initial burst, the complete elastic recovery, the softening effect, the modulus and the hardness.

2 EXPERIMENTAL METHODOLOGY

In the present investigation, the effects of temperature on properties of single crystal tungsten (110) were studied. The experiments were performed with tailor made Berkovich tip of radius 100 nm at temperatures of 265 K, 373 K, 473 K and 623 K. To perform the experiment at high temperature, insulating material was used to protect the piezoelectric setup of the indenter. A small heater capable of maintaining a constant temperature was added to the stage. The hot stage itself consisted of a thermally insulating ceramic block attached to the nanotest sample holder. The sample surface was brought to a constant temperature before the indentation for each experiment. A new attachment was made to circulate dry nitrogen gas to avoid ice formation during the low temperature experiments. The loading rate was kept constant for different temperatures experiments. The size of the sample was 2 mm thick and 9 mm in diameter. It took about 15 min. to reach the required temperature. The force on the sample during the imaging was of 2 mN. The topography and gradient images were captured to show the surface morphology after the indentation. In-situ imaging provides the capability to observe and quantify material damage while minimizing the time for material recovery. In the case of thin hard films on soft substrates, for instance, the indentation depth should generally not exceed 10 % of the film thickness in order to preclude any influence of the substrate. The sample was prepared with an advanced technique using the ultra precision machining technique producing a very fine surface. This method does not affect the orientation. But in case of other polishing methods, there is possibility of changing the orientation of the sample. Compared to other methods, this method also avoids the surface oxide formation.

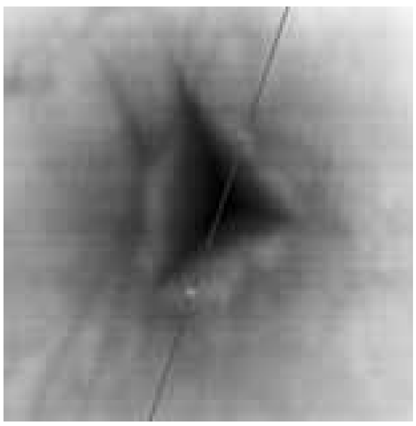

Figure 1: Image of the sample after indentation (6 μm)
Slika 1: Posnetek preizkušanca po vtisu (6 μm)

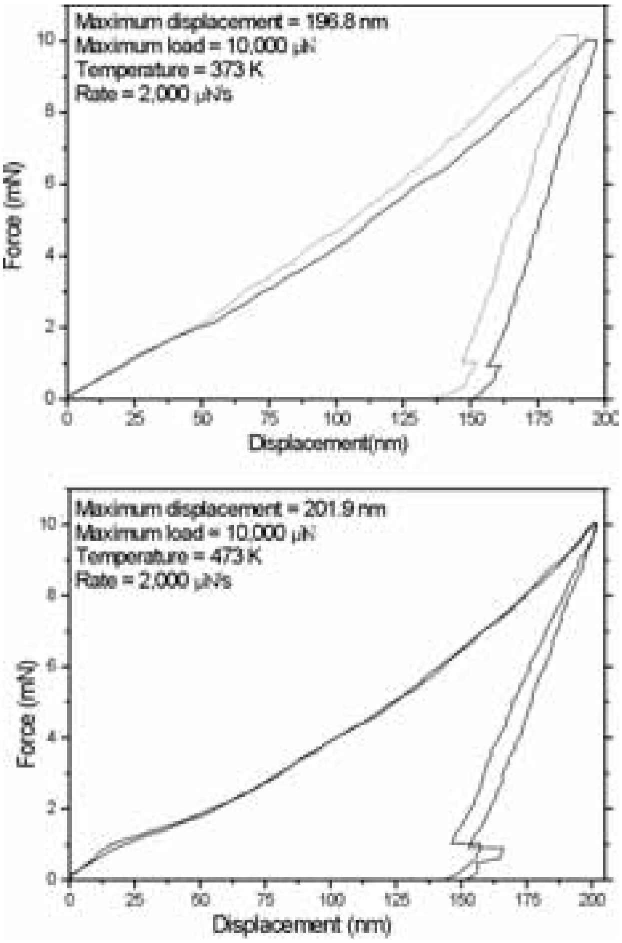

Figure 2: Loading and unloading pattern at 373 K (a) and 473 K (b) for 10,000 μN and loading rate of 2000 μN/s. Multiple curves indicate the experiments at different locations on the sample. The curves starting from the origin till the maximum point indicate the loading.

Slika 2: Značilnosti obremenitve in razbremenitve pri 373 K (a) in 473 K(b) pri 10 000 μN in hitrosti obremenitve 2000 μN/s. Krivulje pomenijo vtise na različnih mestih na preizkušancu. Krivulji z začetkom v koordinatnem središču pomenita obremenitev.

3 RESULTS AND DISCUSSION

Figure 1 shows the AFM image of the sample after the indentation. The tip imprint is very clear. The displacement of the material on the surface indicates the dislocation pile-ups of the material during the indentation.

Figure 2 shows the loading and recovery process during loading and unloading at different temperatures and at the maximum load of 10 000 μN and loading rate of 2000 μN/s. These curves were obtained for the purpose of comparison during the loading and unloading process and to observe the maximal penetration depth and elastic rebound. The end of unloading curve in **figure 2** shows the sudden bump in the unloading curve for a short distance. Also, we observed a small increase in the penetration depth indicating the softening effects at higher temperatures. The softening effects were due to increase in temperature and plasticity. At high temperatures, the material was subjected to great plastic global deformation, instead of periodic local burst of dislocations at low temperature. The pile up was clearly visible an explained with the rotation of the axis of the atoms to conserve the volume during the penetration of the indenter. We could also note the elastic rebound at the end of loading.

Figure 3 shows the loading and recovery process during loading and unloading at higher temperature. The

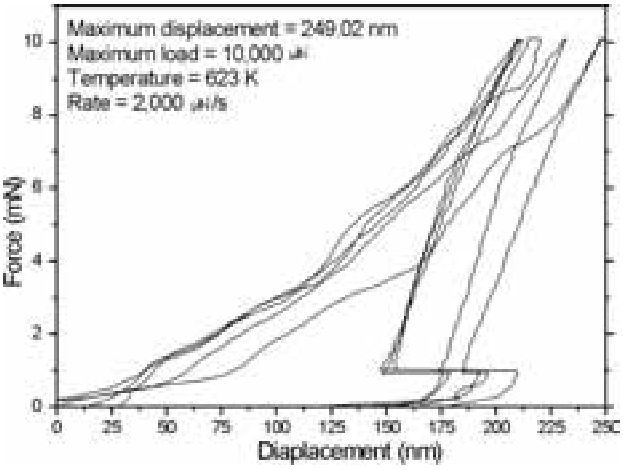

Figure 3: Loading and unloading pattern at 623 K for 10,000 μN and loading rate of 2000 μN/s. Multiple curves indicate the experiments at different locations on the sample. The curves starting from the origin till the maximum point indicate the loading.

Slika 3: Značilnosti obremenitve in razbremenitve pri 623 K pri 10 000 μN in hitrosti obremenitve 2 000 μN/s. Krivulje pomenijo vtise na različnih mestih na preizkušancu. Krivulji z začetkom v koordinatnem središču pomenita obremenitev.

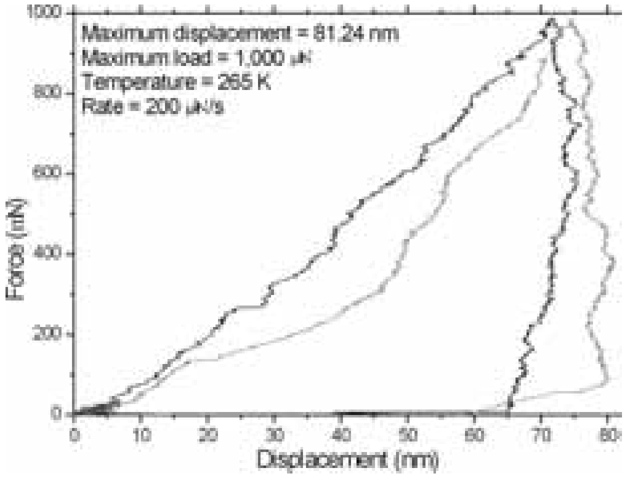

Figure 4: Loading and unloading pattern at 265 K for 1,000 μN and loading rate of 200 μN/s. Multiple curves indicate the experiments at different locations on the sample. The curves starting from the origin till the maximum point indicate the loading.

Slika 4: Značilnosti obremenitve in razbremenitve pri 265 K pri 1 000 μN in hitrosti obremenitve 200 μN/s. Krivulje pomenijo odtise na različnih mestih na preizkušancu. Krivulji z začetkom v koordinatnem središču pomenita obremenitev.

displacement of the material under the indenter was large compared to the displacement at 373 K and 473 K, indicating the softening of the metal at higher temperature. Also, **Figure 3** exhibits a similar kind of unloading curve. The exact data on the amount of softening will enable the proper selection of materials for different thermal loading conditions. The softening effects observed at higher temperatures below the recrystallization temperature are useful for the proper selection of materials for variable thermal loading conditions as the softening effects increase the plasticity. As global, the deformation is understood, which involves the entire body, in contrast to poking and squeezing, which involve relatively small regions of the deformable object. It was also noted that the elastic recovery was smaller at higher temperatures.

Figure 4 was obtained to study the initial burst and the periodic bursts clearly visible at low loading rate and load when the initial burst was clearly observed, while, it was not visible at greater load. During loading and unloading at low temperature, the curves exhibit large vibration. The unloading curve was not linear and was similar to that for copper. The material recovery rate was less and rebounding sharp at the end of the recovery process by low temperatures and loading conditions. The curve shoved, also, that the elastic recovery was minimal as the displacement smaller. Also the curves at 265 K showed a brittle behavior, because the material withstanded higher load before first yield. As evident from the curves, the initial burst occurred very early, while, the metal accommodated in itself without significant pile up. Displacement bursts were due to nucleation of dislocations. At low temperarute, the material exhibited brittle behavior and at 265 K, the recovery rate was less and, at the end of the recovery process, the rebounding was sharp.

Table 1: Data for a load of 1000 μN and loading rate of 200 μN/s
Tabela 1: Podatki za obremenitev 1 000 μN in hitrost obremenitve 200 μN/s

Temp/ Data	h_{max}/nm	$h_{plastic}$ /nm	H/GPa	E/GPa
265 K	76.16	72.31	4.02	576.64
	81.24	77.83	3.55	6297.58
	52.48	49.38	7.53	9190.52

Table 2: Data for a load of 10,000 μN and loading rate of 2,000 μN/s.
Tabela 2: Podatki za obremenitev 10 000 μN in hitrost obremenitve 2 000 μN/s

Temp/ Data	h_{max} / nm	$h_{plastic}$ / nm	H/GPa	E/GPa
373 K	190.62	154.24	12.22	202.16
	196.79	163.75	10.9	209.17
473 K	201.9	163.98	10.91	182.65
	201.29	149.86	12.69	144.8
623 K	249.02	191.92	8.28	105.82
	231.93	172.25	10.01	111.22
	211.49	141.71	14.09	112.84
	212.03	148.37	13.01	118.86
	231.93	172.25	10.01	111.22

In **Tables 1 and 2**, it can be noted that the difference between $h_{plastic}$ and h_{max} varied with the tests temperature showing that the plasticity range increased with the temperature and the displacement. Also, the recovery range increased comparatively and it was proportional to the displacement. The overall plastic region increased as the displacement was large at higher temperatures. In other words, the elastic recovery was lower at higher temperature, while the displacement was greater. As shown in **Tables 1 and 2**, the loading rate and load also affected the hardness value as seen in **Tables 1 and 2**. The elastic modulus (E) was high by great difference $h_{plastic}$ and h_{max} and h_{eff} was high, also. The elastic modulus decreases with the temperature, while, the penetration depth increases.

The difference $h_{plastic}$ and h_{max} was less at smaller loading rate and indicated that the loading rate affects the range of plasticity. Also, it was noted that the elastic modulus (E) was high when the difference between plastic displacement ($h_{plastic}$) and maximum displacement (h_{max}) was large. The hardness (H) value does not depend on the difference $h_{plastic}$ and h_{max}. The maximum displacements (h_{max}) in **Tables 1 and 2** shows the softening effects at different temperature and can be used for the proper materials selection for different thermal loading conditions.

4 CONCLUSIONS

The tests on tungsten showed that different events occur in the metal during the penetration of the indenter and the unloading at different temperature. Clear bursts showed the nucleation of dislocations and pile up was observed. It was found that the load displacement was not linear during unloading and that at higher peak load the indentation contact in tungsten was not purely elastic. At high temperature, the material showed higher dislocation mobility and a greater plastic deformation. It was concluded that the stored dislocations and the thermal recovery were responsible for maintaining a high mobile dislocation density that was temperature-dependent and sustained a large uniform elongation. At higher temperature the deformation was global, unlike the multi-bursts (brittleness) due to local dislocation motion at low temperatures. Changes of force and rate indicate to strain bursts due to the breakout of dislocations. The maximual displacements (h_{max}) at higher temperatures indicate that significant softening may occur at sufficiently high stress. Tungsten (111) planes showed lower hardness values than (110) planes as also evident from the results from investigations on aluminum.

The noise in the loading and unloading curves at low temperature can be improved by cooling to a lower level and then heating to the required temperature. The small variations in the penetration depths in the *p-h* curves for same testing conditions must be avoided in case of single crystal. Our experiments clearly showed the onset of the first strain burst, the onset of plastic deformation in connection with the periodic bursts, and the strain hardening/softening/ recovery effects. The elastic modulus was lower at higher temperaturesand the softening increased with the increase of indentation depth for the same loading conditions. The elastic recovery was smaller at higher temperatures. The pop-ins shown on *p-h* curves correspond to the formation of dislocations. The contact pressure (nanohardness) increased with decreasing indentation depth. We attribute the temperature effect to the increased dislocation mobility and the reduced dislocation density. Dislocation pile up around the indentation was clearly visible.

The onset of plastic deformation was identified from the periodic bursts. The difference in pile up was observed for different temperature and the new phenomenon of material under the indenter bouncing back at the end of unloading was established, also. Because of dynamic softening the penetration depth and plastic deformation were greater at higher temperature. The indentation rate affected the modulus and the hardness.

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