

# Strain Burst and Strain Rate Sensitivity of Iridium Micropillars

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**Abstract:**  $\langle 110 \rangle$ -oriented single-crystalline iridium micropillars with diameters ranging from 1000 nm to 7400 nm were fabricated by focused-ion-beam (FIB), and uniaxial compression tests for iridium micropillars were performed using a nanomechanical instrument. The results reveal that the yield strength of micropillars follows a power law depending on diameter, as reported in other face-centered cubic (fcc) metals. Unlike the mechanical behavior of bulk iridium when uniaxial compression is performed, the micro- and nano-scale flow stress increases with decreasing the micropillar diameter, which is attributed to the size-dependent effect. Moreover, the plastic deformation shows a periodical jerky and strain bursts, and the rate of strain bursts and the sensitivity of strain rate for iridium micropillars under uniaxial compression were mainly investigated. The experiments also indicate that the displacement jump increases with increasing the duration time and micropillar diameter. In addition, the strain rate sensitivity (SRS) index  $m$  increases with decreasing the diameter of iridium micropillar.

**Key words:** iridium micropillars; FIB; nano-indentation; compression test; size effect; strain burst; SRS

It is very important to understand the complex mechanical behavior of materials at micro- and nanoscale, as more and more miniaturized devices are used in modern technological applications<sup>[1,2]</sup>. It is found that the flow stress of micro- and nano-scale depends on micropillar diameter, which is the size effect<sup>[3-12]</sup>, whereas it is a size-independent property for the bulk<sup>[13-15]</sup>. In 2004, Uchic et al<sup>[3]</sup> conducted the micro-compression test for Ni micropillars fabricated FIB, and the phenomenon “smaller is stronger” was observed. The compression tests for micropillars have been performed by many researchers since then<sup>[13-20]</sup>. Many experiments show that the flow stress depends on the micropillar diameter in a power-law:  $\sigma \propto d^n$ , where  $\sigma$  is the flow stress,  $d$  is the micropillar diameter, and  $n$  is the exponent ranging from 0.6 to 1.0 for face-centered cubic (fcc) metals<sup>[1,2,21]</sup>. Some theories have been put forward to explain size effect at micro- and nano-scale<sup>[22-29]</sup>, and the dislocation starvation model is generally accepted<sup>[6,30]</sup>.

Recently, uniaxial compression experiments were carried out using nano-indenter with a flat punch for Ni, Al, Au and Cu micropillars fabricated by focused-ion-beam (FIB)<sup>[3-15]</sup>. On the one hand, the method can supply the information of stress-strain curves during the compression tests; on the other hand, it reduces the possibility of the presence of large strain gradients<sup>[22,25]</sup>. There are still some differences between micro-scale and bulk samples according to numerous compression tests: (1) smaller is stronger, which means that the yield strength increases drastically with the reduction of the diameter of micropillar<sup>[4]</sup>; (2) the stress-strain curves during plastic deformation show intermittently strain bursts<sup>[6]</sup>.

Most previous studies have mainly focused on the size-dependent effect of micro-scale micropillars, but few have focused on the stochastic nature of plasticity and the strain rate sensitivity, and the studies on compression of single-crystalline iridium (Ir) micropillars are particularly less. Ir has a high melting point (2443 °C), which is one of the attractive

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metals in high-temperature applications due to its excellent corrosion resistance and good mechanical properties above 1600 °C. For example, iridium crystals have been widely used in nuclear fuel container, coatings of rocket thrusters, automotive spark plugs and crucibles for single-crystal growth<sup>[31]</sup>. In this work, the velocities of strain bursts and strain rate sensitivity were investigated.

## 1 Experiment

The <110>-oriented single-crystalline iridium sample with dimensions of 10 mm×10 mm×0.5 mm was supplied by MaTecK-Material-Technologie&Kristalle GmbH, which was non-residual stress and defect-free. The single-crystalline iridium micropillars with diameters of 1, 2, 3, 5, and 7.4 μm were fabricated using focused-ion-beam (FIB) and subsequently compressed in the Hysitron Ti 950 nano-indenter at room temperature. The aspect ratio (height/diameter) of all micropillars is 2.5, as shown in Fig.1. The micropillars (Fig.1a) produced by FIB were compressed using a diamond micro-indenter and the strain rate ranged from 10<sup>-4</sup> s<sup>-1</sup> to 10<sup>-2</sup> s<sup>-1</sup>.

## 2 Results and Discussion

### 2.1 Velocities of strain bursts

Fig.2 shows the engineering stress-strain curve of a micropillar with 3000 nm in diameter, and there are many strain bursts on the curve. The inset highlights a zoomed-in data portion of three displacement jumps, which indicates that the decrease of compressive stress partly occurs during the forward surge. Then the stress rapidly drops over dozens of MPa at the end of the displacement jump, and the subsequent unloading is linearly elastic.

Fig.3a shows the relationship between the axial displacement and the temporal duration, which includes more than 100 strain bursts. The displacement jump increases with the duration time, and the larger the micropillar diameter, the higher the axial displacement. Here an average axial displacement rate was defined to describe the deformation behavior for all micropillars,  $\Delta h/\Delta t$ <sup>[32]</sup>, and the results are shown in Fig.3b. Fig.3b reveals that the clusters strongly correlate with the micropillar size, that is, the axial displacement rate increases

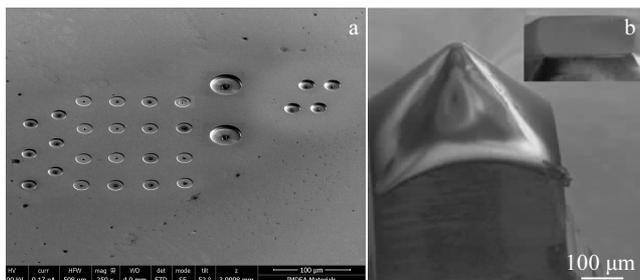


Fig.1 <110>-oriented single-crystalline iridium micropillars with different sizes (a) and SEM image of diamond indenter (b)

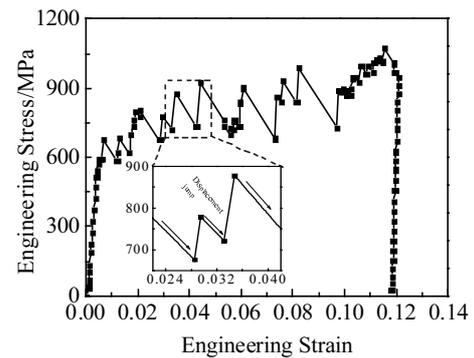


Fig.2 Engineering stress-strain curve of iridium micropillars with 3000 nm in diameter (inset displaying three zoomed-in displacement jumps)

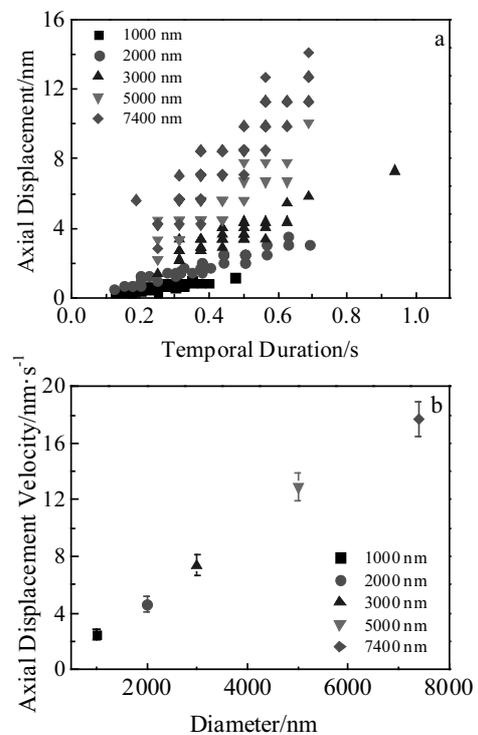


Fig.3 Relationship between axial displacement and corresponding temporal duration (a) and displacement jump velocity vs micropillar diameters (b)

with increasing the micropillar diameter.

### 2.2 Effects of strain rate and sample size on the strength of iridium micropillars

Fig.4a shows the dependence of yield strength with strain rate for <110>-oriented single-crystalline iridium micropillars with different diameters. It indicates that the yield strength increases with increasing the strain rate for micropillars with diameters from 1000 nm to 2000 nm, but it is opposite for micropillars with a diameter of 3000 nm, and the yield strength get much

higher when the diameter of micropillar is smaller at the same strain rate, that is “size-dependent effect”. The relationship between yield strength and the diameter of iridium micropillar can be described by a formula as follows<sup>[33]</sup>:

$$\sigma_{YS} = Ad^{-n} \tag{1}$$

where  $\sigma_{YS}$  is the yield strength,  $A$  is a constant related to materials,  $d$  is the diameter of the micropillar, and  $n$  is the exponent which is generally 0.6~1.0 for most fcc metals. A larger  $n$  indicates a stronger size-dependent effect. Fig.4b shows the yield strength as a function of micropillar diameter, and it can be found that  $n$  is 1.15, demonstrating that the size-dependent effect of single-crystalline iridium micropillars is fairly strong.

The yield strength is shown in Fig.5 for micropillars with different diameters, and the yield strength increases with decreasing the diameter at the same strain rate, which is also attributed to the “smaller is stronger” effect. For comparison, two strain rates of  $1 \times 10^{-2}$  and  $1 \times 10^{-3} \text{ s}^{-1}$  were set. It reveals that the slope of line at  $1 \times 10^{-2} \text{ s}^{-1}$  is larger than at  $1 \times 10^{-3} \text{ s}^{-1}$  when the diameters are less than 3000 nm, that is to say, the size-dependent effect is stronger at  $1 \times 10^{-2} \text{ s}^{-1}$ . It is prominent that yield strength increases obviously with decreasing the diameter of micropillar when the micropillar is less than 3000 nm, and the yield strength increases slowly when the micropillar is larger than 3000 nm at the strain rate  $1 \times 10^{-3} \text{ s}^{-1}$ .

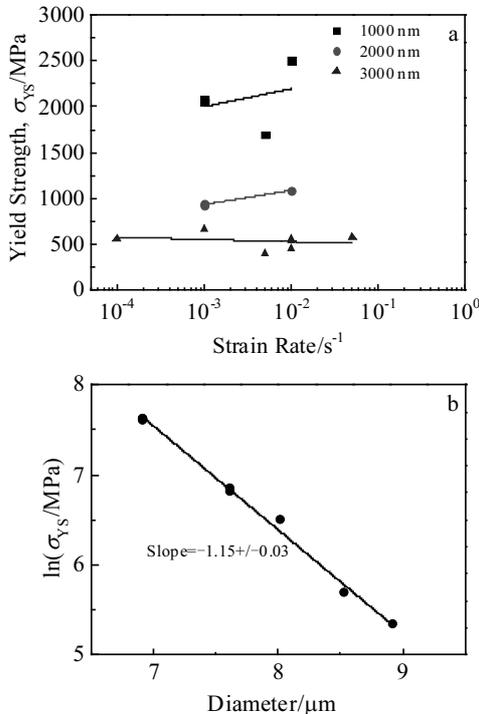


Fig.4 Yield strength of <110>-oriented single-crystalline iridium micropillars as a function of strain rate (a) and relationship between yield strength and diameters at the strain rate of  $1 \times 10^{-3} \text{ s}^{-1}$  (b)

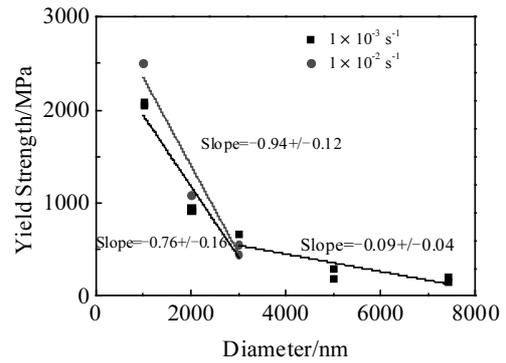


Fig.5 Relationship between yield strength of <110>-oriented single-crystalline iridium micropillars and micropillar diameters

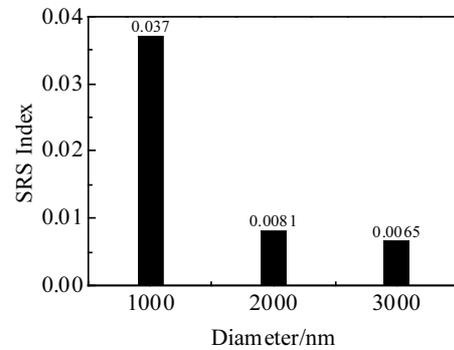


Fig.6 Relationship between SRS index ( $m$ ) and diameters for <110>- oriented single-crystalline iridium micropillars

The strain rate sensitivity can be described by an equation as follows:

$$\sigma = \sigma_0 \dot{\epsilon}^m \tag{2}$$

where  $\sigma$  is the flow stress,  $\sigma_0$  is a constant,  $\dot{\epsilon}$  is the strain rate, and  $m$  is the strain rate sensitivity index. Fig.6 shows the relationship between SRS index ( $m$ ) and the micropillar diameters for <110>-oriented single-crystalline iridium micropillars. It reveals that the SRS index increases with decreasing the diameter of micropillars.

### 3 Conclusions

1) The displacement jump rate and strain rate sensitivity are investigated based on the strain bursts in iridium micropillar. Experiments reveal that the displacement jump increases with the duration time, and the larger the micropillar diameter, the higher the axial displacement. Moreover, the axial displacement rate increases with increasing the diameter, i.e. there is a correlation between strain burst and stress.

2) The <110>-oriented single-crystalline iridium micropillars show strong size-dependent effect, and the size-dependent effect is stronger at  $1 \times 10^{-2} \text{ s}^{-1}$  than at  $1 \times 10^{-3} \text{ s}^{-1}$  when the diameter is less than 3000 nm.

3) The value of strain rate sensitivity index may increase with decreasing the diameter of micropillars.

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## 铌微柱的应变突变和应变速率敏感性

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**摘要:** 以聚焦离子束 (FIB) 制备了直径在 1000 nm 至 7400 nm 范围内的<110>取向铌单晶柱, 并在纳米机械仪器 Sementort 中进行了单轴压缩试验。结果表明, 单晶柱的屈服强度与直径有显著关系, 而与 FIB 制备无关。单晶柱的微压缩实验与整体铌在单轴压缩试验中的力学行为不同, 微纳米尺度的流动应力随着柱直径的减小而增加, 即尺寸效应。此外, 塑性变形过程有明显的突变和大应变, 重点研究了单轴压缩下铌单晶柱的应变突变速度和应变敏感性问题。结果表明, 压缩位移量随着持续时间和立柱直径的增加而增加, 同时指出了应变敏感性 (SRS) 指数  $m$  随着柱直径的减小而增加。

**关键词:** 铌微柱; FIB; 纳米压痕; 压缩试验; 尺寸效应; 应变突变; SRS

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