Mechanical Response of Single Crystal (0001) ZnO and Micropillar under Nanoindentation and Microcompression

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Abstract

Zinc oxide has become a potential material due to its wide band gap (3.3 eV), strong chemical and thermal stability. It can be the core material in light emitting diode, high power laser devices or piezoelectricity generator. Moreover, the high surface/volume ratio can enhance light extract efficiency and reduce the residual stress. In this study, we focus on the mechanical response of single crystal wurtzite ZnO micro-pillar, fabricated by focus ion beam (FIB) from bulk ZnO. The micro-pillars were measured in the uniaxial-axis compression in micro-scale, preformed by the nanoindentation system with a flat punch tip. The resulting stress-strain curves exhibit a burst at ~7% engineering strain. The Young's modulus and compression yield stress of the ZnO micro-pillars are 122 GPa and 4.4 GPa, respectively. Comparing to the modulus (140 ± 26 GPa) and yield stress (4.2 ± 1 GPa) measured and transformed by the data from nanoindentation, the values are slightly lower due to the small volume constrain effect. The cross section transmission electron microscopy observation reveals the massive dislocation slip trace on pyramidal plane {10-11}. No cracks or phase transformation is observed in this study. The relationship between the plastic deformation, residual stress and optoelectronic properties, especially in micro scale, are also examined via cathodoluminescence, Raman spectrum analysis and transmission electron microscopy observation. It is found that the contact loading during processing or packaging can significantly degrade the performance of these devices.

Results and discussion

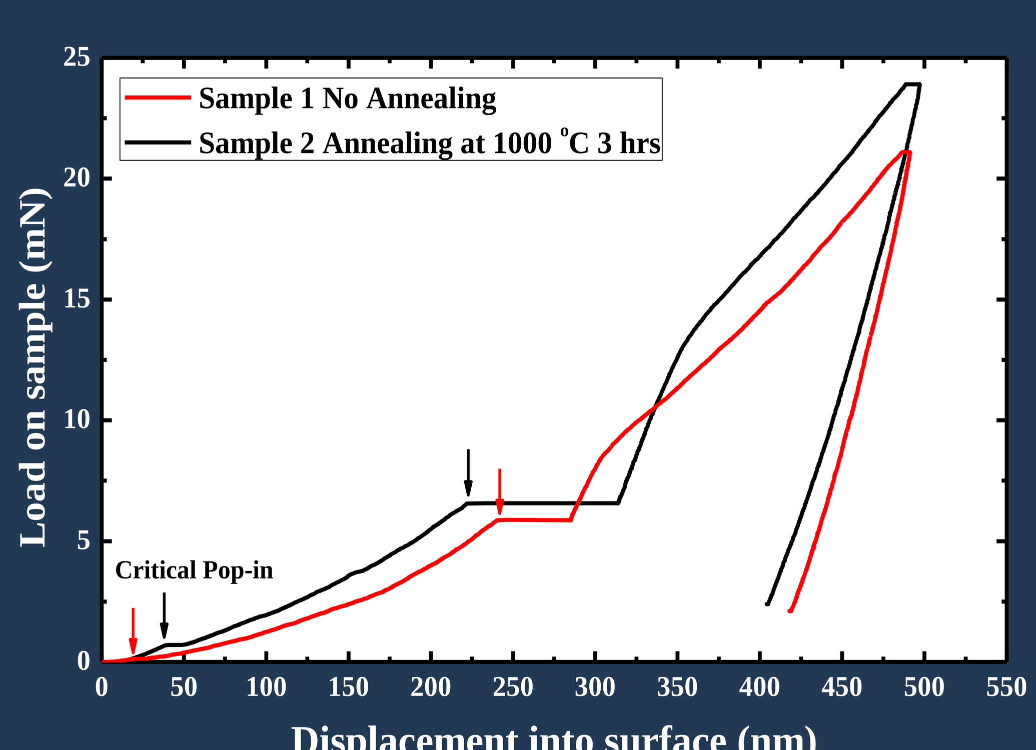

Figure 1 The nanoindentation load-displacement curves of bulk single crystal ZnO with CSM mode at strain rate $1x10^{-3}$ s^{-1}.

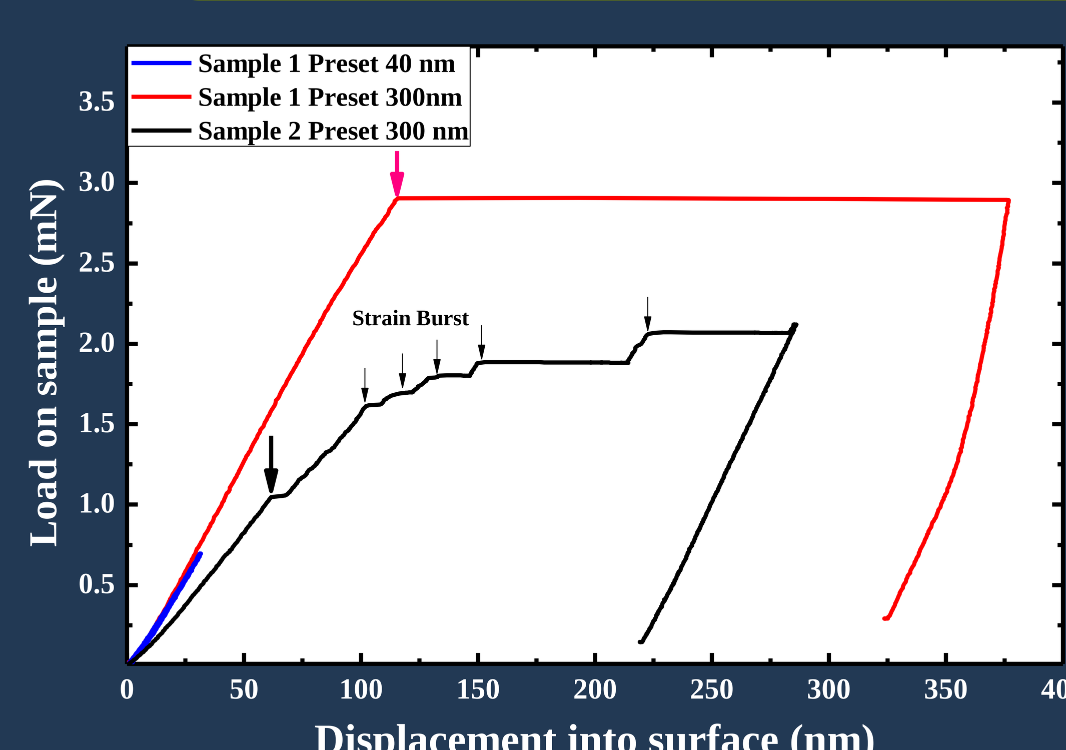

Figure 2 The microcompression load-displacement curves of ZnO micro-pillars at strain rate $1x10^{-3}$ s^{-1}.

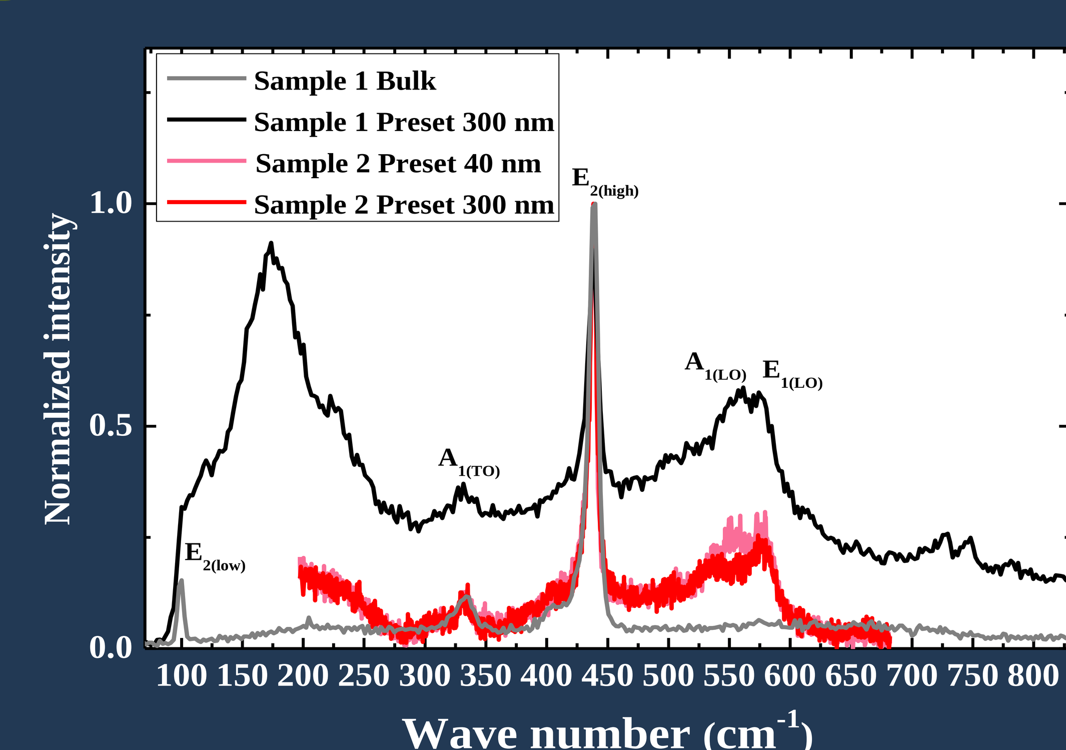

Figure 3 The Raman spectrum of bulk ZnO/micro-pillars.

Table 1 The mechanical properties of bulk and micro-pillar single crystal ZnO .

	Sample 1		Sample 2	
Annealing temperature	None		1000 °C	
Type	Bulk	Pillar	Bulk	Pillar
Pop-in (mN)	0.11	4.38	0.7	1.04
E (GPa)	140 ± 26	122	124 ± 3	121
H (GPa)	4.2 ± 1	-	4.6 ± 0.1	-
Yield Stress (GPa)	4.2	4.4	-	-

Table 2 The peak positions and full weight at half maximum of sample one Raman spectrum.

(cm^{-1})	Bulk	As-milled micro-pillar	Preset 40 nm micro-pillar	Preset 300 nm micro-pillar
$A_{1(TO)}$	331.6	332.0	329.2	331.4
FWHM	13.9	19.8	17.1	12.5
$E_{2(high)}$	438.6	438.0	438.2	438.2
FWHM	4.9	6.0	6.0	6.4
$A_{1(LO)}$	-	549.0	551.1	538.9
FWHM	-	77.3	70.5	88.9
$E_{1(LO)}$	-	576.8	578.9	576.7
FWHM	-	18.2	19.2	21.7

Equation

$$\sigma_{ys} = \tau_{max} / (\cos\theta \cos\varphi)_{max}$$

$$\tau_{max} = 0.12\left(\frac{P_{crit}E_s^2}{R^2}\right)^{\frac{1}{3}}$$

$$\frac{1}{E_R} = \left(\frac{(1-\nu_{in}^2)}{E_{in}} + \frac{(1-\nu_s^2)}{E_s}\right)$$

$$E = \left(\frac{P}{\Delta h}\right)\left(\frac{\ln(1+\frac{h_0 2\theta}{d_0})}{\pi d_0 2\theta}\right)$$

σ_{ys}: the normal yield stress
($\cos\theta \cos\varphi$): Shimid factor
P_{crit}: the critical pop-in load in nanoindentation
R: tip radius
E_s: the actual modulus of the material
E_R: the measured modulus by nanoindentation
E_{in}: the diamond tip modulus (1141 GPa)
ν_{in}: Poisson ratio of diamond indenter tip
ν_s: Poisson ratio of c-ZnO
($P/\Delta h$): The slope of pillar unloading curve
h_0: Pillar height
d_0: Diameter of micro-pillar

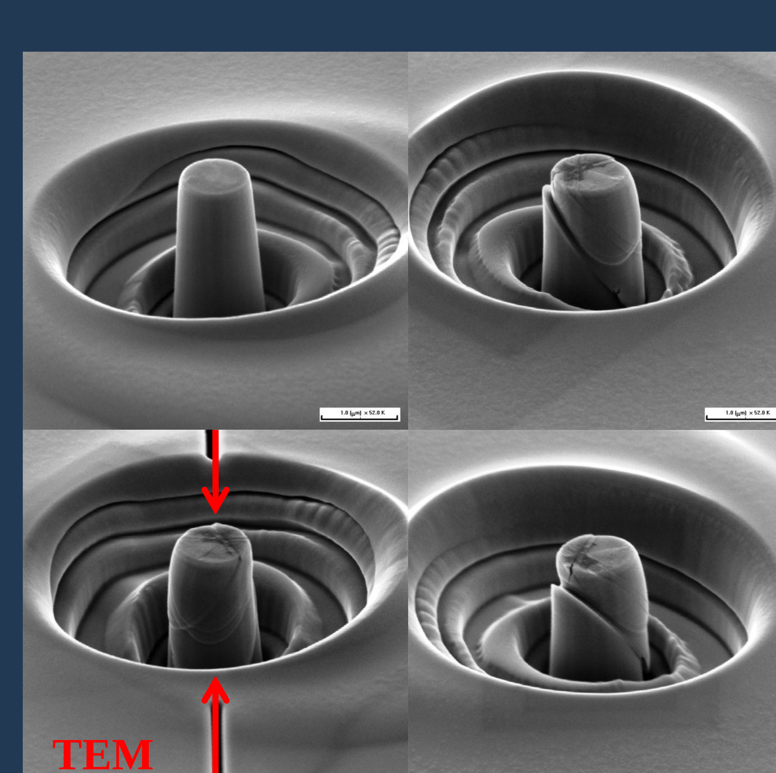

Figure 3 SEM micrographs showing the morphology of 1 μm micro-pillar at strain rate $1x10^{-3}$ s^{-1}.

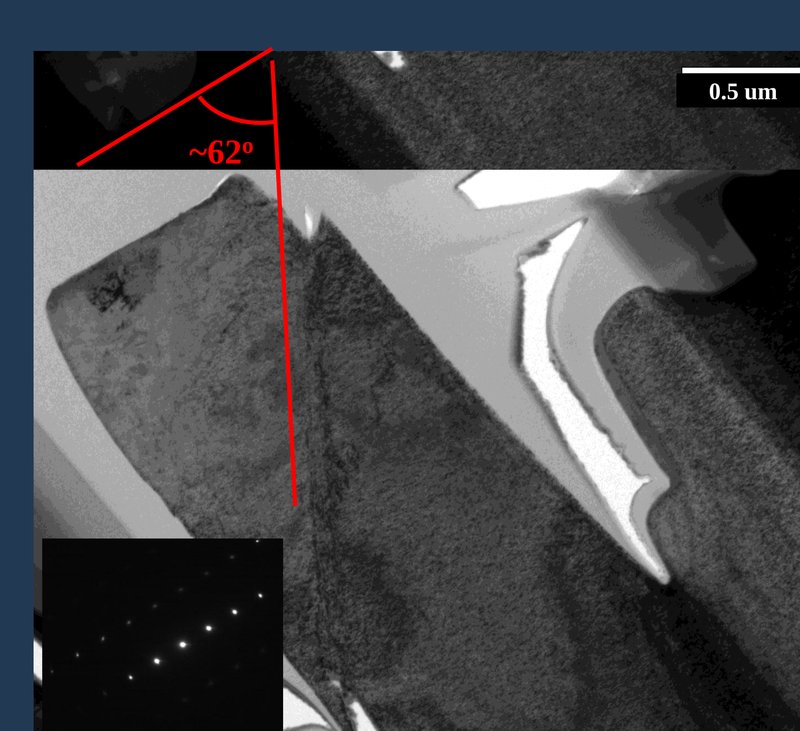

Figure 4 TEM results of the micro-pillar with strain rate $1x10^{-3}$ s^{-1}. Zone axis of diffraction pattern is [1 0 0]. Bright field image shows the slip band oriented at ~62° to the basal plane (0001).

Conclusions

1. **The micro-scaled compressive elastic modulus of the (0001) ZnO (122 GPa) , it can be compared with the modulus and hardness measured by nanoindentation (ZnO ~140 ± 26 GPa).**
2. **The micro-scaled compressive yield stress of the (0001) ZnO micropillars (~4.4 GPa) can be compared with estimating yield stress (~4.2 GPa) measured by nanoindentation.**
3. **The Raman spectrum measurements and TEM observations reveal that no residual stress induced in the ZnO after microcompression. Suggesting the residual stress can be released due to the high surface ratio of micro-pillar.**