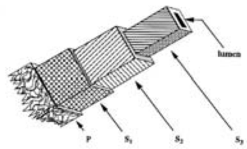
The micro mechanical properties of the wood cell wall



Xinan Zhang
M.S. Candidate

Tennessee Forest Products Center

Introduction

- Nowadays, with the fast development of nanotechnology, more attention has been paid to micro and sub-micro scale area.
- The micro mechanical properties and sub-micro mechanical properties of wood cell wall are paramount because of its potential applications in wood based nano-composites in future.
- As far as wood science aspect is concerned, the mechanical contribution of process in the cell wall is not fully understood .

Tennessee Forest Products Center

Introduction

Nanoindentation testing

- Wimmer was the first to prove the capability of nanoindentation to determine mechanical properties of cell walls in wood.

- It allows an indenter to penetrate the sample of a material, at the same time, the penetration depth and load can be recorded. As a result, the stiffness and hardness of the indented location can be calculated.

Tennessee Forest Products Center

Introduction

$$H = \frac{P_{\max}}{A}$$

$$E_r = \frac{dP}{dh}\frac{1}{2}\frac{\sqrt{\pi}}{\sqrt{A}}$$

$$E_s = (1-v^2{}_s)\left(\frac{1}{E_r} - \frac{1-v_i^2}{E_i}\right)^{-1}$$

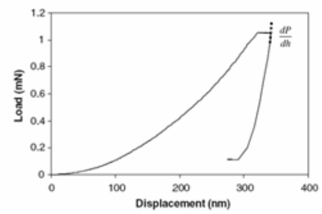


Fig. 1. The load–displacement curve during indentation. *Note*: The initial slope of the unloading curve was used to calculate stiffness (E_u).

Strain gradient effects are unavoidable

Introduction

Micro-pillar compression test

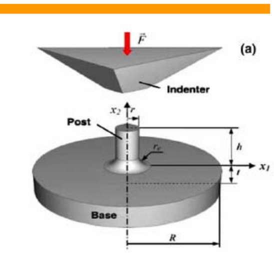
- Through this effective experiment, the yield stress and modulus of the material can be tested.
- Strain gradients can be avoided.

Introduction

Micro-pillar model

Zhang H, Schuster BE, Wei Q, Ramesh KT.
The design of accurate micro-compression experiments. Scripta Materialia 2006;54:181-186

- Fillet radius/post radius ratios of 0.2–0.5 are recommended
- The post aspect ratio (defined as height/diameter ratio) of 2–3 is reasonable
- Taper of the pillar should be minimized during micro-pillar fabrication, it would results in overestimated elastic modulus, spurious strain hardening and increased apparent yield



Introduction

Michael D. Uchic, Dennis M. Dimiduk. A methodology to investigate size scale effects in crystalline plasticity using uniaxial compression testing. Materials science and engineering A 400-401 (2005) 268-278

- Invested a methodology for performing uniaxial compression tests on samples having micron-size dimensions.
- Sample fabrication is accomplished using focused ion beam milling to create cylindrical samples of uniform cross-section that remain attached to the bulk substrate at one end.
- Once fabricated, samples are tested in uniaxial compression using a nanoindentation device outfitted with a flat tip, and a stress–strain curve is obtained.
- a single-crystal Ni superalloy, D<5 um, size effect appeared.

Tennessee Forest Products Center

Introduction

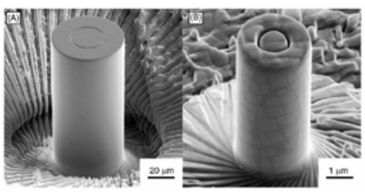
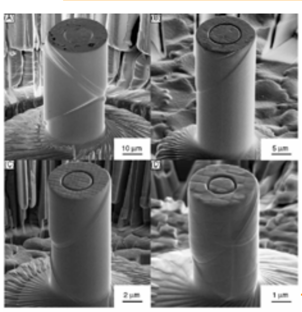


Fig. 3. (A) A 43 µm diameter micro-compression sample machined into a $Ni_3(Al, Hf)$ single crystal, where the gage length of this sample is approximately 90 µm. The circular pattern on the top surface of the sample is a fiducial mark used as part of an automated machining program for the FIB. (B) A 2.3 µm diameter micro-compression sample machined into a single-crystal Ni superalloy (UM-F19). The gage length of this micro-sample is approximately 4.6 µm. One can clearly observe the Ni_3Al precipitates on the surface of the micro-sample.

Tennessee Forest Products Center

Introduction



Tennessee Forest Products Center

Introduction

C. A. VOLKERT and E. T. LILLEODDEN. Size effects in the deformation of sub-micron Au columns. Philosophical Magazine 2006 (86) 5567-5579

- Single crystal Au columns ranging in diameter from 180nm to 8 um.
- The compressive yield stress scales roughly as the inverse square root of the column diameter.
- Stresses as large as 1 GPa are reached.

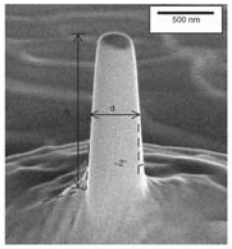


Figure 1. SEM image taken at 52° tilt of a column in Grain B with 440 nm diameter and 1.66 μm height. The sidewall taper, typically 2°, is indicated.

Tennessee Forest Products Center

Introduction

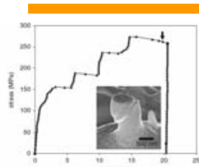


Figure 2. Engineering stress-strain curve for a 710 nm diameter column (Grain I 1.75 μm). The arrow indicates the point at which the 15 second hold begins. The ima deformed column in the inset shows deformation by single slip.

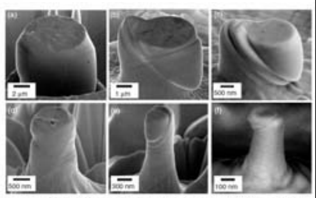


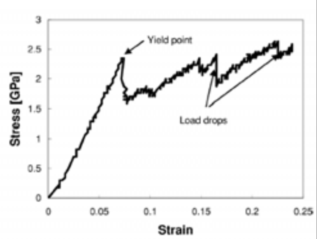
Figure 4. Deformed columns with initial diameters of (a) 8.14 μm, (b) 4.06 μm, (c) 2.00 μm, (d) 1.05 μm, (e) 530 nm and (f) 210 nm. The columns shown in (b) and (c) are from Grain B, all others are from Grain A.

Tennessee Forest Products Center

Introduction

Johann Michler, Kilian Wasmer, Stephan Meier, and Fredrik Östlunda. Plastic deformation of gallium arsenidemicropillars under uniaxial compression at room temperature. Applied physics letters 2007 (90) 043123

- Compressive strength of gallium arsenide (**GaAs**) pillars with a diameter of 1 um by uniaxial compression tests.
- The micropillars were found to exhibit ductile plasticity comparable to macroscopic tests.
- The yield stress was 1.8±0.4 GPa

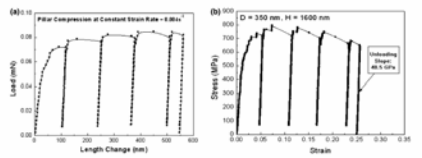


Tennessee Forest Products Center

Introduction

Julia R. Greer, Warren C. Oliver, William D. Nix. Size dependence of mechanical properties of gold at the micron scale in the absence of strain gradients. Acta materialia 2005(53) 1821-1830

- Uniaxial compression experiments on gold at the sub-micron scale, without stress/strain gradients.
- A significant flow stress increase, up to several GPa. These high strengths appear to be controlled by dislocation starvation, unique to small crystals.



Products Center

Introduction

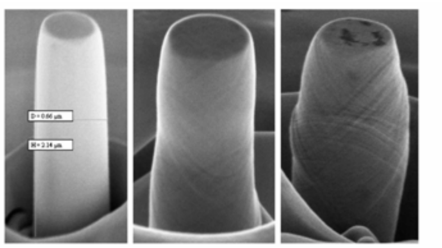


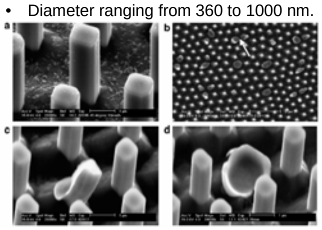
Fig. 7. SEM images of undeformed, deformed, and severely deformed pillars. Slip lines are clearly present in the deformed states.

Tennessee Forest Products Center

Introduction

H. Bei, S. Shim, E.P. George, M.K. Miller, E.G. Herbertc and G.M. Pharr. Compressive strengths of molybdenum alloy micro-pillars prepared using a new technique. Scripta materialia 2007 (57) 397-400

- chemically etching
- Single-crystal micro-pillars of a molybdenum alloy
- Diameter ranging from 360 to 1000 nm.

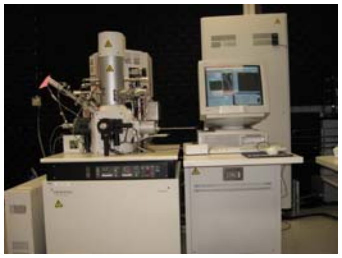


nessee Forest Products Center

Introduction

Focused ion beam (FIB)

- FIB is a useful technology using gallium liquid metal ion source (LMIS) for sample preparation.



Tennessee Forest Products Center

Introduction

FIB

- Started in 1974
- Liquid source: Cesium, ***<u>gallium</u>***, mercury and so on
- Long lifetime (over 500 h) and high stability variation (less than ±1%)
- The typical accelerating voltage is between 5 and 50 keV while the primary ion penetration depth is ~20 nm (25 keV Ga^+)

Tennessee Forest Products Center

Introduction

FIB Application

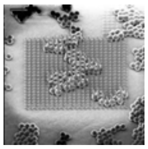
- FIB technology was first used to improve semiconductor devices for maskless implantation techniques. Then the FIB became a powerful tool for transmission electron microscopy (TEM) sample preparation.
- Although the most popular application area of FIB is semiconductor field, FIB is a versatile tool, it can mill most kinds of materials. It can be used in solid state devices for lithography, etching, deposition and doping.

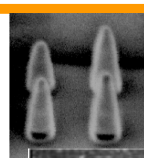
Tennessee Forest Products Center

Introduction

lithography

etching





deposition

doping

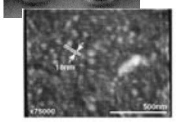


Fig. 1-1 A secondary electron image for evaluating a beam diameter in the modified focused ion beam (FIB) optics.

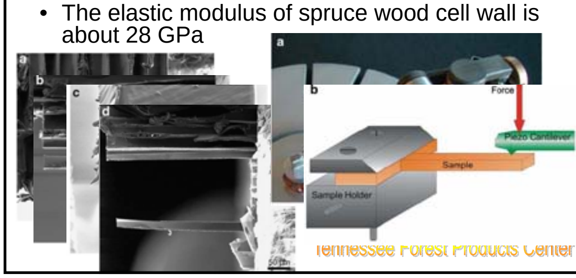
Tennessee Forest Products Center

Introduction

Application in wood science

Steffen Orso Ulrike G. K. Wegst Eduard Arzt

- The elastic modulus of spruce wood cell wall is about 28 GPa



Tennessee Forest Products Center

Introduction

Damage in the sample surface

It was reported that at 30 keV, the top 25 nm of a silicon substrate will be amorphized.

- Minimize the milling time
- Lowering the gallium ion energy in the final step of milling
- A plasma cleaner.

Low energy Ar ions were induced by immersing the sample in the plasma source, while the ratio of Ar:O_2 is 3:1.

Yet some researchers said that the results obtained from a wide range of materials show that important results can be received without damage layer removal.

Tennessee Forest Products Center

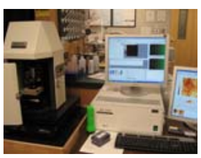
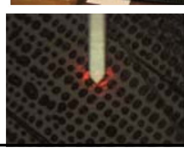
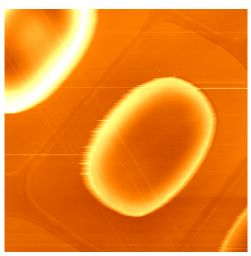
Objectives

- The objective of this work is to investigate the micro mechanical properties of wood cell wall through a micro-pillar uniaxial compression test.
- The sample will be prepared by a focused ion beam (FIB) system.
- Yield stress and modulus of wood cell wall will be invested, and this engineering modulus can be compared with the result got by nanoindentation test.

Tennessee Forest Products Center

Methods

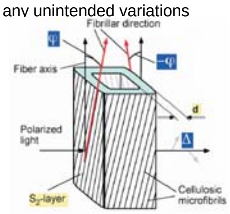
- AFM non-contact mode for surface scan

Tennessee Forest Products Center

Methods

Materials
- Softwood --loblolly pine
- Hardwood -- teak
- In the same growth ring to minimize any unintended variations



Tennessee Forest Products Center

Methods

Equipment
- Microtoming (glass knife and diamond knife)
- Optical microscopy
- Atomic force microscopy (AFM)
- Scanning electron microscopy (SEM)
- Focused ion beam (FIB)
- Micro-compression system
- Nanoindentation

Methods

Initial sample preparation
- Late wood part of the sample will be chosen and cut into 3mm×3mm×2mm.
- Embede in Spur resin, which is commonly used as an embedding medium for electron microscopy of biological samples.
- Microtoming—glass knife and diamond knife

Name	ERL-4221	DER-736	NSA	DAME
proportion	5	3	13	0.2
weight	2.5g	1.5g	6.5g	0.1g

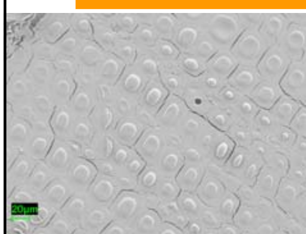
Methods

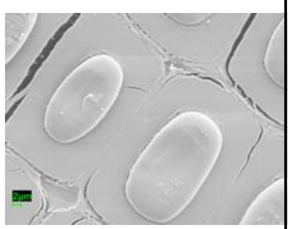
Methods

SEM
- beam energy 3KV
- working distance about 6mm
- aperture 60mm

Tennessee Forest Products Center

Methods





loblolly pine

Tennessee Forest Products Center

Methods

FIB milling
- Beam voltage 30 keV
- Bean current 7.617 nA
- Extraction voltage 6.33kV
- Emission current 2.4 kV

Species	Number	Micro-column diameter (um)	Height (um)	Fillet radiuses (um)
Loblolly pine	10	6	12	1.2
Teak	10	4	8	0.8

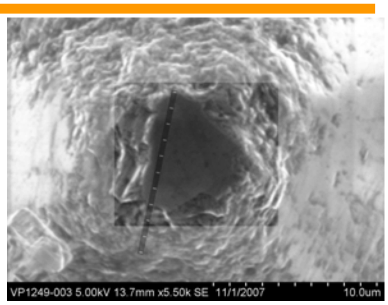
Tennessee Forest Products Center

Methods

Micro-compression system
- Optical Microscopy
- Moving stage
- Micro-compression tip

Methods

- Tip



Methods

- Micro-pillar uniaxial compression test

$$\varepsilon = \frac{\Delta l}{l_0} = \frac{l_0 - l}{l_0}$$

$$\sigma = \frac{P}{A} = \frac{P}{\frac{1}{4}\pi d^2}$$

$$E = \frac{\sigma}{\varepsilon}$$

Acknowledgements

- Dr. Siqun Wang (UTK)
- Dr. John Dunlap (UTK)
- Dr. Cheng Xing (UTK)
- Dorothy Coffey (ORNL)
- Rosa Trejo (ORNL)

Questions?

Thank you