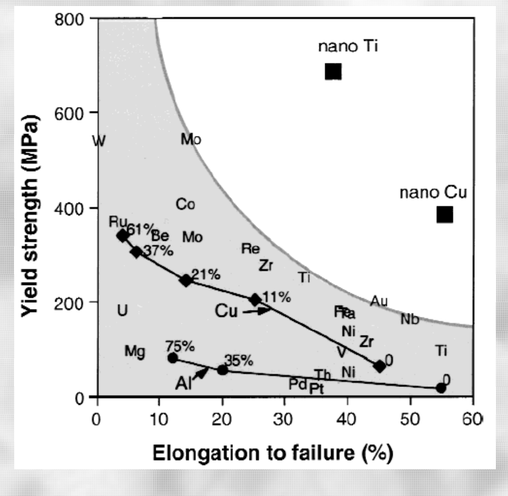
A new approach for in-situ TEM straining investigations of nanocrystalline Pd and PdAu using orientation mapping

Aaron Weis, Anna Castrup, Christian Kübel (INT)

Introduction

- Nanocrystalline metals show unique mechanical properties, especially high mechanical strength while maintaining good ductility in comparison to their coarse grained counterparts
- This potentially opens the road for new applications as structural and functional materials
- Deformation mechanisms of nanocrystalline metals are not understood



R.Z. Valiev at al.; J. Meter. Res. Vol. 17, No.1, Jan 2002

Recent investigations

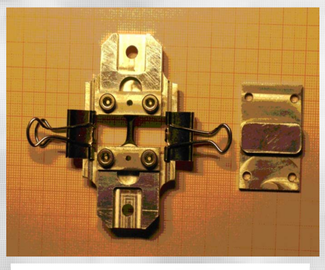
- Ex-situ tensile testing at different strains are compared using TEM cross-sections
- In-situ tensile testing using XRD, SEM and TEM

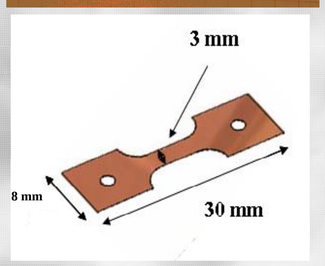
New approach

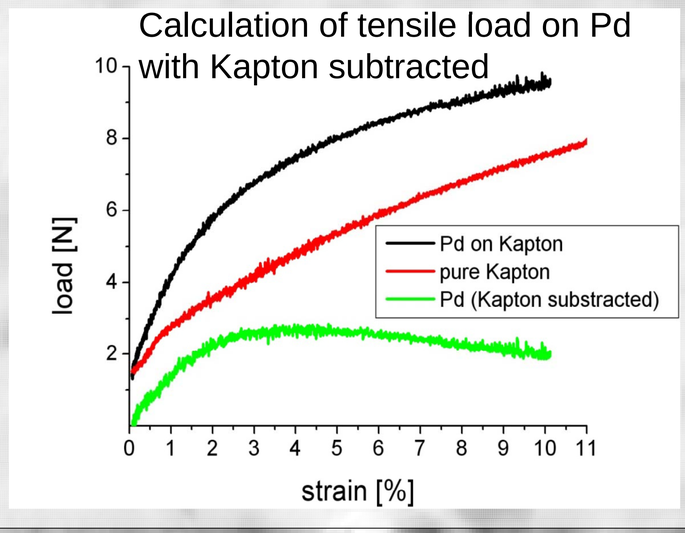
- Using sputtered nanocrytalline thin films on Polyimide for in-situ TEM tensile tests in combination with orientation mapping

Ex-situ tensile testing

- Special thin film clamping and sample shape
- Kapton is used to transfer strain from holder to the sample and to support the (in tension) brittle metal

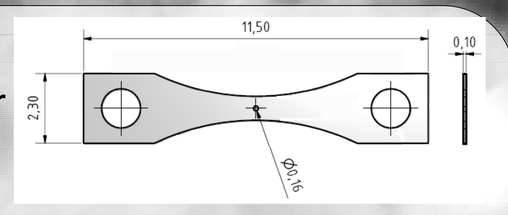






In-situ tensile testing

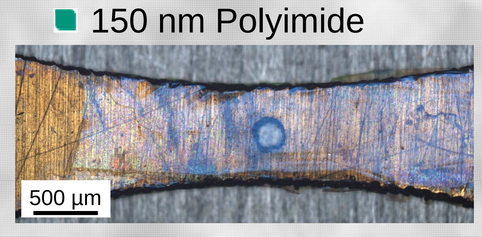
- Special TEM dog bone holder

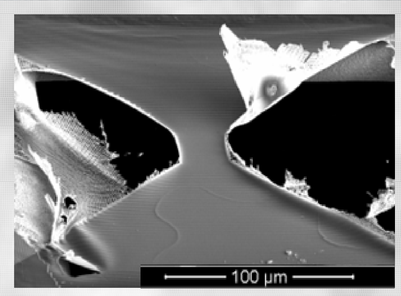


In-situ sample preparation

- Spin coating of liquid Polyimide onto substrate
- Transfer onto TEM dog bone holder
- Sputter metal onto Polyimide
- “Micro” dog bone preparation by FIB for defined TEM area

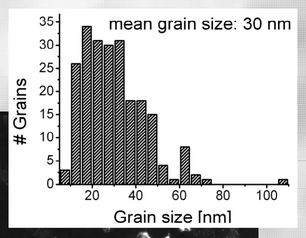
- 150 nm Polyimide

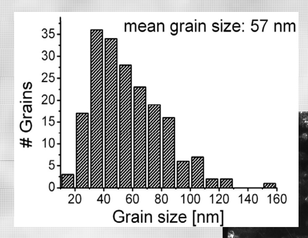




TEM investigations before and after tensile testing

- plane view dark field TEM analysis of 50 x 10 nm layers of sputtered Pd thinned to electron transparency
  - Before
  - After 15% tensile deformation





- HRTEM of Pd 110

In-situ tensile testing outlook

- In-situ deformation paused for orientation mapping snapshots
- Orientation mapping through spot electron diffraction patterns using ASTAR

Advantages:

- Simultaneous detection of all grain orientations during deformation
- Direct analysis of grain rotation and growth as one of the major deformation mechanisms

nc PdAu 6 nm thickness, step 0.4 nm, 100 x100 steps

- diffraction
- orientation map
- index map
- overlay