# Extracting the Activation Parameters of Flow Defects from Nanoindentation Experiments

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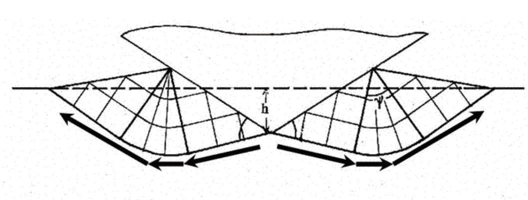
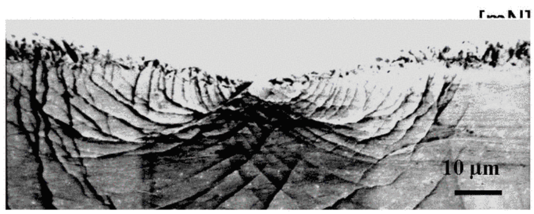
Dr. T. G. Nieh (ORNL)

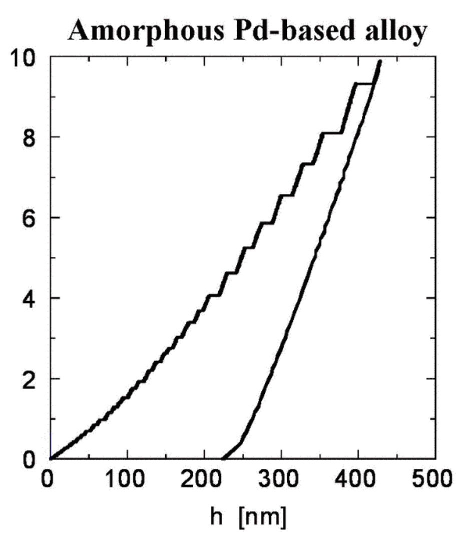
U.S. Army Research Office, U.S. Office of Naval Research

## Discrete Deformation Events in Nanoindentation




**Zr-Cu-Ni-Al:** Courtesy of S. Jana and U. Ramamurty, Indian Institute of Science



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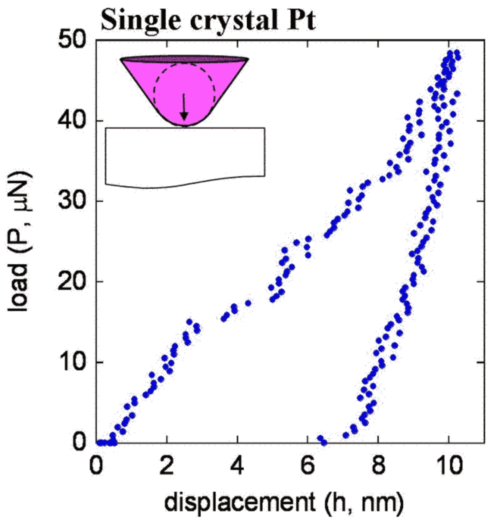


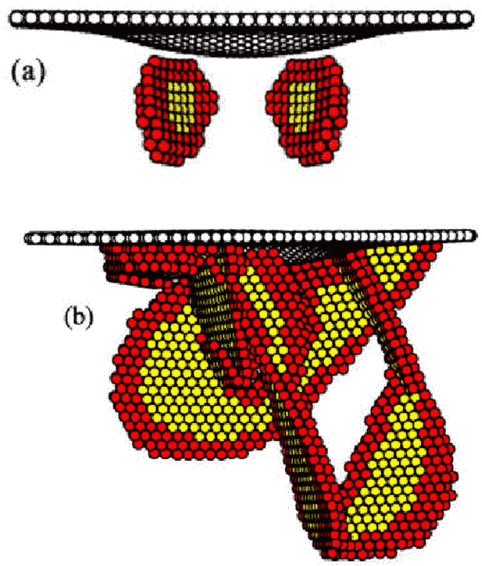


Kelchner, Plimpton, Hamilton,
Physical Review B 1998

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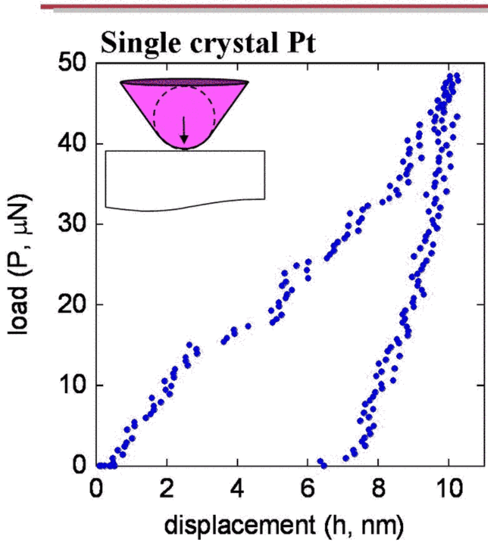


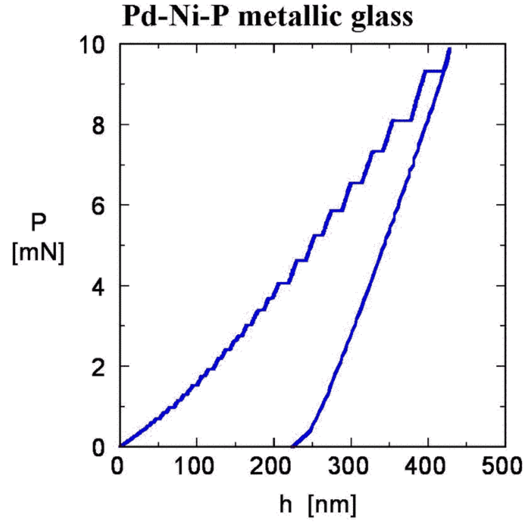


**Dislocation bursts**

**Shear band formation**

## Quantitative Analysis: Two Strategies

**CONSTITUTIVE APPROACH:**

Treat indentation like any mechanial tests

Map deformation behavior in rate-temperature space

Fit constitutive laws to the data to extract mean activation volume, etc.

**STATISTICAL APPROACH:**

Focus on the first pop-in, which is assumed to be nucleation controlled

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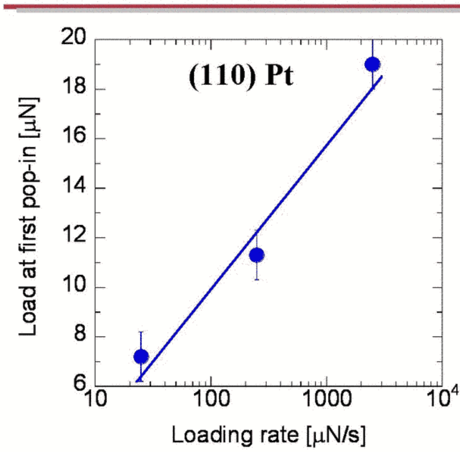
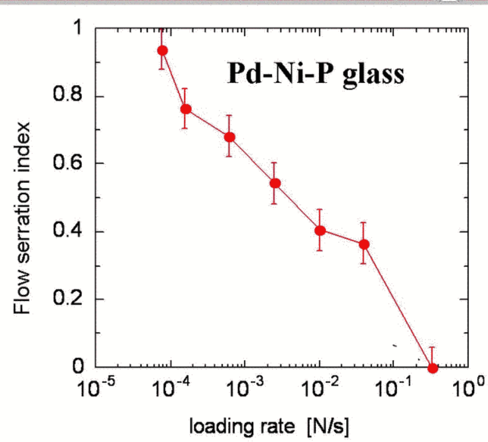
## Variation of Rate is Straightforward…



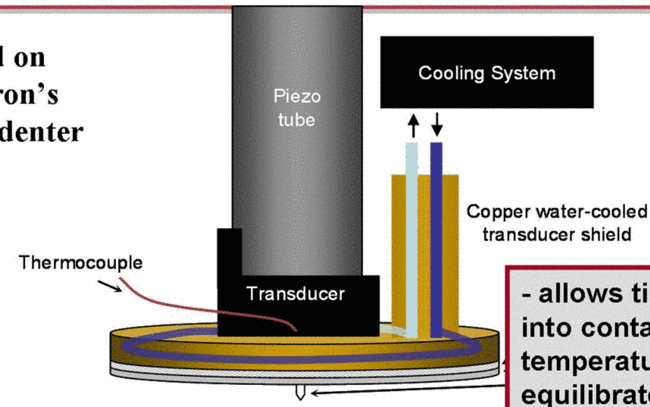

**Dislocation nucleation (?)**

**Shear band nucleation**

**These discrete events are significantly rate dependent!**

## …Variation of temperature is more difficult

**Based on Hysitron's Triboindenter**




- allows tip to be brought into contact at temperature, and to equilibrate while scanning

- electronics are maintained at room temperature, while the specimen can be heated to ~410 C: only little increase in noise or loss of resolution

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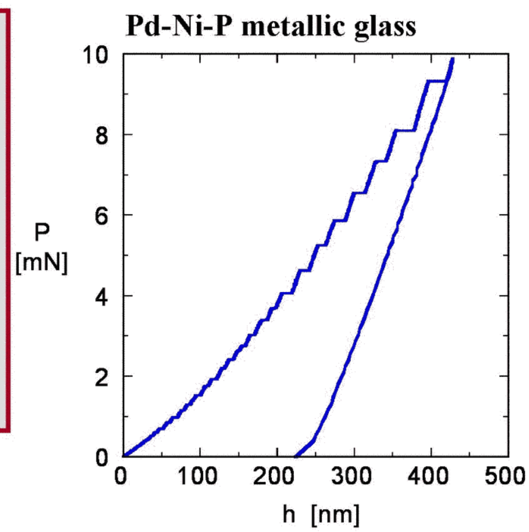
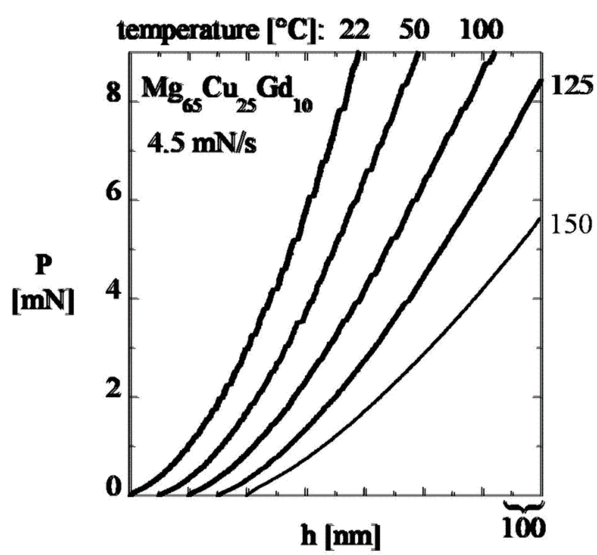



Shear band formation

## Temperature effects




Classical glass behavior:

Higher temperatures lead to homogeneous flow

# Collapsing the data: Deformation Map

**Spaepen, Acta Metall 1977**

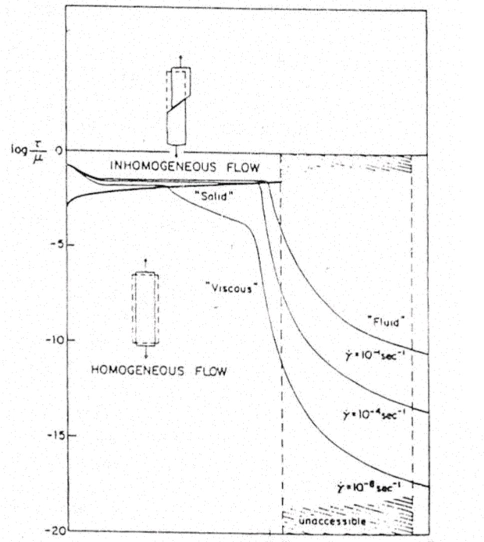
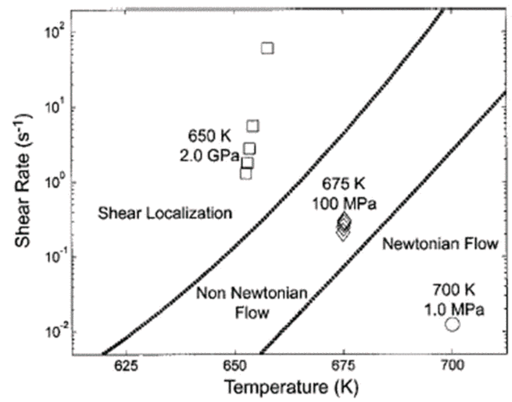
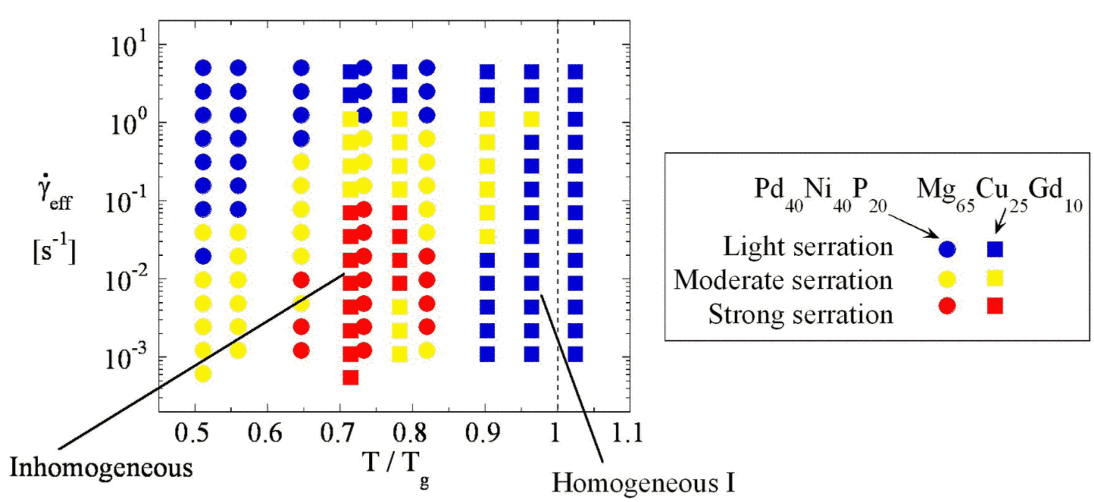



Fig. 2. Schematic deformation map of a metallic glass. The various modes of deformation are indicated.

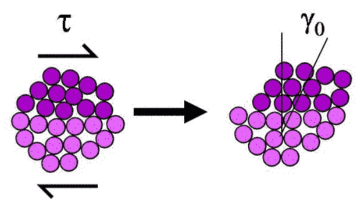



**Demetriou and Johnson, 2004**

# Deformation Map derived from High Temperature Nanoindentation



## STZ dynamics: Argon Model (1979)




$$\dot{\gamma} = \alpha_o \nu_o \gamma_o \cdot \exp\left(-\frac{\Delta F_o}{kT}\right)\sinh\left(\frac{\tau\gamma_o\Omega_o}{kT}\right)$$

$$\Delta F_o = \left[\frac{7-5\nu}{30(1-\nu)} + \frac{2(1+\nu)}{9(1-\nu)}\beta^2 + \frac{1}{2\gamma_o}\cdot\frac{\tau_o}{\mu(T)}\right]\cdot\mu(T)\cdot\gamma_o^{\,2}\cdot\Omega_o$$

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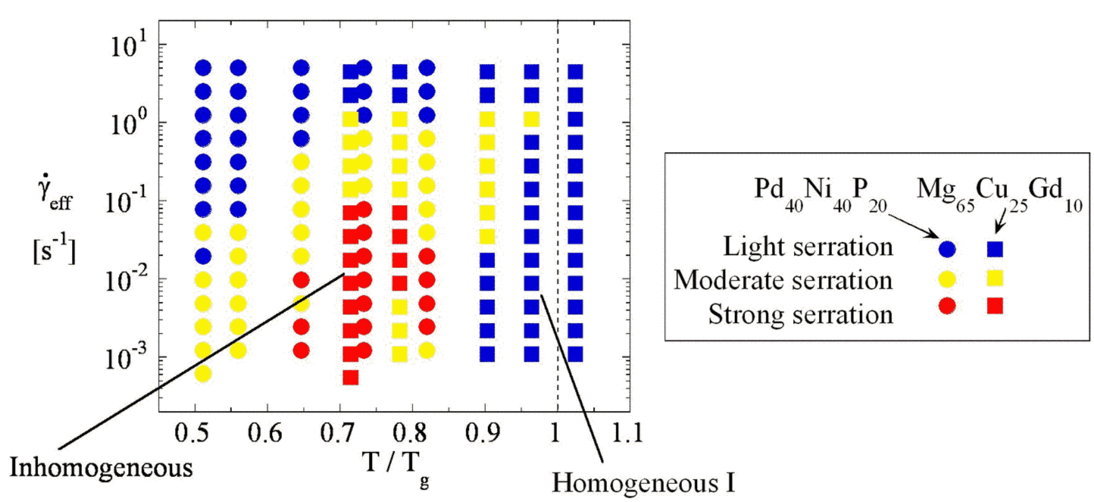

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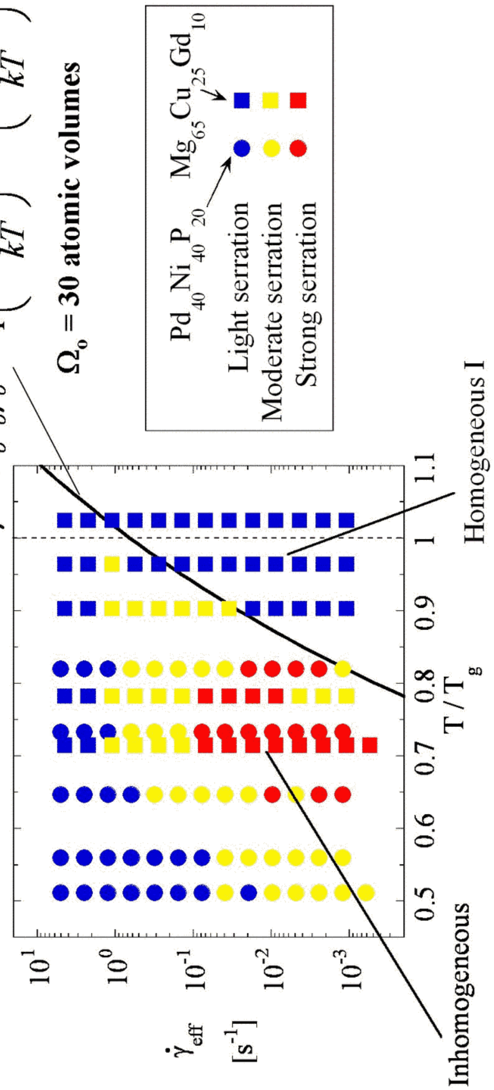
$$\dot{\gamma} = \alpha_o \nu_o \gamma_o \cdot \exp\left(-\frac{\Delta F_o}{kT}\right)\sinh\left(\frac{\tau\gamma_o\Omega_o}{kT}\right)$$

$\Omega_o$ = **30 atomic volumes**

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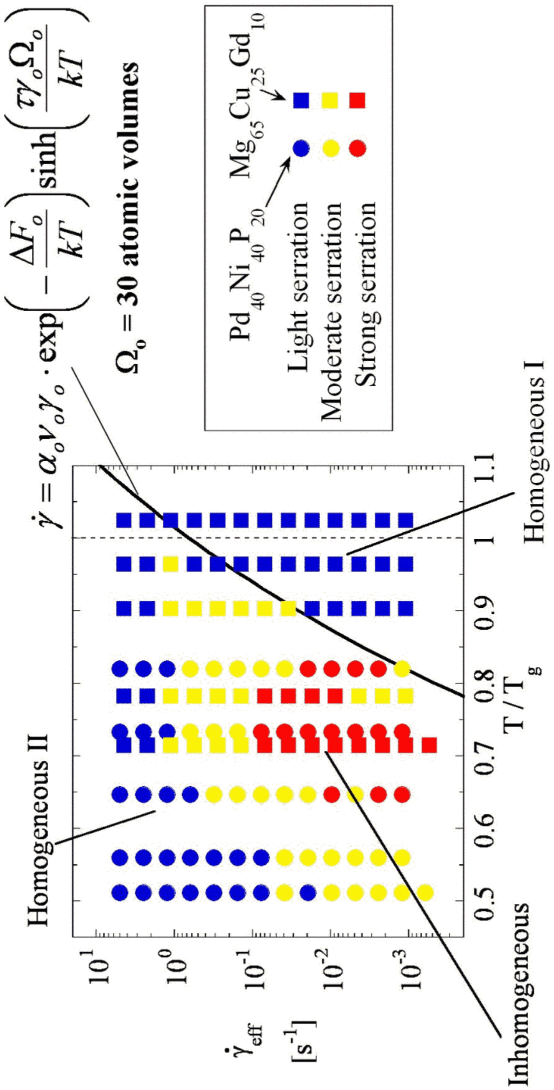
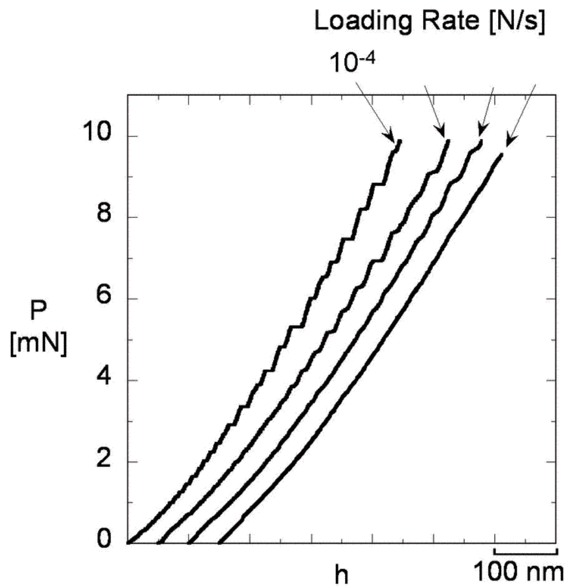


## Rate Effect

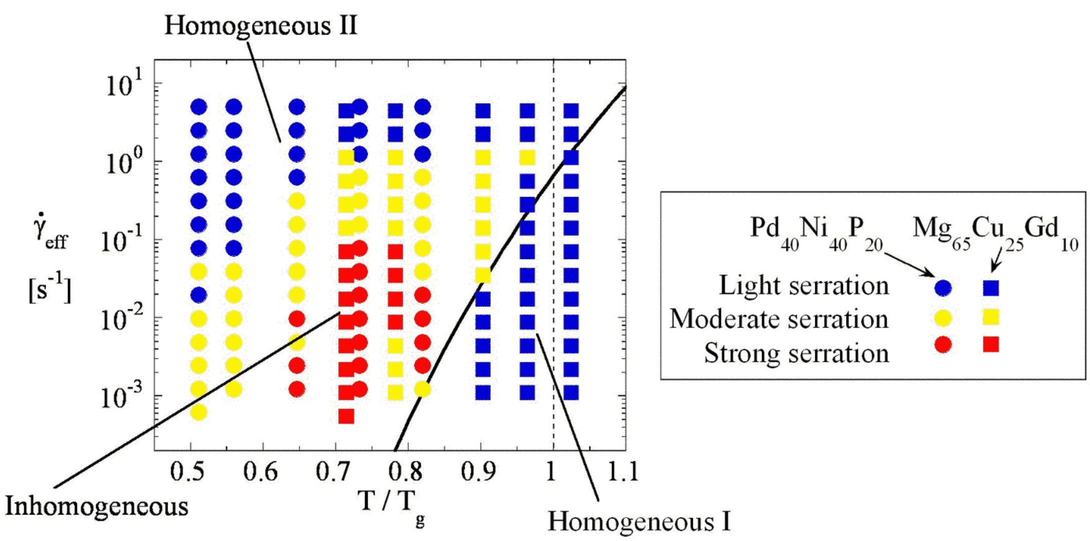



Pd-30Cu-10Ni-20P

Serrations are more evident at low indentation rates, and disappear at high rates.

Schuh and Nieh,
Acta Mater, v51, p87, 2003

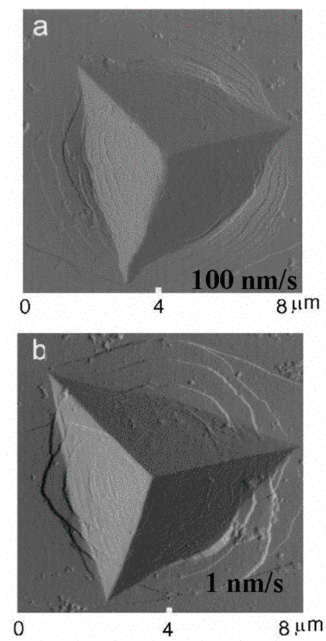
## Deformation Map derived from High Temperature Nanoindentation



## Kinetic limitations on shear banding

Kinetic limitations, with thermal activation:

Indenting too fast for a single shear band…




Jiang and Atzmon,
Al-5Fe-5Gd
JMR 2003, p756

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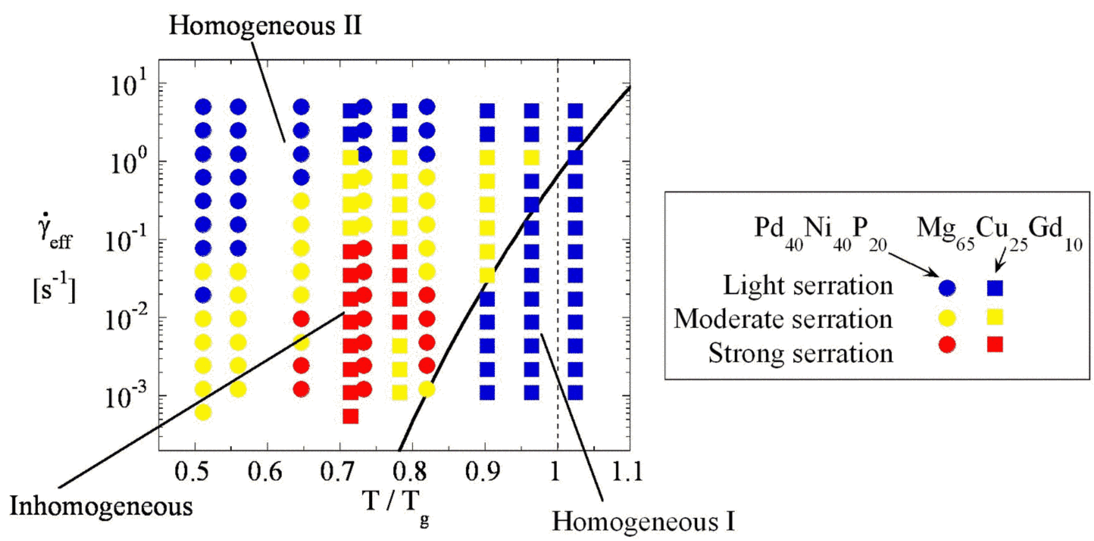

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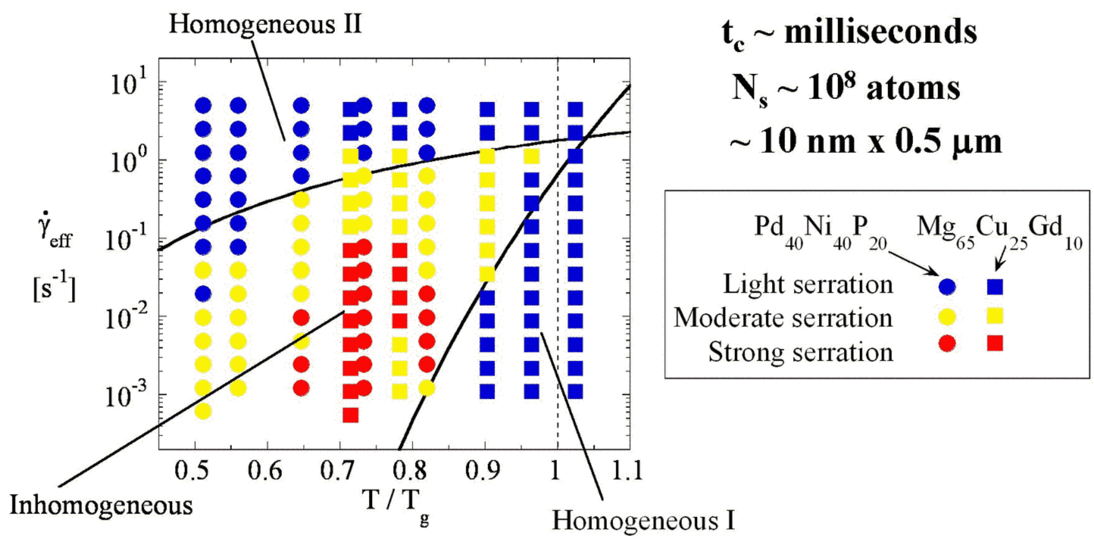


**$t_c$ ~ milliseconds**

**$N_s$ ~ $10^8$ atoms**

**~ 10 nm x 0.5 μm**

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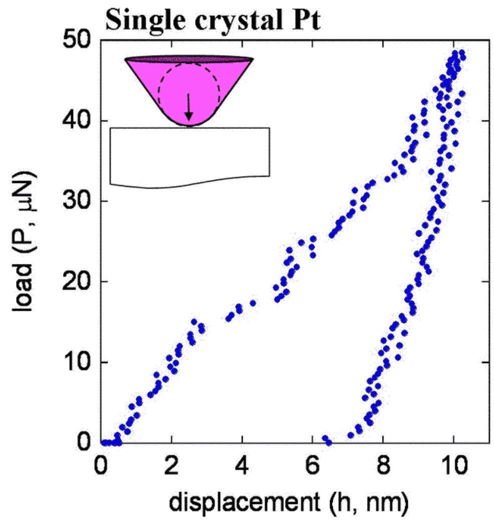


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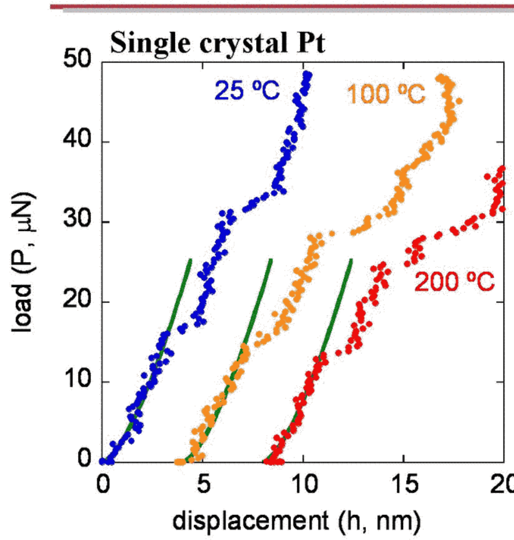


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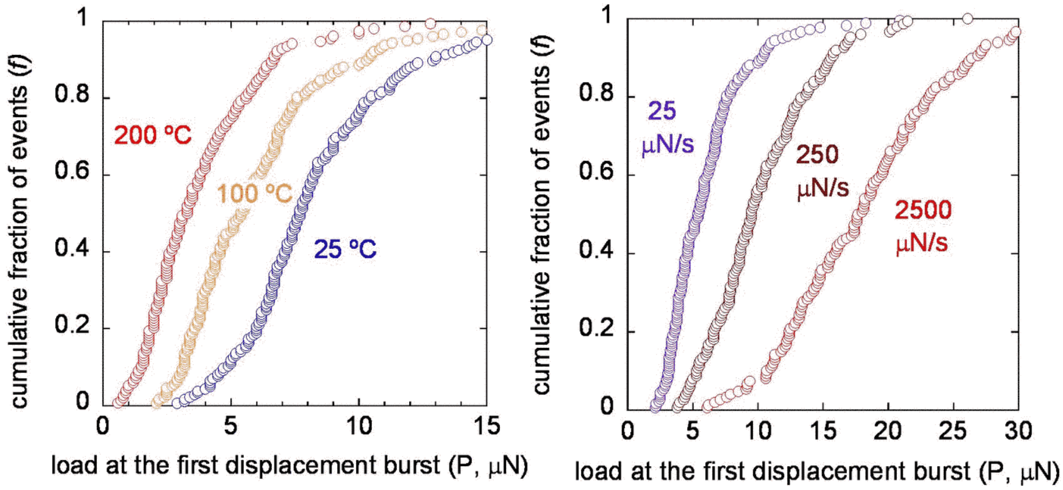
## Incipient Plasticity in (110) Pt




## Quantitative Analysis
## Strategy #2: Nucleation Statistics Approach

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Fit with statistical nucleation models based on shear-biased thermal activation

$$\dot{n} = \eta \exp\left(-\frac{\varepsilon - \tau V}{kT}\right)$$

$$\dot{N} = \eta \cdot \exp\left(-\frac{\varepsilon}{kT}\right) \cdot \iiint_{\Omega} \exp\left(\frac{\tau V}{kT}\right) d\Omega$$

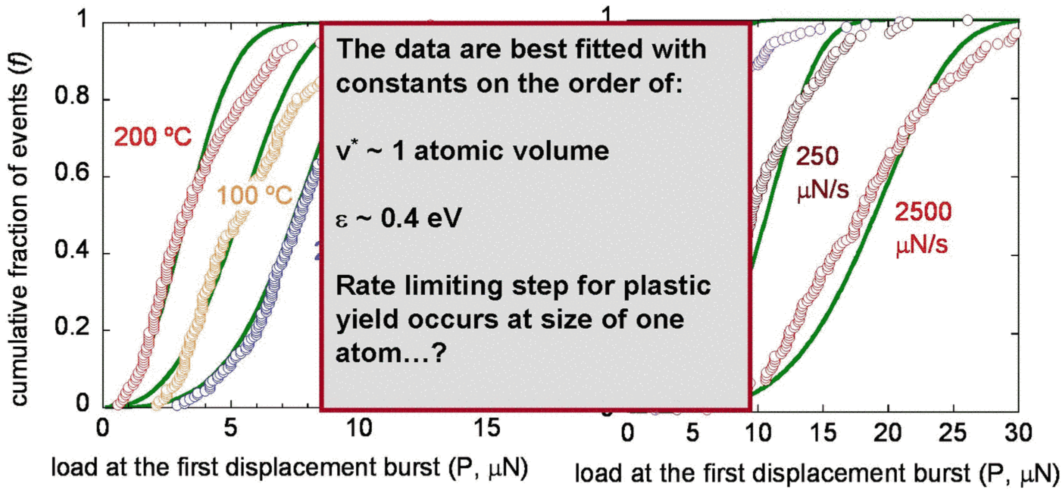
$\tau$ from Hertz theory

$$\dot{f} = (1 - f) \cdot \dot{N}$$

Linearized solution

$$F(P) = 1 - \exp\left\{ \begin{array}{l} -\dfrac{9K\rho\eta}{4E_R\dot{P}\alpha^6}\exp\left(-\dfrac{\varepsilon}{kT}\right)\cdot \\ \cdot\left[120 + \exp\left(P^{1/3}\alpha\right)\cdot\left(P^{5/3}\alpha^5 - 5P^{4/3}\alpha^4 + 20P\alpha^3 - 60P^{2/3}\alpha^2 + 120P^{1/3}\alpha - 120\right)\right] \end{array} \right\}$$

## Incipient Plasticity in (110) Pt



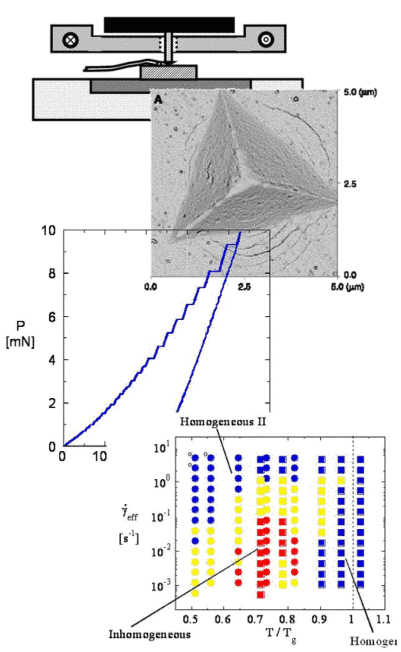

The data are best fitted with constants on the order of:

$v^* \sim 1$ atomic volume

$\varepsilon \sim 0.4$ eV

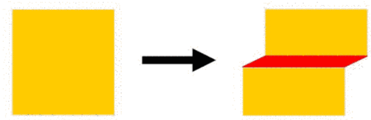
Rate limiting step for plastic yield occurs at size of one atom…?

## Summary




1) High temperature nanoindentation allows detailed study of thermally-activated discrete deformation phenomena
2) The "Constitutive Approach" to data analysis fits global flow laws to data, and has been used to extract the size of flow defects and shear bands in metallic glasses
3) The "Statistical Approach" focuses upon a well-defined event under the indenter, and matches experiment to a statistical nucleation model; the rate limiting event for plastic flow is of atomic dimensions in crystals of Pt.

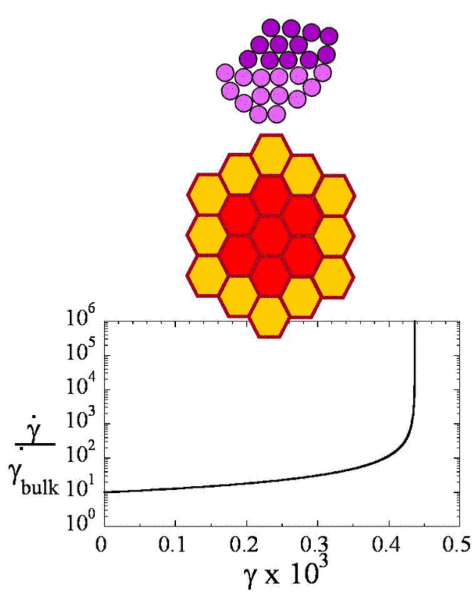
## Phenomenology of shear bands



$$\dot{\gamma} \approx \gamma_n \frac{\nu_D}{N_s} \cdot \cancelto{1}{\exp\left(-\frac{\Delta F - \tau\gamma_n\Omega_s}{kT}\right)}$$

**The critical strain rate occurs when the nucleation kinetics are 'swamped out' by the applied strain rate**

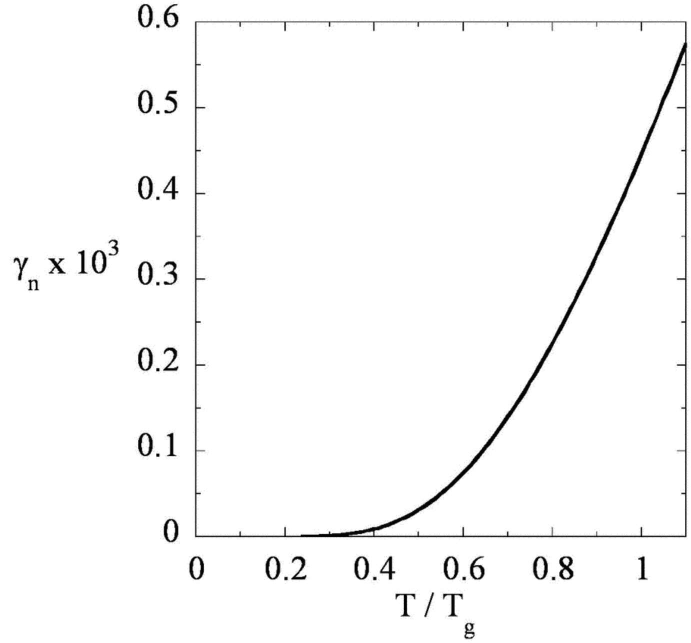
## Shear band dynamics




**Kinetics of a Shear Band:**

1. Single STZ action (subcritical)
2. Multiple STZ action (nucleus formation)
3. Partitioning of strain rate

## Temperature-Dependent Shear Band Nucleation

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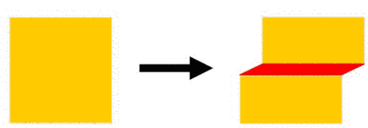

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**The critical strain rate occurs when the nucleation kinetics are 'swamped out' by the applied strain rate**

## Temperature Effect

We see that shear bands are promoted by modest increases in temperature

Acta Materialia (2004), volume 52, p5879