Scaling and size effects in fatigue of micro- and nano-structured fcc metals

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Motivation from modern applications
Scaling and size effects
Challenges in experimental mechanics
Fatigue in microsamples and thin film fatigue
Wrap-Up
Scaling effects

electric resistance \[ R = \rho \frac{L}{A} \propto \ell^{-1} \]
\( \rho \) - material dependent resistivity

diffusion \[ \tau = \frac{L^2}{2D} \propto \ell^2 \]
\( D \) - diffusion

friction \[ \mu = \frac{F_{\text{tan g}}}{F_{\text{norm}}} \propto \ell^0 \]
Scaling Effect

\[ R = S_{\text{spec}} \cdot \frac{l}{A} \]

\( S = \text{specific resistivity} \}
\( l = \text{conductor length} \}
\( A = \text{cross section} \}

\( \text{Mukral} \}
\( \text{Geometry} \)
Scaling Effect

\[ R = S_{\text{spec}} \cdot \frac{l}{A} \]

Trivial but important

→ Geometric effects can easily be determined but need to be accounted for.
Scaling Effect + Size Effect

\[ R = \frac{S_{\text{spec}}}{A} \sim S(l) \cdot \frac{A}{l} \]

\( S \) = specific resistivity \( M \) = metal \( L \) = conductor length \( A \) = cross section

Scaling Effect + Size Effect

\[ R = S_{\text{spec}} \cdot \frac{l}{A} \sim S(l) \cdot \frac{1}{l} \]

Strong effects on materials property

→ Characteristic length of defects interferes e.g. with sample dimensions or microstructural length scales.

Mechanical properties: larger is weaker?

Galilei G., *Discorsi, e dimostrazioni matematiche, Intorno à due nuoue scienze, Attenenti alla Mecanica, & i Mouimenti Locali*, Bologna, Per gli HH. del Dozza, 1655
Mechanism based size effects – thin film fatigue

Stress Amplitude $\sigma_a$ [MPa]

- 120
- 110
- 100
- 90
- 80
- 70
- 60
- 50
- 40
- 30
- 20
- 10
- 0

Film thickness [µm]

- 0.0
- 0.2
- 0.4
- 0.6
- 0.8
- 1.0
- 1.2
- 1.4
- 1.6
- 1.8

Schwaiger, Kraft, Acta 2001

3 µm Cu

G. P. Zhang et al., Phil. Mag. Let., 2003
Classical size effects $\Rightarrow$ Fatigue at 20kHz

(a) $\phi 8 \times 10$ mm specimen (ultrasonic)

- Volume of high stress: 781 mm$^3$
- Life time

(b) $\phi 7$ mm specimen (ultrasonic)

- Volume of high stress: 254 mm$^3$

(c) $\phi 3$ mm specimen (ultrasonic)

- Volume of high stress: 33 mm$^3$

High strength steel—Cracks nucleate at inclusions, Y. Furuya, Scripta Mat. 2008
Defect free vs. predeformed Mo crystals under compression

Defect free vs. predeformed Mo crystals under compression

Materials have a natural defect distribution - McDowell

→ Defect distribution, density or distance interfere with sample dimension.

Frequency effects
E.g. internal friction

Gremaud, 2001

Blind spot

Dislocation-phonon relaxation of segment L

Dislocations can not move

Hysteric IF background

Peaks of interaction with obstacles

Micro creep
Frequency effects
E.g. internal friction

- Frequency strongly influences active defect mechanisms
  - loading frequency interferes with characteristic frequency of defects
  - time-temperature superposition

Gremaud, 2001
Size, scaling and frequency effects

- Scaling effects – purely geometric

- Size effects can be
  - mechanism based
    - characteristic material property
  - e.g. depending on their defect distribution
    - processing parameters

- Frequency effects are strongly depending on active defect mechanisms
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Evaluate Properties

- Bioinspired
- Metamaterials
- Thin Films and Alloys
- Carbon Allotropes
- Nano metals: e.g. nanoporous, -crystalline, and -twinned

Materials maturity

- Micromolded, Printed Materials
- Gradient Materials

Optimize Properties

- Coatings
Challenges for multiscale testing

Gianola und Eberl, JOM 2009
Challenges for multiscale testing

Load - bridging 13 orders of magnitude
Displacement – bridging 7 orders of magnitude

→ Multiscale testing needs to overcome these challenges
Non contact optical strain measurement

- 2D/3D Digital Image Correlation and Tracking (DICT)
  ca. 5 Frames per Second

- Digital Image Tracking (DIT)
  ca. 10,000 Lines per Second

- Interference Strain/Displacement Gage (ISDG)
  up to $10^6$ measurements per Second

W.N. Sharpe (JHU)

S. Bundschuh, D. S. Gianola, C. Eberl
http://www.mathworks.com/matlabcentral/fileexchange/12413
Bridging dimensions by experiments

- **Microsample uniaxial fatigue**

- **Microsample multi-axial fatigue of Al, Cu, Ni**

- **Thin films: High throughput fatigue testing of Cu and Al**
Methods and Experiments for Small Scale Fatigue Testing

Uniaxial fatigue from Hz to kHz
Novel Custom Build for Small Scale Testing:
Tension, Compression and Bending under Static and Cyclic Loading

T. Kennerknecht

load cell
piezo actuator
sample
load cell

T. Kennerknecht
Novel Custom Build for Small Scale Testing: Tension, Compression and Bending under Static and Cyclic Loading

Specs:

- **Force:** 5, 50 N Load cell, mN Resolution
- **Strain rate:** $10^{-5} \ldots 10^1$ 1/s
- **Strain meas.:** optical, 2D, 1D, rel displacement up to 10 nm
- **Frequency:** ..190Hz, needs stiffer load cell
- **PID control:** displacement and load, at 200kHz through FPGA
- **Time Res.:** ~ns

C. Eberl - VHCF and UHCF in small volumes and metal thin films – novel experiments and modeling
Novel Custom Build Resonant Micro Fatigue Setup

Probe

Masse 30 gr.

Wegsensor

T. Kennerknecht
Novel Custom Build Resonant Micro Fatigue Setup

Specs:
- Force ampli.: up to 25N @1kHz
- Strain meas.: optical, 2D (static 5fps), 1D 10kHz, 0D:200kHz
- Frequency: 0.5..5kHz, higher frequency, smaller Piezo
- PID control: load amplitude, up to 750kHz by NI FPGA
- Time Res.: ~ns
Micro molded Al-Bronze @ 2.5 Hz – 2 kHz
Micro molded by Durime Buqesi-Ahmeti

(A) Loading direction
Top surface
Side surface

(B) 10 μm

130 μm thick
260 μm
1 mm

- 2.5 Hz, r = 0.1
- 25 Hz, r = 0.1
- 100 Hz, r = 0.1
- 868 Hz, r = -0.9
- 900 Hz, r = -0.9
- 1000 Hz, r = -0.9
- 1079 Hz, r = -0.89
- 2154 Hz, r = -0.98
- 2362 Hz, r = -0.98

Cycles to failure

T. Kennerknecht
Methods and Experiments for Small Scale Fatigue Testing

Multiaxial fatigue
Novel Custom Build Resonant Micro Fatigue Setup

Multiaxial Fatigue

- spring
- laser beam
- support
- clamp
- area detector
- sample
- piezo actuators

T. Straub
Custom Build Resonant Micro Fatigue Setup

Specs:
- Displ. meas.: Laser, Bending and Torsion 30kHz
- Frequency: 0.1..1kHz
- PID control: displ. amplitude, by NI FPGA
- Time Res.: ~ns
Damage formation in Ni-foils – 200 µm thickness

Crack nucleation needs certain microstructural compositions:
- Transmission of dislocations at GBs – GB character
- Extrusion formation across grains

T. Straub

Institute for Applied Materials,
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Fatigue of thin films – high throughput experiments

S. Burger, A. Siegel, A. Ludwig, O. Kraft: KIT und RUB
Cu and Al thin film preparation
A. Siegel, A. Ludwig

etching process for structuring cantilever arrays

cantilever geometry

<table>
<thead>
<tr>
<th>grain size (µm)</th>
<th>Al</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td>‘as deposited’</td>
<td>0.8</td>
<td>0.2</td>
</tr>
<tr>
<td>‘annealed’ 1 h @ 450 °C</td>
<td>1.2</td>
<td>1.2</td>
</tr>
<tr>
<td>‘on Ti’ seed layer</td>
<td>0.4</td>
<td></td>
</tr>
</tbody>
</table>

4” (100) oxidized Si wafer

Si substrate thickness 200 µm

thin films (thickness ~ 1 µm) deposited by magnetron sputtering
High Throughput Fatigue Setup

High Throughput Fatigue Setup

Lebensdauerkriterium:
80 % Intensität

Position Y [mm]
Position X [mm]

Damaged zone
Position for strain amplitude

1 mm

Lifetime Criterion

Intensity scans of Al thin film

Strain amplitude [%]

Position Y [mm]

Cycles to failure

0.00
0.0160
0.0180
0.0200
0.0210
0.0230
0.0240
0.0260
0.0280
0.0300
0.0320
0.0340
0.0360
0.0380
0.0400

0.00
0.02
0.04
0.06
0.08
0.10
0.12
0.14

0.00
10^8
10^9
10^10
10^11

Position for strain amplitude

Damaged zone
Lifetime is influenced by grain size, material and interlayer.

Basquin equation:

$$\varepsilon_a = \frac{\sigma'_f}{E} \cdot (2N_f)^b$$

- $\varepsilon_a$: strain amplitude
- $\sigma'_f$: fatigue strength
- $E$: Young’s modulus
- $N_f$: number of cycles to failure
- $b$: fatigue sensitivity exponent
Fatigue of thin films - Cu and Al

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Novel experiments and modeling

S. Burger

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Phenomenological Lifetime Model

reflectivity depending on cycle number

transition of plot for fitting

limited exponential growth

Basquin type fit

adapted from Mitscherlich 1909

\[ F = F_{\text{max}} \left(1 - \exp\left(-\frac{1}{N_{0.5} \cdot N_F}\right)\right)^n \]

with

\[ N_{0.5} = K \cdot \varepsilon_a^q \]

\[ F \] fraction of damage
\[ F_{\text{max}} \] maximum fraction of damage
\[ N_{0.5} \] half lifetime
\[ N_F \] number of cycles for specific \( F \)
\( n \) growth mode
\( K \) fit parameter
\( q = 1/b \) inverse fatigue sensitivity exponent
\( \varepsilon_a \) strain amplitude

\[ \varepsilon_a \] strain amplitude

adapted from Mitscherlich 1909
Wrap-Up – Size and scaling effects -

![Graph showing the relationship between stress amplitude ($\sigma_a$) and number of cycles (Zyklen)](image)

- **CuAl10Ni5Fe4**: 2.5 Hz, $R = 0.1$
- **CuAl10Ni5Fe4**: 25 Hz, $R = 0.1$
- **CuAl10Ni5Fe4**: 100 Hz, $R = 0.1$
- **CuAl10Ni5Fe4**: 900 Hz, $R = -0.9$
- **CuAl10Ni5Fe4**: 1000 Hz, $R = -0.9$
- **CuAl10Ni5Fe4**: 2000 Hz, $R = -1$
- **Cu thin film**: 566 Hz, $R = -1$
- **Cu bulk [Thompson et al.]**
- **Cu Biegung Überkritische Amplitude 900 Hz, R = -1**
- **Cu Multiaxial Überkritische Amplitude 900 Hz, R = -1**
- **Cu Torsion Überkritische Amplitude 900 Hz, R = -1**
- **Cu Biegung Subkritische Amplitude 900 Hz, R = -1**
• Scaling effects – purely geometric

• Size effects can be
  • mechanism based
    → characteristic material property
  • e.g. depending on their defect distribution
    → processing parameters

• Frequency effects are strongly depending on active defect mechanisms
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