Spatial distribution of defects in ultra fine grained metals prepared by high pressure torsion

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Ultra fine grained (UFG) materials

polycrystalline material

\[
\delta/2
\]

\[
d
\]

volume fraction of GB’s

\[
f_{GB} = 1 - \left( \frac{d - \delta}{d} \right)^3
\]
Ultra fine grained (UFG) materials

crystallographic width of GB’s: $\delta \approx 1 \text{ nm}$

polycrystalline material

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Ultra fine grained (UFG) materials

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grain interior

grain boundary (GB)

crystallographic width of GB’s: $\delta \approx 1$ nm

volume fraction of GB’s

$$f_{GB} = 1 - \left(\frac{d - \delta}{d}\right)^3$$

UFG materials

$\delta / 2$

$\delta$

$d < 100$ nm nanocrystals

$100-1000$ nm UFG materials

$d > 1$ $\mu$m polycrystals
Introduction

Severe plastic deformation

• very strong grain size reduction → down to nanoscale ( ~ 100 nm)
• conventional plastic deformation
Introduction

Severe plastic deformation

• very strong grain size reduction → down to nanoscale ( ~ 100 nm)

• conventional plastic deformation → formation of cracks
Introduction

Severe plastic deformation

- very strong grain size reduction $\rightarrow$ down to nanoscale ($\sim 100$ nm)
- conventional plastic deformation $\rightarrow$ formation of cracks $\rightarrow$ material failure
Introduction

Severe plastic deformation

• very strong grain size reduction → down to nanoscale (~ 100 nm)

• P.W. Bridgman, 1930, Harvard University
  Large hydrostatic pressure (10 GPa) + shear deformation
  Nobel prize in Physics 1946

• R.Z. Valiev, 1980, Ufa State Aviation Technical University
  High pressure torsion (HPT)
  Equal channel angular pressing (ECAP)
**Introduction**

**High pressure torsion (HPT)**

- the strongest grain refinement
- grain size 50 – 150 nm
- limited size: disk shaped samples
diameter 10 – 20 mm, thickness 0.3 - 0.5 mm

*UFG Cu sample prepared by HPT*

Introduction

High pressure torsion (HPT)

• the strongest grain refinement

• grain size 50 – 150 nm

\[ e = \frac{1}{\sqrt{3}} \frac{2\pi rN}{l} \]

• \( e \) - true strain
• \( N \) – number of rotations
• \( r \) - distance from center
• \( l \) - sample thickness

Equal Channel Angular Pressing (ECAP)

Equal Channel Angular Pressing (ECAP)

- grain size 100 - 500 nm
- no porosity
- high purity samples
- good reproducibility

- massive samples
  (diameter up to \( \approx 100 \) mm, length up to \( \approx 500 \) mm)

Samples

High pressure torsion (HPT)
• Ufa State Aviation Technical University
• $p = 6 \text{ GPa}, 1-25 \text{ HPT revolutions}$
• Cu (99.99%)

Equal channel angular pressing (ECAP)
• Ufa State Aviation Technical University
• route $B_c$, 1-10 passes
• Cu (99.99%)
Mechanical properties of UFG metals prepared by SPD

Hall-Petch relationship:

$$\sigma_y = \sigma_{y,0} + \frac{k}{\sqrt{d}}$$

UFG metals
↓
superior mechanical properties:

• high strength
• good ductility

Mechanical properties of UFG metals prepared by SPD

UFG metals

\[ \uparrow \]

superior mechanical properties:

- high strength
- good ductility

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**UFG Cu, \( d = 110 \text{ nm} \)**

**coarse-grained Cu, \( d \approx 30 \text{ \(\mu\)m} \)**

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Microstructure of UFG metals prepared by SPD

- grain size 105(9) nm
- fragmented structure
- high-angle misorientation
- non-equilibrium GB’s

**UFG Cu prepared by HPT, p = 6 GPa**

![Image](image_url)
Microstructure of UFG metals prepared by SPD

polycrystalline material

equilibrium GB: $\delta \approx 1$ nm
non-equilibrium GB: $\delta \approx 10$ nm

grain interior

grain boundary (GB)

volume fraction of GB’s

$$f_{GB} = 1 - \left(\frac{d - \delta}{d}\right)^3$$
Generation of vacancies during SPD

movement of dislocation with jogs

1. no stress
Generation of vacancies during SPD

movement of dislocation with jogs

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Generation of vacancies during SPD

movement of dislocation with jogs

1. no stress

2. applied stress
Generation of vacancies during SPD

movement of dislocation with jogs

1. no stress

2. applied stress

3. jogs dragged by dislocation
Generation of vacancies during SPD

- X-ray line profile analysis:
  - dislocation density $\rho_D \approx 10^{14} - 10^{15} \text{ m}^{-2}$
  

- differential scanning calorimetry (DSC) & electrical resistivity
  - dislocation density $\rho_D \approx 10^{15} - 10^{16} \text{ m}^{-2}$
  

![Diagram showing XRD and DSC data for UFG Cu](image-url)
Generation of vacancies during SPD

defor­ma­tion-induced vacancies contribute to the total stored energy and electrical resistivity, but do not cause broadening of XRD profiles

• indirect evidence for deformation-induced vacancies

• deformation-induced vacancies can be detected probed by positron annihilation

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UFG Cu
T. Ungár et al.
LT spectroscopy


- Two photomultipliers Hamamatsu H3378
- BaF₂ scintillators truncated cone (Ø 18-36 mm, thickness 12 mm)
- Two 8-bit digitizers Acqiris DC211, sampling rate 4 GHz
- Time resolution 145 ps (FWHM $^{22}$Na)
- At least $10^7$ annihilation events collected in each LT spectrum
### Positron lifetime studies of Cu

- pure Cu (99.99%)

<table>
<thead>
<tr>
<th>sample</th>
<th>$\tau_1$ (ps)</th>
<th>$I_1$ (%)</th>
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<td>well annealed 800°C/1h</td>
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- theoretical calculations: Cu bulk lifetime
  - $\tau_B = 108$ ps (ATSUP, LDA)
  - $\tau_B = 118$ ps (LMTO, GGA)

free positrons
# Positron lifetime studies of Cu

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<td>cold rolled, 50%, $\varepsilon = 0.69$</td>
<td>68(4)</td>
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<td>165(1)</td>
<td>84.7(4)</td>
<td>258(3)</td>
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- theoretical calculations: Cu monovacancy
  - $\tau_V = 170$ ps (ATSUP, LDA)
  - $\tau_V = 178$ ps (LMTO, GGA)
Positron lifetime studies of UFG Cu prepared by ECAP

- pure Cu (99.99%), ECAP $B_c$ route
- saturated trapping at dislocations and vacancy clusters
- fraction of $e^+$ trapped at vacancy clusters increases with number of passes
# Positron lifetime studies of Cu

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<td>HPT, N = 5, $r = 0$ mm, $\varepsilon = 40$</td>
<td>-</td>
<td>-</td>
<td>164(1)</td>
<td>82.0(8)</td>
<td>255(3)</td>
<td>18(1)</td>
</tr>
<tr>
<td>HPT, N = 5, $r = 3$ mm, $\varepsilon = 180$</td>
<td>-</td>
<td>-</td>
<td>163(1)</td>
<td>70.5(5)</td>
<td>310(3)</td>
<td>29.5(7)</td>
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![Diagram](image.png)

- free positrons
- dislocations
- vacancy clusters
Positron lifetime studies of UFG Cu prepared by HPT

• pure Cu (99.99%), HPT p = 6GPa
• center $r = 0$
• intensity of the cluster component increases with the number of HPT revolutions

$\tau_2$ - dislocations
$\tau_3$ - vacancy clusters, center

J. Čížek et al., Acta Mater. 59, 2322 (2011)
Positron lifetime studies of UFG Cu prepared by HPT

- pure Cu (99.99%), HPT p = 6GPa
- center $r = 0$, periphery $r = 3$ mm
- diameter of positron source spot $\approx 3$ mm
- vacancy clusters at periphery are larger

---

**Graph 1:**
- $\tau_2$ - dislocations
- $\tau_3$ - vacancy clusters, center
- $\tau_3$ - vacancy clusters, periphery

**Graph 2:**
- $I_3$ - vacancy clusters
- $I_3$ - vacancy clusters, periphery

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*J. Čížek et al., Acta Mater. 59, 2322 (2011)*
Size distribution of vacancy clusters

• movement of dislocation with a jog

• monovacancies in Cu are mobile at room temperature

Size distribution of vacancy clusters

- movement of dislocation with a jog

- some fraction of vacancies disappear by diffusion to sinks at grain boundaries

- monovacancies in Cu are mobile at room temperature

- remaining vacancies agglomerate to small vacancy clusters

UFG microstructure

- dislocations are located mainly in distorted layers along GB’s
- vacancy clusters are located predominantly inside grains

UFG Cu prepared by HPT, $p = 6$ GPa, $N = 5$

Size of vacancy clusters

- *ab-initio* calculations of positron lifetimes for vacancy clusters of various size
- ECAP Cu: clusters consist on average of 4 vacancies
Size of vacancy clusters

- *ab-initio* calculations of positron lifetimes for vacancy clusters of various size
- HPT Cu: cluster size increases with radial distance $r$ from the center
- HPT Cu: clusters in the center consist of 4 vacancies
- HPT Cu: clusters at the periphery consist of 6 vacancies
Size distribution of vacancy clusters


- decomposition of LT spectrum: discreet components + cluster distribution

\[
S(t) = \left( \sum_{i=1}^{n-1} \frac{I_i}{\tau_i} e^{-\frac{t}{\tau_i}} \right) + I_d \left( \sum_{N=1}^{N_{\text{max}}} P(N) \nu_N \right)^{-1} \sum_{N=1}^{N_{\text{max}}} \frac{P(N) \nu_N}{\tau_N} e^{-\frac{t}{\tau_N}} \right) \otimes R(t) + B \sum_{i=1}^{n-1} I_i + I_d = 1
\]

discreet components
- **dislocations**,  
- **free positrons (if any)**

\( \tau_N \) - positron lifetimes for clusters consisting of \( N \) vacancies  
- obtained from theoretical calculations

\( \nu_N \) - the specific positron trapping rates for a clusters of various sizes

\( P(N) \) - the relative population of clusters consisting of \( N \) vacancies
- Poisson distribution

\[
P(N) = \frac{\nu_d^N e^{-\nu_d}}{N!} \quad \sum_{N=1}^{N_{\text{max}}} P(N) = 1
\]
Development of deformation-induced vacancy clusters

- HPT-deformed Cu, \( p = 6 \) GPa
- comparison of centre and periphery of the sample disk
- periphery of the sample
  - higher vacancy concentration
- bigger vacancy clusters

1 HPT revolution

25 HPT revolutions

[pdf: 1 HPT revolution vs 25 HPT revolutions with graphs showing vacancy clustering comparison]
Development of deformation-induced vacancy clusters

• HPT-deformed Cu, $p = 6$ GPa
• comparison of centre and periphery of the sample disk
• periphery: higher vacancy concentration, bigger clusters
• increasing number of HPT revolutions:
  - concentration of vacancies increases
  - size distribution remains constant

![Diagram showing concentration of vacancies at center and periphery with increasing HPT revolutions.](image)
Production of deformation-induced vacancies

- production rate of deformation-induced vacancies

\[ \Pi = \alpha \frac{\sigma \Omega_0}{E_f} \dot{\varepsilon} \]

- strain rate

- coefficient \( \alpha \approx 0.1 \)  

- \( \sigma \): applied stress

- \( \Omega_0 \): atomic volume

- \( E_f \): vacancy formation energy

- \( \dot{\varepsilon} \): strain rate

- strain rate increases with \( r \)
Mapping of spatial distribution of defects

- spatially resolved LT investigations (1 point ≈ 1 day)
  - diameter of positron source spot < 1 mm
  - spatial resolution ≈ 0.5 mm

- spatially resolved DB mapping (1 point ≈ 30 min)
  - diameter of positron source spot < 1 mm
  - spatial resolution ≈ 0.5 mm

J. Čižek et al., Scripta Mater. 65, 171 (2011)
Mapping of spatial distribution of defects

- Vickers microhardness mapping
- automatic hardness tester Struers Durascan
- loading force 1 N applied for 10 s
- spatial resolution \( \approx 50 \, \mu\text{m} \)
• HV is influenced
  - by dislocations
    \[ HV \approx \sqrt{\rho_D} \]
    (strain hardening)
  - by grain size
    \[ HV \approx d^{-1/2} \]
    (Hall-Petch relation)
  - but not by vacancies
Spatial distribution of defects - microhardness (HV)

UFG Cu, HPT deformed, $p = 6$ GPa

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Spatial distribution of defects - microhardness (HV)

- HV is almost uniform after 15 HPT revolutions
- except of a small region just in the center

UFG Cu, HPT deformed, $p = 6$ GPa

1 HPT revolution

3 HPT revolutions

15 HPT revolutions

25 HPT revolutions
Homogeneity of microstructure - TEM

- HPT deformed Cu, $p = 6$ GPa, $N = 15$

center ($r = 0$)  

periphery ($r = 3.5$ mm)
Spatial distribution of defects – S parameter

UFG Cu, HPT deformed, $p = 6$ GPa

- $S$ parameter is sensitive to
  - dislocation density
  - size and concentration of vacancy clusters

1 HPT revolution
Spatial distribution of defects – $S$ parameter

UFG Cu, HPT deformed, $p = 6$ GPa

- $S$ parameter is sensitive to
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UFG Cu, HPT deformed, $p = 6$ GPa

- $S$ parameter is sensitive to
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Spatial distribution of defects – S parameter

UFG Cu, HPT deformed, p = 6 GPa

• S parameter is sensitive to
  - dislocation density
  - size and concentration of vacancy clusters

• S parameter increases with r
  • increasing size of vacancy clusters
Spatial distribution of defects - comparison of HV and S parameter

UFG Cu, HPT deformed, $p = 6$ GPa, 25 HPT revolutions

- **microhardness (HV)** is influenced by
  - grain size
  - dislocation density
  for more than 25 HPT revolutions HV becomes uniform

- **S parameter** is influenced by
  - dislocation density
  - concentration and size of vacancy clusters
  after 25 HPT revolutions S is still higher at the periphery

- spatial distribution of vacancy clusters is far from being uniform even after 25 HPT rotations
Spatial distribution of defects – LT spectroscopy

- HPT deformed Cu, $p = 6$ GPa, $N = 2$
- size & concentration of vacancy clusters increase with $r$

![Graph showing spatial distribution of defects](image)
Thermal stability of UFG structure – HV mapping

• HPT deformed Cu, $p = 6$ GPa, $N = 2$

• isochronnal annealing $20^\circ$C/20 min
Thermal stability of UFG structure – HV mapping

- HPT deformed Cu, $p = 6$ GPa, $N = 2$
- Isochronnal annealing $20^\circ$C/20 min
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- HPT deformed Cu, $p = 6$ GPa, $N = 2$
- Isochronnal annealing $20^\circ C/20$ min
- Radial dependence of microhardness
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- Radial dependence of microhardness

![Graph showing radial dependence of microhardness for different temperatures.](image-url)
Thermal stability of UFG structure – HV mapping

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- isochronnal annealing $20^\circ$C/20 min
- radial dependence of microhardness

![Graph showing radial dependence of microhardness](image)
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Thermal stability of UFG structure – HV mapping

- HPT deformed Cu, $p = 6$ GPa, $N = 2$
- isochronnal annealing $20^\circ$C/20 min
- recovery of UFG structure starts at the periphery → higher stored deformation energy

![Graph showing HV mapping for different temperatures](image-url)
Thermal stability of UFG structure – HV mapping

- HPT deformed Cu, $p = 6$ GPa, $N = 2$
- isochronnal annealing $20^\circ C/20$ min
- averaged hardness

![Graph showing HV mapping with temperature (T) in °C on the x-axis and hardness (HV) on the y-axis. The central region ($r < 1$ mm) is highlighted.](image)
Thermal stability of UFG structure – HV mapping

- HPT deformed Cu, $p = 6$ GPa, $N = 2$
- Isochronnal annealing $20^\circ C/20$ min
- Averaged hardness

![Graph showing HV mapping vs. temperature for different regions]
Thermal stability of UFG structure – HV mapping

- HPT deformed Cu, $p = 6$ GPa, $N = 2$
- isochronnal annealing $20^\circ C/20$ min
- averaged hardness
Thermal stability of UFG structure – $S$ parameter

UFG Cu, HPT deformed, $p = 6$ GPa, $N = 2$

- recovery of UFG structure
- annealing of vacancy clusters
- recovery of dislocations
- recovery starts at the periphery
Thermal stability of UFG structure – LT mapping

- HPT deformed Cu, $p = 6$ GPa, $N = 2$
- isochronnal annealing $20^\circ C/20$ min
- recovery of vacancy clusters starts at the periphery
- free positron component appears firstly at the periphery ($200^\circ C$)

\[ \tau_1 \text{ - free positrons} \]
\[ \tau_2 \text{ - dislocations} \]
\[ \tau_3 \text{ - vacancy clusters} \]

$I_3$ - vacancy clusters

![Graph](image)
Conclusions

• Severe plastic deformation introduces dislocations and vacancies which subsequently agglomerate and form small vacancy clusters.

• Size distribution of vacancy clusters was determined. In UFG Cu vacancy clusters consist of 4-6 vacancies.

• Size of vacancy clusters increases with the strain rate due to higher production rate of vacancies → size of vacancy clusters in HPT-deformed sample increases from center towards periphery.

• Spatial distribution of defects was mapped by microhardness and S-parameter.
  - microhardness is sensitive to dislocation density and grain size.
  - S-parameter is influenced by dislocations and vacancy clusters.

• Recovery of UFG structure starts at the periphery of HPT-deformed sample due to higher stored deformation energy.