MICROSTRUCTURAL INVESTIGATION OF SPD PROCESSED MATERIALS – CASE STUDY

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Cold rolled Grain Oriented Si Steel
Deep drawing of Aluminum

- Al cans - weight reduction by 15-20%
  - Cost reduction: ~25%
  - 80-90% of market shares for soft drinks and food items

- \{111\}<110> & {111}\{112> are most preferred, next is {554}\{225>
LENGTH SCALE EFFECTS ON THE EVOLUTION OF TEXTURE IN MULTILAYERS

Basic micromechanisms of deformation and recrystallization
Introduction & Motivation

• Metallic multilayers through ARB
  • High potential for commercial manufacture of bulk nanocrystalline materials
  • Designing new microstructure \( \rightarrow \) control texture of bulk through interfaces
  • Interfaces \( \rightarrow \) Change in both deformation & recrystallization mechanism
    • Restricts cross slip across interface, & grain/twin growth

• Single phase laminates
  • Restriction in cross slip at lower length scale
  • Constrained grain growth during annealing

• Two-phase laminates
  • Control evolution of texture using OR at interfaces
  • Orientation Relationship changes at different length scales
  • Control of interface texture \( \rightarrow \) bulk texture

• Enhance mechanical, electrical and thermal properties
  • Increase fraction of special boundaries \( \rightarrow \) Better stability against electro-migration
  • Improvement in strength & fatigue properties
What are the length scale effects?

What are the length scale effects?

- Deformation mechanisms at different regime
- Layer size > sub micrometer: Hall-Petch hardening based on dislocation piling up against the interface
- Few nm to few 10 nm: Confined layer slip $\rightarrow$ single dislocation loop confined to individual layer
- Couple of nm: Interface barrier strength $\rightarrow$ single dislocation cutting across the interface

Existing Lacunae

- Are the deformation mechanisms are very similar for the entire range of layer thickness > sub-$\mu$m? Do we get similar deformation texture?
- What are the micromechanisms leading to different texture at smaller length scale?
- Does the interface decides the texture of bulk or vice-versa?
### Accumulative Roll Bonding (ARB) of Multilayer

<table>
<thead>
<tr>
<th>Pass</th>
<th>Material</th>
<th>Total Thickness</th>
<th>Reduction</th>
<th>Strain</th>
<th>Total Layers</th>
<th>Total Reduction</th>
<th>Strain</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Pass</td>
<td>2 Layers (CuCu)</td>
<td>1 mm</td>
<td>50%</td>
<td>0.8</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>8th Pass</td>
<td>256 layers</td>
<td>1 mm</td>
<td></td>
<td></td>
<td>384 Layer</td>
<td>99.7%</td>
<td></td>
</tr>
<tr>
<td>50% deformation</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>7.2</td>
<td>10.95</td>
</tr>
<tr>
<td>93% Deformation</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>9.22</td>
<td>12.92</td>
</tr>
</tbody>
</table>

**Temperature of ARB** = 300°C

**Annealing @ 600°C**

**Roller Speed** = 32 rpm
Grain Size Distribution of deformed Cu multilayer

- R plot indicator for Log-Normal distribution
- Deviation from Log normal distribution after 50%
- Grain Size > Layer thickness for small fraction of grains

➢ Finer grains → restrict cross slip → Change in deformation texture? Coarser Grains – Un deformed or Recrystallized??
Strain induced grain boundary migration – in the starting material

Rex & grain growth – at higher reductions

How does it affect evolution of deformation texture?
Rolling of monolithic Cu leads to high fraction of S, Cu and Bs texture components
  - For medium & high SFE materials
    - Cu/Bs > 1 at temperatures above RT
  - ARB of Cu/Cu multilayer leads higher Bs component compared to Cu
    - Spread of Cube component along RD

Changes in deformation texture in Cu multilayers
  - layer thickness 100 to 1000 nm
Changes in Deformation Texture

- Higher fraction of \(\{110\}<112>\) Brass component compared to \(\{112\}<111>\) Cu component
  - Large grain Aspect Ratio
  - Effects of stress relaxation \(\rightarrow\) Bs texture development

- Stability of RD-Cube component
  - Relatively weak Cube component
  - Texture transition during deformation

Texture Transition

- Cube \(\rightarrow\) RD-Cube \(\rightarrow\) Goss \(\rightarrow\) BS
Deformed Vs Dy. Recrystallized

<table>
<thead>
<tr>
<th></th>
<th>Deformed (GOS&gt; 3°)</th>
<th>GOS&lt;2°</th>
</tr>
</thead>
<tbody>
<tr>
<td>Starting Material</td>
<td><img src="Deformed_0_45_65.png" alt="Image" /></td>
<td><img src="GOS_0_45_65.png" alt="Image" /></td>
</tr>
<tr>
<td>50% deformed</td>
<td><img src="Deformed_0_45_65.png" alt="Image" /></td>
<td><img src="GOS_0_45_65.png" alt="Image" /></td>
</tr>
<tr>
<td>75% Deformed</td>
<td><img src="Deformed_0_45_65.png" alt="Image" /></td>
<td><img src="GOS_0_45_65.png" alt="Image" /></td>
</tr>
</tbody>
</table>

- Deformed regions $\rightarrow$ RD-Cube & Bs
- Recrystallized regions $\rightarrow$ Cube & Bs

- Combination of CDRX & DDRX
- DDRX up to 75% reduction, beyond which CDRX dominates

Does it hold true during static recrystallization?
Constricted annealing

- Comparable fraction of Cube, S and BR
- Deformation texture components are retained even after static Recrystallization
- Till 50% rolling reduction → Fraction of Cube component increases with prior rolling reduction
- Beyond 50% reduction → Fraction of Cube decreases with prior rolling reduction
- What Changes at 50%???

Samples annealed at 300°C for 30 mins

- Cu
- Bs
- Goss
- S
- Cube
- BR

{111} Pole figure

50%  75%  83%

Annealed Material (in pct.)
Grain Size distribution of annealed Cu multilayer

- Deviation from log-normal distribution
- Saturation in grain growth
- Interfaces restricts grain growth across the layers
- Abnormal grain growth
Restricted & Abnormal Grain Growth

- Grain growth $\rightarrow$ more along the rolling direction and less along the transverse direction

- Presence of thin oxide layer $\rightarrow$ Restricts grain growth across TD

- High stored energy surrounding oxide interface $\rightarrow$ enhance nucleation activity

- Absence of Oxide layer $\rightarrow$ Low nucleation probability $\rightarrow$ enhanced growth conditions
Deformation texture vs annealing texture

With increasing rolling reductions, pole figures of annealed material resemble more of deformed material.
Single phase multilayer

✓ Deformation mechanisms at the submicron level layer are different than conventional coarse grained material

✓ Restoration mechanisms strongly influence the texture of deformed multilayer

✓ Interfaces/layer thickness in homo-phase layers not only influence deformation texture but also the recrystallization texture

➢ Does the Cu layer behave differently in the presence of Fe or Nb layer?
➢ Does the hard phase influence the texture of softer phase or vice versa?
➢ Role of interface in deciding the texture
Effect of Second phase on microstructure and texture – Cu/Fe multilayer

- Shear bands across the layers
- Continuous Cu layer & Fragmented Fe Layer
- Size of Fe segments vary from submicron to 25μm
- Variation of Fe segments not only across the layers but also in between two Cu layers
Changes in Deformation Texture – Cu layer

- Unlike Cu-Cu ARB, volume fraction of Cu is higher than Bs in Cu layer in Cu/Fe
- No RD-Cube component
- Fraction of Cube component is also negligible

➢ Saturation in intensities of texture components after 87%

Twin formation in the deformed Cu layer at 75%
Changes in Deformation Texture – Fe layer

- Strong $\alpha$ and $\gamma$ fibers only after 93% reduction
- Up to 87% deformation moderate texture development
- Large strain partitioning to Cu layer
- Cu acts a lubricant layer $\Rightarrow$ both strong $\alpha$ and $\gamma$ fibers
  - ARB of IF w/o lubrication $\Rightarrow$ Strong $\alpha$ and weak $\gamma$ fiber
  - ARB of IF with lubrication $\Rightarrow$ Strong $\alpha$ and $\gamma$ fibers
XRDLPA of Cu-Fe-Cu Multilayers

![Graph showing crystallite size and deformation percentage as x-axis and dislocation density as y-axis. The graph includes data points for Cu Layer and Fe Layer, indicated by black and red markers respectively. The crystallite size increases with deformation, while dislocation density decreases with deformation.](image-url)
Orientation relationship in deformed Cu/Fe multilayers

<table>
<thead>
<tr>
<th></th>
<th>Starting</th>
<th>25 %</th>
<th>50 %</th>
<th>75 %</th>
<th>87 %</th>
<th>93 %</th>
<th>Intensity</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Cu Layer</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Deformed</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Fe Layer</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

- KS relationship: \( \{111\}_\text{Cu} \parallel \{110\}_\text{Fe} \) and \( \langle 110 \rangle_\text{Cu} \parallel \langle 111 \rangle_\text{Fe} \).
• Very weak Cube and Goss components
• Strong deformation texture components
• Maximum intensity at 87% prior rolling reduction
Orientation relationship in annealed Cu/Fe multilayers

<table>
<thead>
<tr>
<th></th>
<th>Starting</th>
<th>25 %</th>
<th>50 %</th>
<th>75 %</th>
<th>87 %</th>
<th>93 %</th>
<th>Intensity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu Layer</td>
<td><img src="image1" alt="Graph" /></td>
<td><img src="image2" alt="Graph" /></td>
<td><img src="image3" alt="Graph" /></td>
<td><img src="image4" alt="Graph" /></td>
<td><img src="image5" alt="Graph" /></td>
<td><img src="image6" alt="Graph" /></td>
<td><img src="image7" alt="Graph" /></td>
</tr>
<tr>
<td>Fe Layer</td>
<td><img src="image8" alt="Graph" /></td>
<td><img src="image9" alt="Graph" /></td>
<td><img src="image10" alt="Graph" /></td>
<td><img src="image11" alt="Graph" /></td>
<td><img src="image12" alt="Graph" /></td>
<td><img src="image13" alt="Graph" /></td>
<td><img src="image14" alt="Graph" /></td>
</tr>
</tbody>
</table>

KS relationship: $\{100\}_{\text{Cu}} \parallel \{100\}_{\text{Fe}}$ and $<001>_{\text{Cu}} \parallel <001>_{\text{Fe}}$. 
Cube –on- Cube

Bulk texture influence the interface texture
Summary & Conclusions

- In monolithic multilayers (Cu/Cu):
  - Layer thickness plays a crucial role in deciding the deformation and annealing texture.
    - Restoration mechanisms strongly influence the texture of deformed multilayer
  - Interfaces control recrystallization texture → constrained annealing
- In multilayers composites (Cu/Fe):
  - Fe layer restricts DRX in Cu layer.
  - KS Orientation relation is followed in deformed layers for layer thickness up to 100 nm
    - Bulk texture decides the texture of interface
  - In annealed material \( \{100\}_\text{Cu} \parallel \{100\}_\text{Fe} \)
    - Interface decides the texture of Fe layer
DEVELOPMENT OF NEAR ALPHA TITANIUM ALLOYS FOR AIRCRAFT APPLICATIONS

Effect of alloying additions on strength, interface structure, large strain plastic deformation, dwell fatigue
High Temperature Titanium alloys

- Higher operating temperature
  - Increased engine efficiency
  - Need for high performance alloys
Increases the Strength by
- Solid Solution Strengthening
- Precipitation hardening

Modify the Interface to restrict slip across the interphase interface

**Base Material**
Ti-5Al-2Sn-4Zr-2Mo

Thermally stable grain boundary precipitates to reduce grain boundary sliding
Controlling microstructural parameters
Dwell fatigue
Macrozones

- Large regions of primary equiaxed grains with similar orientations
- Macrozones with sharp local texture
  - Basal planes are perpendicular to the loading axis → preferential sites for crack initiation
- Even after large strain hot deformation: primary alpha and beta phases retain Burgers orientation relation

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Al</th>
<th>Sn</th>
<th>Zr</th>
<th>Mo</th>
<th>Si</th>
<th>Ge</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti5242</td>
<td>4.8</td>
<td>2.3</td>
<td>4.2</td>
<td>2.0</td>
<td>-</td>
<td>-</td>
<td>Bal.</td>
</tr>
<tr>
<td>Ti5242Ge</td>
<td>5.0</td>
<td>2.4</td>
<td>4.0</td>
<td>2.1</td>
<td>-</td>
<td>4.1</td>
<td>Bal.</td>
</tr>
<tr>
<td>Ti5242Si</td>
<td>4.9</td>
<td>2.4</td>
<td>4.1</td>
<td>2.1</td>
<td>0.9</td>
<td>-</td>
<td>Bal.</td>
</tr>
<tr>
<td>Ti-Sn</td>
<td>4.8</td>
<td>6.0</td>
<td>3.9</td>
<td>1.9</td>
<td>-</td>
<td>-</td>
<td>Bal.</td>
</tr>
<tr>
<td>Ti-SnGa</td>
<td>5.0</td>
<td>3.3</td>
<td>4.1</td>
<td>1.8</td>
<td>2.7</td>
<td>-</td>
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<tr>
<td>Ti-Ga</td>
<td>4.9</td>
<td>-</td>
<td>4.0</td>
<td>2.1</td>
<td>6.0</td>
<td>-</td>
<td>Bal.</td>
</tr>
</tbody>
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Aluminum Equivalent ~ 7.5
Casting

Forging at 1100°C

Rolling at 950°C

Microstructural Characterization
JSM 7001F and JEM 2100F

Heat treatment
At 950°C for 4hrs + 590°C for 8Hrs
Mechanical Properties of cast samples

- **0.2% Yield stress (MPa)**
  - **RT**: 806, 855, 1114, 798, 1051, 947
  - **650°C**: 274, 338, 445, 213, 637, 381

- **Composition**
  - Base
  - 1Ge
  - 4Ge
  - 0.1Si
  - 0.9Si
  - 1Ge0.1Si
BSE- Microstructures

Base alloy                0.1% Si                1% Si

1% Ge                      4% Ge                      1% Ge 0.1% Si
Effect of Si & Ge on Mechanical properties

With T. Kitashima & Y. Mitarai in MSEA 597 (2014) 212
Study on globularization response

- Alloys deformed to a 70% reduction at 650°C in tensile test
- Ge containing alloys tend to undergo easy globularization
  - attributed to larger dislocation accumulation at the interface

*Mater Sci Forum (2016) Accepted*
Structure of $\alpha/\beta$ interface
Interaction of dislocation with precipitate

**Ti5242Ge**

Dislocations in hot deformed grains

Slip Bands – Planar Slip

Knitting of dislocation

200 nm
Summary and Conclusions

• Addition of Ge and Si to Ti5242 → changes the misorientation distributions
  • Ti5242 alloy maintains the Burgers OR between primary alpha and beta
  • Ge and Si addition probably leads to orientations away from Burgers OR
• Texture of Ge added alloy is significantly different from others
  • Ge: Strong <10-10> || RD fiber and weak <0002>||RD fiber
  • Si and base: Strong <0002>||RD fiber
• After large strain hot deformation
  • Si: Changes the texture in primary alpha from <0002> to <10-10>
• Globularization is faster in Ge added systems
  • Restriction of slip transfer across alpha lamellae
Thank You...