Irradiation study of Ti-6Al-4V and Ti-6Al-4V-1B for FRIB beam dump:

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Outline

• Irradiation of samples with high energy heavy ions (NSCL-MSU)(Ca 40 @ 2000 MeV) and low energy heavy ions at CIMAP-France
• XRD and TEM observations (in collaboration with CIMAP)
• Surface characterization using SEM-EBSD
• Nano-indentation tests
• Vickers Hardness tests
• Insitu-tensile tests
## Irradiation experiments

<table>
<thead>
<tr>
<th>Facilitie s</th>
<th>Beam</th>
<th>Energy [MeV]</th>
<th>Range [µm]</th>
<th>$S_e$ [keV/nm]</th>
<th>Fluence [ions/cm$^2$]</th>
<th>Max dpa in sample</th>
<th>Date</th>
<th>Number of samples</th>
<th>Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>IRRSUD</td>
<td>$^{82}$Kr</td>
<td>25</td>
<td>4.73</td>
<td>9.9</td>
<td>$5.10^{11}$-$5.10^{12}$-$2.10^{14}$</td>
<td>0.6</td>
<td>Jul-13</td>
<td>6</td>
<td>Foils</td>
</tr>
<tr>
<td>IRRSUD</td>
<td>$^{131}$Xe</td>
<td>92</td>
<td>8.5</td>
<td>19.7</td>
<td>$2.10^{11}$</td>
<td>0.001</td>
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<td>2</td>
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<td>6.43</td>
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<td>8</td>
<td>Oct-13</td>
<td>6</td>
<td>Foils</td>
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<tr>
<td>IRRSUD</td>
<td>$^{36}$Ar</td>
<td>36</td>
<td>6.8</td>
<td>7.5</td>
<td>$10^{15}$</td>
<td>1.5</td>
<td>Dec-13</td>
<td>23</td>
<td>TEM and dogbone</td>
</tr>
<tr>
<td>IRRSUD</td>
<td>$^{129}$Xe</td>
<td>92</td>
<td>8.5</td>
<td>19.7</td>
<td>If $3 \cdot 10^{14}$ (~10h)</td>
<td>Estimated 1.7</td>
<td>Planned in June-2014</td>
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<td></td>
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<tr>
<td>NSCL</td>
<td>$^{40}$Ca</td>
<td>2000</td>
<td>800</td>
<td>1.5</td>
<td>$6 \cdot 10^{12}$</td>
<td>$10^{-5}$</td>
<td>Aug-13</td>
<td>1 x Ti64</td>
<td>Dogbone</td>
</tr>
</tbody>
</table>
XRD and TEM observations (in collaboration with CIMAP)

- No evidence of phase transformation or ion track formation in Ti-6Al-4V

**Graphical Elements**
- **Graph 1:**
  - Title: Ti & Ti-6Al-4V
  - Data points: Various Ti phases indicated by temperatures and pressures.

- **Graph 2:**
  - Title: Pressure (GPa)
  - X-axis: Pressure (GPa)
  - Y-axis: Intensity (u.a.)
  - Legend: (002), (101), (112) ± (201), (100), (103), & (213), (114), & ± (212)
  - Xe 92 MeV – 2 \(10^{11}\) ions/cm\(^2\)

- **Graph 3:**
  - Title: TEM image of a Ti-6Al-4V foil irradiated with Kr 45 MeV – 5 \(10^{13}\) ions/cm\(^2\)

- **Graph 4:**
  - Title: Ti-alloys are not sensitive to electronic excitation by swift heavy ions compared to pure Titanium

**Textual Elements**
- TEM image of a Ti-6Al-4V foil irradiated with Kr 45 MeV – 5 \(10^{13}\) ions/cm\(^2\)
- No evidence of phase transformation or ion track formation in Ti-6Al-4V

**References**
- M. Toulemonde et al. / NIMB 277 (212) 28-39
Characterization of the microstructure and mechanical properties:

- Scanning electron microscopy (SEM) as well as electron backscatter diffraction (EBSD) were used to characterize the microstructure of the samples before and after irradiation.
- Nano-indentation, Vickers Hardness and in-situ tensile tests were used to investigate the change in the mechanical properties.
Observations

- Deterioration of the quality of the EBSD scan after irradiation.

Ti-6Al-4V

Ti-6Al-4V-1B

Ti-6Al-4V Irradiated at NSCL: Ca@2000MeV T=20°C and a fluence of 6.10^{12} ions.cm^{-2} and dpa at the surface of 10^{-5}dpa

Ti-6Al-4V-1B Irradiated at CIMAP: Ar@36MeV T=350°C and a fluence of 10^{15} ions/cm and dpa at the surface of 0.038dpa
SEM and EBSD characterization of the surface of the samples:

No change in the microstructure or the orientation of the grains at the surface.

Irradiated at CIMAP: Ar@36MeV
T = 350 °C
Fluence = $10^{15}$ ions.cm$^{-2}$
Dose at the surface = 0.038dpa

SEM image of the EBSD area before (a) and after (b) irradiation for Ti-6Al-4V

IPF and local average misorientation maps the grains before and after irradiation of Ti-6Al-4V

IPF and local average misorientation maps the grains before and after irradiation of Ti-6Al-4V-1B
Local Average misorientation charts
Low energy irradiation: Ar\(36@36\text{MeV}\)
Comparison between Ti-6Al-4V and Ti-6Al-4V-1B only alpha phase

**Ti-6Al-4V**

- Ti-6Al-4V- HT-LF
  - Fluence = \(10^{14}\) ions.cm\(^{-2}\)
  - Temperature = 350\(^0\)C

**Ti-6Al-4V-1B**

- Ti-6Al-4V-1B- HT-LF
  - Fluence = \(10^{14}\) ions.cm\(^{-2}\)
  - Temperature = 350\(^0\)C

- Ti-6Al-4V-1B- HT-HF
  - Fluence = \(10^{15}\) ions.cm\(^{-2}\)
  - Temperature = 350\(^0\)C

Unexpected changes in the local average misorientation for Ti-6Al-4V-1B
Local Average misorientation charts
Comparison between high energy and low energy irradiation

At high energy irradiation, the local average misorientation are more affected in Ti-6Al-4V
Mechanical testing: Nano-indentation

Obtain the properties of the materials in depth.

Parameters:
- Berkovich tip
- Strain rate: 0.05 s\(^{-1}\)
- Poisson ratio=0.33
- Distance between indents: 50µm

In all samples one grip was masked in order to compare irradiated to non-irradiated properties in the same sample.

**SEM image of nanoindents matrix in Ti-6Al-4V irradiated at T= 20°C and a fluence of 10\(^{15}\) ions.cm\(^{-2}\)**

**dpa for Φ=1e15 ion/cm\(^2\)**

Ar 36 @ 36 MeV

In all samples one grip was masked in order to compare irradiated to not irradiated properties in the same sample.
Nano-indentation results

![Graph showing E(GPa) vs Test number for irradiated and non-irradiated samples.]

![Graph showing Hardness (GPa) vs Test number for irradiated and non-irradiated samples.]

No change in hardness was observed
Decrease in the elastic modulus after irradiation

Ti-6Al-4V
Ar36 @36MeV
T= 20 °C
Fluence = $10^{15}$ Ions.cm$^{-2}$
Dose= 0.038dpa
Vickers Hardness tests:

No change in hardness

Ar @36 MeV
In-situ Tensile tests: Preliminary results

Ion beam: Ca 40 @2000MeV
T = 20°C
Fluence = 6.10^{12} ions.cm^{-2}
Max dpa = 10^{-5} dpa

SEM images of the same area (a) before the tensile test and (b) at 13% strain

IPF map, \{0001\} and \{10-10\} pole figures of the α phase in the same area before irradiation
Evolution of the microstructure during the test

Microstructure of irradiated Ti-6Al-4V (a) before the tensile test and (b) at 5.27% strain
In-situ Tensile tests: Preliminary results

Ion beam : Ar@36MeV
T= 350 °C
Fluence = 10^{15} ions.cm^{-2}
Max dpa= 1.5 dpa

SEM images of the same area (a) before the tensile test (b) at 18.7 % strain, (c) failure surface

IPF map, \{0001\} and \{10-10\} pole figures of the α phase in the same area before irradiation

Ti-6Al-4V
RT Tensile test

Stress(MPa)
0 200 400 600 800 1000 1200
Displacement(mm)
0 1 2 3

SEM HV: 25.0 kV
View Field: 127 μm
Det. SE
25 μm

IPF
\{0001\}
\{10-10\}
pole figures of the α phase

max = 10.149

6.000
5.302
3.817
1.000
0.550

0001
2110
0001
1010
Evolution of the microstructure during the test

Microstructure of irradiated Ti-6Al-4V (a) at 6.77% strain and (b) at 9.38% strain
In-situ Tensile tests: Preliminary results
Texture of the tested Ti-6Al-4V

Non-irradiated Ti6Al-4V sample [Li.]

Ion beam: Ca 40 @2000MeV
T = 20°C
Fluence = 6.10^{12} ions.cm^{-2}
Max dpa = 10^{-5} dpa

Ion beam: Ar@36MeV
T = 350°C
Fluence = 10^{15} ions.cm^{-2}
Max dpa = 1.5 dpa

In-situ Tensile tests: Comparison with other Ti64 RT tension Test

For low energy irradiation no change in the mechanical properties (the damage is only on the surface (7 microns).

For high energy irradiation, even at low doses, we observed a significant decrease in the UTS.

More tests are required
Conclusion

• Ongoing analyses:
  • Nano-indentation tests on the cross sections of the samples will allow extraction of hardness and Young modulus for different dpa doses.
  • In-situ tensile tests: Comparison between non irradiated and irradiated Ti-alloys and slip trace analysis

• Future analyses
  • New EBSD analyses planned after electro-polishing the samples
  • Swelling measurements on each samples
  • Possibility to use FIB (Focused Ion Beams) to study the damage in the depth of the sample for TEM, SEM/EBSD analyses
  • In-situ irradiation and creep test