Quality of polyimide foils for nuclear physics applications in relation to a new preparation procedure

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IRMM - Institute for Reference Materials and Measurements
Geel - Belgium
http://irmm.jrc.ec.europa.eu/
http://www.jrc.ec.europa.eu/
Quality of polyimide foils for nuclear physics applications in relation to a new preparation procedure

- IRMM and Target Preparation Laboratory
- Purpose of the work
- Method to prepare polyimide foils
- Standard procedure and modifications
- Mechanical, heat and irradiation tests
- Impurities
- Conclusion
Analytical laboratories

Radionuclide metrology laboratories

Underground laboratory

Two accelerators for neutron production
  - 150 MeV linear electron accelerator (GELINA)
  - 7 MV light-ion Van de Graaff accelerator

Reference materials production and storage

http://irmm.jrc.ec.europa.eu/
http://www.jrc.ec.europa.eu/
vacuum deposition of $^{235}\text{UF}_4$ from a resistance-heated Ta-crucible source
Thin actinide targets by electrodeposition

\[ ^{233}\text{U}, \quad ^{235}\text{U}, \quad ^{240}\text{Pu}, \quad ^{242}\text{Pu} \]

electrodeposition of e.g. \( \text{UO}_2(\text{NO}_3)_2 \)
on metallic backing in isopropanol


intro --- purpose --- method --- modifications in procedure --- tests --- impurities --- conclusion
activity measurement and
distribution of the active material
by low-geometry alpha counting
using a Si surface barrier detector.
Thin deposits of:

- $^6$LiF, nat. LiF
- $^{10}$B
- Au
- tristearin

Characterisation:

- Total mass of deposit by weighing
- Amount of atoms ($^6$Li, nat. Li, $^{10}$B, H) by ?

intro --- purpose --- method --- modifications in procedure --- tests --- impurities --- conclusion
Mechanical transformation techniques:

- **Rolling**
  thin metallic foils: thickness 1.0 – 0.05 mm

- **Wire drawing**
  thin metallic wires: Ø 1.0 – 0.5 mm

- **Punching**
  metallic discs Ø 1 – 100 mm, thickness < 0.5 mm

- **Pressing**
  homogeneous powder compacts
Polyimide foils

areal density 20 - 500 µg·cm²

intro --- purpose --- method --- modifications in procedure --- tests --- impurities --- conclusion
To investigate if polyimide foils

- for nuclear physics applications
- with an areal density around 35 µg·cm⁻²
- prepared in another and faster way than the standard procedure
- have similar or better properties
  - good resistance to temperature
  - good resistance to irradiation with charged particles
  - mechanically strong
1979 Pauwels et al. developed method at IRMM

via amide-acid spreading

in situ polymerisation on glass plates
Preparation method for thin polyimide foils

**preparation of polycondensate solution**

1,2,4,5 - benzenetetracarboxylicdianhydrid
+ 4,4′ - diaminodiphenylether

N,N' – dimethylformamide

**intro --- purpose --- method --- modifications in procedure --- tests --- impurities --- conclusion**
Preparation method for thin polyimide foils

cleaning the glassplates

4h at 100°C
12 min at 350°C

covering the glassplates with the polycondensate solution

intro --- purpose --- method --- modifications in procedure --- tests --- impurities --- conclusion
Preparation method for thin polyimide foils

Transfer of polyimide foil onto frame

intro --- purpose --- method --- modifications in procedure --- tests --- impurities --- conclusion
Characterisation of polyimide foils

thickness measurement with spectrophotometer

reflection mode

transmission mode

intro --- purpose --- method --- modifications in procedure --- tests --- impurities --- conclusion
1979 Pauwels et al. developed method at IRMM via amide-acid spreading and in situ polymerisation on glass plates
4 hours at 100 °C, 25 hours at 230 °C

1981 Van Gestel et al.
4 hours at 100 °C, 30 min at 340 °C → stronger foils

1995 Eykens et al.
dry atmosphere
4 hours at 100 °C, 15 min at 360 °C → stronger foils
## Foil preparation procedures

<table>
<thead>
<tr>
<th>Preparation step</th>
<th>Standard procedure in air</th>
<th>Standard procedure in argon</th>
<th>Fast procedure in air</th>
<th>Fast procedure in argon</th>
</tr>
</thead>
<tbody>
<tr>
<td>polycondensate</td>
<td>fume hood</td>
<td>Ar-glove box</td>
<td>fume hood</td>
<td>Ar-glove box</td>
</tr>
<tr>
<td>spin coating</td>
<td>fume hood</td>
<td>Ar-glove box</td>
<td>fume hood</td>
<td>Ar-glove box</td>
</tr>
<tr>
<td>thermal treatment</td>
<td>oven 4 h at 100 °C</td>
<td>oven 4 h at 100 °C</td>
<td>oven 12 min at 350 °C</td>
<td>oven 12 min 350 °C</td>
</tr>
<tr>
<td></td>
<td>12 min at 350 °C</td>
<td>12 min at 350 °C</td>
<td></td>
<td></td>
</tr>
<tr>
<td>foil transfer</td>
<td>laminar flow</td>
<td>laminar flow</td>
<td>laminar flow</td>
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</tr>
</tbody>
</table>

**Fast procedure** in argon

**Standard procedure** in argon

**Introduction** --- **Purpose** --- **Method** --- **Modifications in Procedure** --- **Tests** --- **Impurities** --- **Conclusion**
Quality tests

- Resistance to heat
- Pressure test
- Dropping test
- Irradiation with charged particles
Resistance to heat

foils
34 µg·cm⁻²
standard in air
fast in argon

20 min 360 °C → 20 min 380 °C → 20 min 400 °C

intro --- purpose --- method --- modifications in procedure --- tests --- impurities --- conclusion
Pressure test

foils
- 20 µg·cm\(^{-2}\) - 80 µg·cm\(^{-2}\)
- standard in air
- standard in argon
- fast in air
- fast in argon

rupture coefficient \( R \) in J·kg\(^{-1}\) of Chen

\[
R = \frac{p \times D}{t}
\]

- \( p \): pressure in Pa
- \( D \): diameter foil in cm
- \( t \): areal density foil in mg·cm\(^{-2}\)
Dropping test

foils
20 µg·cm⁻² - 30 µg·cm⁻²
standard in argon
fast in argon

foils
35 µg·cm⁻² - 55 µg·cm⁻²
standard in air
fast in argon

intro --- purpose --- method --- modifications in procedure --- tests --- impurities --- conclusion
Irradiation

foils
40 µg·cm⁻² - 47 µg·cm⁻²
standard in air
standard in argon
fast in air
fast in argon
on frames with aperture Ø 15 mm

Van de Graaf accelerator of SINS in Warsaw
2.0 MeV single charged ⁴He ions
1.5 MeV protons
beam spot of 4 mm
<table>
<thead>
<tr>
<th>Areal density range $\mu g \cdot cm^{-2}$</th>
<th>20 - 30</th>
<th>35 - 55</th>
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<th>70 - 80</th>
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<td>Air</td>
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<td>Tests</td>
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<td>Fast</td>
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<tr>
<td>Heat resistance temp [°C]</td>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
<td>Pressure R [J·mg$^{-1}$]</td>
<td>0.25(5)</td>
<td>0.15-0.30</td>
<td>0.20(5)</td>
<td>0.17(6)</td>
</tr>
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<td></td>
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<td></td>
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</tr>
<tr>
<td>Dropping height [m]</td>
<td>0.9(3)</td>
<td>1.0(3)</td>
<td>3.7(5)</td>
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<tr>
<td>Irradiation 2.0 MeV $^4$He$^+$ beam intensity [nA]</td>
<td>500</td>
<td>500-900</td>
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<td>500</td>
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<td>deposited beam power [W]</td>
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<tr>
<td>charge [C]</td>
<td>3.0·10$^{-4}$</td>
<td>(4.5-15)·10$^{-4}$</td>
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<tr>
<td>Irradiation 1.5 MeV protons beam intensity [nA]</td>
<td>–</td>
<td>–</td>
<td>400-4500</td>
<td>800</td>
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<td>deposited beam power [W]</td>
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<td>–</td>
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<tr>
<td>charge [C]</td>
<td>–</td>
<td>–</td>
<td>(0.3-2.1)·10$^{-3}$</td>
<td>1.2·10$^{3}$</td>
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Summary
Pressure test

- intro
- purpose
- method
- modifications in procedure
- tests
- impurities
- conclusion
Strength for 20 $\mu$g·cm$^{-2}$ foils


intro --- purpose --- method --- modifications in procedure --- tests --- impurities --- conclusion
### Summary

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<td>500-900</td>
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<tr>
<td></td>
<td>3.0·10⁻⁴</td>
<td>(4.5-15)·10⁻⁴</td>
<td>5·10⁻⁴</td>
<td>4.0·10⁻⁴</td>
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<tr>
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<td>protons</td>
<td>–</td>
<td>–</td>
<td>400-4500</td>
</tr>
<tr>
<td></td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>3.3·10⁻³</td>
</tr>
<tr>
<td></td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>(0.3-2.1)·10⁻³</td>
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</table>
Irradiation

resisted a beam of only 50 nA

resisted a beam up to 4.5 µA

intro --- purpose --- method --- modifications in procedure --- tests --- impurities --- conclusion
Impurities in the polyimide foil by PIXE

intro --- purpose --- method --- modifications in procedure --- tests --- impurities --- conclusion
### Impurities in foil and bulk material

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<th>Element</th>
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<tbody>
<tr>
<td></td>
<td>PIXE - foil</td>
</tr>
<tr>
<td>Ca</td>
<td>(0.40 - 0.90)·10⁻³</td>
</tr>
<tr>
<td>Cl</td>
<td>(0.15 - 0.28)·10⁻³</td>
</tr>
<tr>
<td>Fe</td>
<td>(0.15 - 0.98)·10⁻³</td>
</tr>
<tr>
<td>K</td>
<td>(0.20 - 0.94)·10⁻³</td>
</tr>
<tr>
<td>Mn</td>
<td>(0.03 - 0.30)·10⁻⁶</td>
</tr>
<tr>
<td>S</td>
<td>0.21 - 0.42</td>
</tr>
<tr>
<td>Ti</td>
<td>(0.02 - 0.29)·10⁻³</td>
</tr>
</tbody>
</table>
Conclusion

- fast preparation way in a dry atmosphere
- improved mechanical and thermal strength
- life time under charged particles
- less impurities in the foil → stronger foils
- new procedure:
  fast preparation way in a dry atmosphere
Thank you for your attention!