Recent Achievements in Titanium Proceeded by Equal Channel Angular Pressing (ECAP)

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OUTLINE

• AIT Austrian Institute of Technology
• Equal Channel Angular Pressing
• Properties of ECAP materials
• Conclusions & outlook
AIT Austrian Institute of Technology

Ownership Structure

50.46%  REPUBLIC OF AUSTRIA
(through the Federal Ministry for Transport, Innovation and Technology)

49.54%  FEDERATION OF AUSTRIAN INDUSTRIES

1.370  EMPLOYEES

162.9 m EUR  TOTAL REVENUES
as of YE 2018

87.1 m EUR  contract research revenues (incl. grants)
50.4 m EUR  basic funding
21.3 m EUR  other operating income

M. Krystian, Recent Achievements in Titanium Proceeded by Equal Channel Angular Pressing (ECAP)
Titanium Europe 2019, 15th May 2019, Vienna, Austria
Principle of Equal Channel Angular Pressing (ECAP)

- The metallic material is pressed through a die consisting of two equal channels intersecting at a set angle.
- It undergoes a very high plastic deformation by simple shear under enhanced hydrostatic pressure without change in the cross-sectional dimensions.
- Thus, the ingot can be pressed repetitively through the same die and can also be rotated between consecutive passes (routes) to activate different slip systems and to achieve extremely large, multidimensional strain ($\varepsilon >> 1$).
- These facts lead to a microstructural refinement down to ultrafine-grained (UFG) microstructure and consequently to altered (mainly mechanical) properties i.e. exceptionally high strength at still decent ductility as well as enhanced fatigue limit.
ECAP facility at AIT

- Different dies sets
  - angles of intersection: 120°, 105° and 90°
  - suitable for cylindrical bolts
    - a wide range of diameters: 12mm, 20mm, 30mm & 40mm
  - length up to 120mm (limited by press stroke)
- single and double ECAP tools
- Pressing force up to 700 kN (70 tons)
- Process temperature:
  - die set up: RT to 500°C
  - workpiece: –196°C to 900°C
- Pressing speed: 1 to 20 mm/s
- Manual, semi-automatic and fully-automatic (robotized) modes
- Monitoring of all process parameters
Special designs of ECAP at AIT

- Double ECAP
  - higher degree of deformation per pass
  - higher back-pressure
  - higher strength saturation limit

- ECAP of hollow profiles (tubes)
  - made of titanium

M.Krystian et al., US Patent 2019/0126333 A1

M.Krystian et al., *Equal Channel Angular Pressing (ECAP) of hollow profiles made of titanium*, Materials Science and Engineering, 194 012010 (2017) 1-6
ECAP postprocessing

- Forging
- Plan and profile rolling
- Extrusion
- Rotary swaging
- Heat treatments
Ti-based materials and typical ECAP parameters

• Commercially pure (CP) titanium
  • CP Ti grade 2 (3.7035; ISO 5832-2 grade 2; ASTM B348 grade 2)
  • CP Ti grade 4 (3.7065; ISO 5832-2 grade 4; ASTM B348 grade 4)
    • temperature: RT – 400°C
    • number of passes: 4 – 6
    • die intersection angle: 105° – 120°

• Titanium alloys
  • Ti-6Al-4V grade 5 (3.7164, 3.7164; ISO 5832-3 grade 5; ASTM B348 grade 5)
  • Ti-6Al-4V ELI grade 23 (3.7165.1; ISO 5832-3 grade 23; ASTM B348 grade 23)
  • Ti-6Al-7Nb (TAN; ISO 5832-11; ASTM F1295)
    • temperature: 500°C
    • number of passes: 4 – 6
    • die intersection angle: 120°
Microstructure of ECAP processed materials

- Grain refinement of CP-Ti grade 2 by ECAP
  - Initial coarse-grained microstructure with mean grain size of 50 μm (left, optical microscopy) change to ultra-fine grained structure after ECAP with mean grain size below 500 nm (right, optical microscopy and TEM in insert)
Increase in strength by ECAP: general tendency
Improvement of mechanical properties by ECAP: CP titanium

- CP-Ti grade 4 after ECAP and extrusion surpasses the strength of Ti-6Al-4V ELI alloy
## Improvement of mechanical properties of by ECAP: Ti materials

<table>
<thead>
<tr>
<th>Material</th>
<th>Yield Strength</th>
<th>Ultimate Tensile Strength</th>
<th>Elongation at fracture</th>
<th>Comments</th>
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<tr>
<td></td>
<td>[MPa]</td>
<td>Δ</td>
<td>[MPa]</td>
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<tr>
<td>CP-Ti grade 2</td>
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<tr>
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</table>

- Δ: change compared to conventional state
Improvement of mechanical properties by ECAP: other materials

- Enormous increase in tensile yield strength (YS) of lean silver alloy from 98 MPa to 490 MPa (+392 MPa or +400%) after ECAP
- Remarkable increase in yield strength of as-cast magnesium alloy (EZ33) from 87 MPa to 325 MPa after ECAP (+270%) while retaining ductility
Homogenisation by ECAP

- Colour-coded space-resolved hardness mapping (HV1) of as-received (left) and ECAP-processed (right) CP-Ti grade 2
Homogenisation by ECAP

- Colour-coded space-resolved hardness mapping (HV1) of as-received (left) and ECAP-processed (right) CP-Ti grade 2
Biological properties of ECAP processed materials

- Cell vitality of L929 cells after 24-hour incubation on the surface of as-received (left) and ECAP processed (right) Ti-6Al-4V ELI is significantly higher compared to as-received material.
ECAP related AIT patents

• International patents covering different aspects of ECAP
  • Krystian at al. *Device and method for the plastic forming of a workpiece of metal or a metal alloy by means of ecap*
  
  • Krystian at al. *Method for producing an object from a metal or an alloy by means of large plastic deformation, object produced therefrom, and pressing tool therefor*
    App/Pub No.: PCT/AT2011/050030, WO/2012/071600A1, EP11810543.6A
  
  • Krystian at al. *Tool for forming a metal object under high pressure*
    App/Pub No.: A 50368/2012 513.366, EP13182926.9A, EP2705912A
Conclusions & Outlook

• ECAP processing of titanium results in
  • strong grain refinement
  • homogenisation of the hardness
  • considerable increase in hardness and strength
  • preservation of decent ductility
• Subsequent post-ECAP deformation by a conventional metal forming process further increases strength while maintaining a reasonable ductility
  • Furthermore, the typically cylindrical ECAP bolts can be converted to semi-finished shapes
• The properties of the ultra-fine grained parts can be tuned by heat treatments
• Therefore, commercially-pure titanium processed by ECAP and forging has the potential to overcome the present dilemma related to the limitations of pure titanium in terms of mechanical properties on the one hand, and allergic and toxic concerns of alloying elements in titanium alloys on the other hand

• ECAP materials are going to be used in industrial and medical applications
The Advanced Implant Solutions TEAM

- Bernhard Mingler
- Manfred Bammer
- Laszlo Sajti
- Jelena Horky
- Matthias Schwab
- Wolfgang Wild
- Georg Schleger
THANK YOU!

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