Canless Extrusion Process Development for Blended Elemental Powder-Based Titanium Ti-6AL-4V Alloy

By

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Abstract

The feasibility of canless extrusion in ambient environment of hydride/dehydride blended elemental Ti-6AL-4V ADMA-processed powder previously direct-consolidated by cold isostatic pressing (CIP), followed by vacuum sintering has been successfully demonstrated. Extrusion process trials of these billets were conducted at both RTI International Metals, Inc. and Plymouth Engineered Shapes, Inc. whereby the extrusion processing sequence and parameters were derived separately based on prior extrusion experience at both RTI and Plymouth Engineered Shapes, but were found to be essentially similar to those used for billets prepared from wrought ingot-based Ti-6AL-4V material. Using the results of a workability study program conducted at RTI, the elevated temperature workability tests of powder-based elevated temperature compression specimens showed that powder-based consolidated billets of similar baseline composition as for wrought ingot-based Ti-6AL-4V billets will require slightly lower extrusion pressures at same extrusion temperatures and strain rates. Laboratory analysis showed that the canless powder-based billet extrusion processing step conducted in air added no more than 200 ppm oxygen to the as-vacuum-sintered billet oxygen content. Preliminary tensile properties of the blended-elemental ADMA powder-based extrusions of a Ti-6AL-4V composition processed both in the beta or alpha-beta ranges of extrusion temperatures showed equivalent or superior tensile properties as compared to identically processed wrought, ingot-based and extruded Ti-6AL-4V billet materials. Additionally, in the blended elemental powder-based extrusions both nitrogen and carbon contents were within specification limits for Ti-6AL-4V alloy, while any excessive residual hydrogen was successfully vacuum degassed after extrusion to within specification limits. Further optimization for fracture toughness, stress-corrosion resistance and fatigue properties will build on these encouraging results, while monitoring and controlling the only remaining powder-based interstitial element, namely oxygen uptake during pre-extrusion powder-consolidation processing steps.
Canless Extrusion Process Development for Blended Elemental Powder-Based Titanium Ti-6AL-4V Alloy

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Presentation Outline

• Standard Requirements of Oxygen Content in Ti-6AL-4V Alloy for Ingot-Based and Powder-Based Alloys

• Highlights of Extrusion Trials Performed To Date Using ADMA Blended-Elemental Powder-Consolidated Billets Extruded by RTI International Metals, Inc. and Plymouth Engineered Shapes, Inc.

• General Conclusions and Recommendations
Standard Oxygen Requirements

• **Ingot Products**
  - Standard Ti-6AL-4V Grade
    - Oxygen up to 2000 ppm (0.2 Wt.%) maximum for Ingot-Based Ti alloy
  - Extra Low Interstitial Grade
    - Oxygen 1300 ppm (0.13 Wt%) for Ingot-Based Ti alloy

• **Applicable Specifications for Ingot-Based Products (Multiple Melted)**

• **Metal Powders and Metal Powder Products**
  - Standard Ti-6AL-4V P/M Titanium per ASTM Committee B-817(2003)
    - Oxygen up to 3000 ppm (0.3 Wt.%) maximum for Powder-Based Ti alloys
    - Typical ASTM-Published Tensile Properties in Blended-Elemental Direct-Consolidated Powder Products:
      - Grade II, Type I, Class A [UTS = 131 ksi, Yield = 118 ksi and e% 8 ]
      - Grade II, Type I, Class B [UTS = 139 ksi , Yield = 125 ksi, and e% = 13 ]
Standard Oxygen Requirements (Cont’d)

The Standard for Aerospace Oxygen Content is Based on AMS Specifications Limiting Oxygen Content < 2000 ppm for Extrusions per AMS 4935
In 2007 Zaporozhe Titanium Sponge Plant ZTMK Delivered Two Lots of Titanium Hydride Powder with Low Oxygen Content

- The ZTMK-reported oxygen concentrations in two powder lots used for blended elemental billet preparation for Boeing was 900 ppm and 700 ppm, respectively.

- It is often claimed that oxygen measurement accuracy in titanium hydride is suspect due to high hydrogen activity (35000 ppm see both lot data below).

<table>
<thead>
<tr>
<th>Quality Characteristics of Goods:</th>
<th>Характеристики качества товара:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Main substance (Titanium) content, % weight, no less than</td>
<td>97,00</td>
</tr>
<tr>
<td>Основное вещество (титан) содержание, % вес, не менее</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Chemical composition, %:</th>
<th>Химический состав, %:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lot No.</td>
<td>Hydrogen в водороде</td>
</tr>
<tr>
<td>№ партии</td>
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</tr>
<tr>
<td>1744</td>
<td>3,50</td>
</tr>
<tr>
<td>1</td>
<td>3,88</td>
</tr>
</tbody>
</table>

Lot 1744

Lot 1

Sami M. El-Soudani, PhD Cantab.
Date: April 7, 2008, Updated September, 2008

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Ti-6AL-4V Canless Extrusion in Air Environment
Extruded Boeing–Obtained Results of Chemical Analyses of Extruded Parts Showed Blended Elemental Powder-Based Parts Conforming to Ti-6AL-4V Except for Oxygen Content

Table - Results of chemical analyses* of blended-elemental powder-based titanium Ti-6AL-4V extrusions per 787 Part DWG # BAC 1670-157 extruded by RTI International Metals, Inc. per AMS 4934G and 4935G.

<table>
<thead>
<tr>
<th>Material Product Form</th>
<th>Extrusion Process Temperature/Regime</th>
<th>Sample Position Within Extrusion Along 8’ length</th>
<th>Sample Identification</th>
<th>Chemical Element Weight Percent</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>AL</td>
</tr>
<tr>
<td>Ti Billet CIP/Sintered</td>
<td>As-Sintered</td>
<td>Billet End OD Sample</td>
<td>Billet # 5</td>
<td>6.13</td>
</tr>
<tr>
<td>Extrusions</td>
<td>R1 Extrusion</td>
<td>Front</td>
<td>R1F2</td>
<td>6.36</td>
</tr>
<tr>
<td></td>
<td>R1 Extrusion</td>
<td>Middle</td>
<td>R1M2</td>
<td>6.32</td>
</tr>
<tr>
<td></td>
<td>R1 Extrusion</td>
<td>Back</td>
<td>R1B4</td>
<td>6.30</td>
</tr>
<tr>
<td></td>
<td>R2 Extrusion</td>
<td>Front</td>
<td>R2F2</td>
<td>6.55</td>
</tr>
<tr>
<td></td>
<td>R2 Extrusion</td>
<td>Middle</td>
<td>R2M2</td>
<td>6.52</td>
</tr>
<tr>
<td></td>
<td>R2 Extrusion</td>
<td>Back</td>
<td>R2B4</td>
<td>6.22</td>
</tr>
<tr>
<td></td>
<td>R2 Extrusion</td>
<td>Front</td>
<td>R4F2</td>
<td>5.88</td>
</tr>
<tr>
<td></td>
<td>R2 Extrusion</td>
<td>Middle</td>
<td>R4M2</td>
<td>6.10</td>
</tr>
<tr>
<td></td>
<td>R2 Extrusion</td>
<td>Back</td>
<td>R4B4</td>
<td>6.22</td>
</tr>
<tr>
<td>Ti-6AL-4V AMS 4934</td>
<td>Extrusion</td>
<td>All Min. Max.</td>
<td></td>
<td>5.50</td>
</tr>
</tbody>
</table>

* All samples were analyzed at Durkee Testing Laboratories, Inc. Los Angeles, CA, using emission spectroscopy and inert gas fusion method. The Laboratory Purchase Order Number is 315427 to AMS 4934D and 4935G, and the Lab. Log Number 207616A.

Sami M. El-Soudani, PhD Cantab.
Date: April 7, 2008, Updated September, 2008

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Boeing—Obtained Results of Chemical Analyses of Extruded Parts Showed Blended Elemental Powder-Based Parts Conforming to Ti-6AL-4V Except for Oxygen Content

<table>
<thead>
<tr>
<th>Material Product Form</th>
<th>Extrusion Process Temperature/Regime</th>
<th>Sample Position Within Extrusion Along 8' length</th>
<th>Sample Identification</th>
<th>Chemical Element Weight Percent</th>
<th>Chemical Element Weight Percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti Billet CIP/Sintered</td>
<td>As-Sintered</td>
<td>Billet End OD Sample Billet # 5</td>
<td></td>
<td>AL 6.13</td>
<td>V 4.33</td>
</tr>
<tr>
<td>Extrusions</td>
<td>Beta-Extruded</td>
<td>Front P-1F</td>
<td></td>
<td>AL 6.27</td>
<td>V 4.22</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Middle P-1M</td>
<td></td>
<td>AL 6.49</td>
<td>V 4.09</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Back (Rear End) P-1B</td>
<td></td>
<td>AL 6.24</td>
<td>V 4.31</td>
</tr>
<tr>
<td>Extruded at Beta Transus</td>
<td></td>
<td>Front P-2F</td>
<td></td>
<td>AL 6.23</td>
<td>V 4.28</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Middle P-2M</td>
<td></td>
<td>AL 6.44</td>
<td>V 4.04</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Back (Rear End) P-2B</td>
<td></td>
<td>AL 6.32</td>
<td>V 4.24</td>
</tr>
<tr>
<td>Alpha-Beta-Extruded</td>
<td></td>
<td>Front P-3F</td>
<td></td>
<td>AL 6.20</td>
<td>V 4.15</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Middle P-3M</td>
<td></td>
<td>AL 6.40</td>
<td>V 4.34</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Back (Rear End) P-3B</td>
<td></td>
<td>AL 6.35</td>
<td>V 4.20</td>
</tr>
<tr>
<td>Ti-6AL-4V AMS 4934</td>
<td>Extrusion</td>
<td>All Min. Max.</td>
<td></td>
<td>AL 5.50</td>
<td>V 3.50</td>
</tr>
</tbody>
</table>

* All samples were analyzed at Durkee Testing Laboratories, Inc. Los Angeles, CA, using emission spectroscopy and inert gas fusion method. The Laboratory Purchase Order Numbers are: 315060 & 314986, to AMS 4934D and 4935G. Lab. Log Numbers: 198208A & 196229A.
Boeing—Obtained Results of Tensile Properties in Extruded Parts Showed Powder-Based Properties Superior to Ingot-Based Same Alloy

Table 2 – Preliminary Tensile Property Comparisons Between Powder-based and Ingot-Based Canless Extruded Product Forms Producing BCA 787 Part per Drawing No BAC 1670-157 Extruded Profile for Boeing by RTI International Metals, Inc., Using ADMA’s Blended-Elemental Titanium Hydride/Dehydride Direct-Consolidated Ti-6Al-4V Alloy Powder.

<table>
<thead>
<tr>
<th>Extruded Product Category</th>
<th>ADMA Powder Lot Ident. , or Ingot-Based Heat Number Ident.</th>
<th>Extrusion Process Description (Above, At or Below Beta Transus)</th>
<th>Tensile Yield Strength</th>
<th>Ultimate Tensile Strength</th>
<th>Elongation Percent</th>
<th>Reduction of Area</th>
<th>Preliminary Oxygen Content</th>
<th>Preliminary Hydrogen* Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blended-Elemental ADMA Powder-Based Canless Extrusions</td>
<td>Canless Beta-Extruded at High Temperature “R1” - [Two Test results: R1M2-1 &amp; R1M2-2]</td>
<td>135</td>
<td>159</td>
<td>12.3</td>
<td>25.4</td>
<td>0.35 Average</td>
<td>340 Average</td>
<td></td>
</tr>
<tr>
<td>ADMA Powder Lot # 2 – Same as ZTMK (Ukraine) Powder Lot No 1744 (April 2007)</td>
<td>Canless Beta-Extruded at Lower Temperature “R2” - [Two Test results: R2M2-1 &amp; R2M2-2]</td>
<td>134</td>
<td>157</td>
<td>12.3</td>
<td>28.3</td>
<td>0.29 Average</td>
<td>294 Average</td>
<td></td>
</tr>
<tr>
<td>Ingot-Based Same-Part Extruded Billet at Same Temperature as R2</td>
<td>Canless Beta-Extruded at Same Temperature as “R2” Extrusion - [Two Test results: R4M2-1 &amp; R4M2-2]</td>
<td>127</td>
<td>142</td>
<td>12.4</td>
<td>26.9</td>
<td>0.20 Average</td>
<td>28 Average</td>
<td></td>
</tr>
</tbody>
</table>

* Hydrogen values above specification (125 ppm) have been reduced to within specification limits (prior runs showing values as low as 23 to 33 ppm hydrogen) upon bake-out (or degassing) of hydrogen in vacuum at 1350F for one hour. Hydrogen is therefore a non-issue for blended-elemental hydride/dehydride powder processing.
### Table 1 – Preliminary Tensile Property Comparisons Between Powder-based and Ingot-Based Canless Extruded Product Forms Producing BCA 787 P/N ES-30377 Panel Support Extruded Profiles for Boeing at Plymouth Engineered Shapes, Inc., Using ADMA’s Blended-Elemental Titanium Hydride/Dehydride Direct-Consolidated Ti-6Al-4V Alloy Powder (Program: 2007 Enabling Technology Project No 2.16.1)

<table>
<thead>
<tr>
<th>Extruded Product Category</th>
<th>ADMA Powder Lot Ident. or Ingot-Based Heat Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blended-Elemental Powder-Based Canless Extrusions 787 Part No ES-30377</td>
<td>ADMA Powder Lot # 2 – Same as ZTMK (Ukraine) Powder Lot No 1744 (April 2007)</td>
</tr>
<tr>
<td>Extrusion Process Description (Above, At or Below Beta Transus)</td>
<td>Tensile Yield Strength</td>
</tr>
<tr>
<td>Canless Beta-Extruded “P-1”</td>
<td>139</td>
</tr>
<tr>
<td>Canless Near-Transus Extruded “P-2”</td>
<td>147</td>
</tr>
<tr>
<td>Canless Alpha-Beta Extruded “P-3”</td>
<td>146</td>
</tr>
</tbody>
</table>

| Ingot-Based Same-Part Extruded Billet for 787 Part No ES-30377 | Heat No 03902DB | Heat No 9113898 | Heat No 8716474 |
| Canless Beta-Extruded | 127 | 147 | 15 | 37 | 1900 | 52 |
| Canless Beta-Extruded | 121 | 140 | 11 | 36 | 1520 | 69 |
| Canless Beta-Extruded | 125 | 146 | 15 | 36 | 1800 | 24 |

* Hydrogen values above specification (125 ppm) have been reduced to within specification limits (23 to 33 ppm hydrogen) upon bake-out (or degassing) of hydrogen in vacuum at 1350°F for one hour. Hydrogen is therefore a non-issue for blended-elemental hydride/dehydride powder processing.
Tracking of Oxygen Absorption in Canless Air-Extruded Blended-Elemental Powder-Based Product Forms

- Largest increments of oxygen absorption in the extrusions totaling 1800 to 2000 ppm occurred during two steps: Grinding of sponge fines to H/DH powder and during vacuum sintering (Latter process un-optimized).
- The extrusion process itself added (if anything) one order of magnitude less oxygen of 180-190 ppm.
- Preliminary tensile properties of fully scaled-up powder-based blended-elemental hydride/dehydride extruded products showed tensile properties superior to identically extruded ingot-based product form.
Microstructure of As-Sintered ADMA Blended-Elemental Powder-Based Ti-6Al-4V Microstructure Was Uniform

• As-sintered densification was on the order of 95.5%
The Extrusion Press Operation Parameter Setup, Sequencing, and Control Were Fully Automated

• At RTI four extrusion trials: Three beta extrusions and one alpha-beta extrusion.

• At Plymouth three extrusion trials: One above beta transus, one at beta transus, and one below beta transus.

• At RTI: Three billets were ADMA BE powder-based, and one billet was Ingot-based RTI-supplied Ti-6Al-4V material.

• All trials successful except for the alpha-beta extrusion at RTI which had to be aborted partially due to excessive press force demand.

RTI Control Room for Extrusion Press
Three ADMA-Fabricated Ti-6AL-4V Blended Elemental Powder-Based Billets Were Extruded Successfully by Plymouth Engineered Shapes, Inc.
Two ADMA-Fabricated Ti-6AL-4V Blended Elemental Powder-Based Billets and One Ingot-Based Billet Were Extruded Successfully by RTI International Metals, Inc.

RTI-Extruded Part Drawing No BAC 1670-157 S-40
UPPR STRG: Full Scale Approximately 36 Feet Long
Extruded Profile Configuration was Relatively Complex

Plymouth-Extruded Profile Tooling for a Boeing 787 A/C Part: SPIRIT Shape ES 30377

Thermal Straightening Operation at Plymouth Engineered Shapes, Inc.
Extruded Profile Configuration was for Actual Aircraft Parts

RTI-Extruded Profile for a 787 A/C Part DWG No BAC 1670-157 [S-40 UPPR STRG]

RTI 36’-Long Extrusions Cut-up for Shipping and Laboratory Characterization
Extruded Profile Configuration was for Actual Aircraft Parts

Plymouth extrusions up to 27-foot long were fabricated and analyzed at front, mid-length, and back and found to be uniform with respect to oxygen content & microstructure - same as RTI extrusions. Longer and larger cross-section fabrication is a non-issue in this process.

Plymouth 27’-Long Extrusions Cut-up for Shipping and Laboratory Characterization
A Detailed Workability Study Was Conducted To Establish Temperature and Strain Rate Sensitivities and to Define Optimum Extrusion Processing Parameters

- Compression testing at different strain rates covered a wide range of temperatures 1200F to 2200F
- True strain rates from 1/sec to 100/sec
- Up to a total true strain limit of 1.33
- Both ADMA blended-elemental powder-based and ingot-based Ti-6AL-4V microstructures were tested and extruded at same temperatures
- As-extruded densification was upwards of 99% approaching theoretical density, but some small gaseous porosity remained with some pores elongated under compression loading
Workability Study Revealed that Powder-Based Extrusion Processing Would Require Some 20% Lower Press Pressures as Compared to Identically Processed Ingot-Based Product Form

ADMA Blended-Elemental Powder-Based Sintered Billet Ti-6AL-4V Material

RMI-Supplied Ingot-Based Ti-6AL-4V Billet Material
Workability Study Showed That Blended Elemental Titanium Powder Billet is Less Demanding on Press Forces Than Ingot-Based Billet, But That a Broad Range of Extrusion Ratios Can be Accommodated

- Generally a lower stress was required for powder-based product form compared to ingot-based same Ti-6Al-4V at same extrusion temperature

- Total Strain: Analog of extrusion ratio at a given temperature

- Strain Rate Sensitivity: Analog of press force demand at same temperature and extrusion ratio
Microstructure of Beta-Extruded ADMA Powder-Based Ti-6AL-4V Alloy

- RTI-extruded transformed beta basket weave microstructure approached theoretical density with alpha-delineated prior beta grain boundaries.
Microstructure of Beta-Extruded Powder-Based Ti-6AL-4V Alloy

- Plymouth-Extruded transformed beta basket weave microstructure approached theoretical density with alpha-delineated prior beta grain boundaries.
Microstructure of Beta Extruded RTI-Supplied Ingot-Based Ti-6AL-4V Alloy

- RTI-extruded transformed beta basket weave microstructure of 100% theoretical density with some alpha-delineated prior beta grain boundaries. Prior beta grains are coarser in ingot-based extrusion as compared to ADMA powder-based extruded billet.
Microstructure of Alpha-Beta Extruded ADMA Powder-Based Ti-6AL-4V Alloy

- RTI-alpha-beta extruded mixture of alpha and transformed beta microstructure interspersed with some residual gaseous voids. Extrusion approached theoretical density upwards of 99%, but the extrusion run was partially aborted due to high press force demand.
Microstructure of Alpha-Beta Extruded Powder-Based Ti-6AL-4V Alloy

- Plymouth-alpha-beta extruded microstructure:
  A mixture of transformed beta basket-weave microstructure interspersed with equiaxed alpha phase approaching theoretical density.
General Conclusions on Canless Air-Extruded Product Form

• Canless full-scale extrusion of blended elemental hydride/dehydride ADMA powder-based product form has been successfully demonstrated.

• But there is a need for further optimization of certain steps for better control and for lower oxygen content, namely at vacuum sintering, sponge fines milling, and initial sponge further oxygen content reduction (ZTMK).

• Any residual hydrogen content within the final extruded parts was thermally degassed in vacuum down to 23-33 ppm.

• Preliminary encouraging powder-based extruded product form shows tensile properties superior to ingot-based identically extruded product form.
General Conclusions on Canless Air-Extruded Product Form (Cont’d)

Extrusions fabricated with blended elemental ADMA Ti-6Al-4V powder up to 36-foot long were analyzed at front, mid-length, and back and found to be uniform with respect to oxygen content and microstructure. Further extrusion product form scale-up is a non-issue.

Within the cross section some areas near free surfaces exhibited somewhat finer grain size compared to the bulk microstructure but no alpha case anywhere.
Recommendations for Follow-On Development

• More elaborate material characterization testing for optimization of the emerging low-cost titanium Ti-6AL-4V powder-based product forms for acceptable aerospace product is needed.

  – Assuming that the powder-based direct-consolidated products may have a different optimum content for best property balance, relative to identical ingot-based product form this is best established by actual testing.

• Optimization of grinding of sponge fines and sintering process for reduced oxygen uptake within inert and/or vacuum environments is necessary.