MELTLESS Ti FOR AEROSPACE APPLICATIONS

Andy Woodfield, Eric Ott, Jon Blank, Mike Peretti, David Linger and Larry Duke

GE Aviation, 1 Neumann Way, Cincinnati, OH, 45215, USA

Abstract

Recent advances in synthesis of titanium (Ti) alloy powders by direct reduction methods (meltless Ti) have led to new opportunities to produce Ti alloys with enhanced capabilities. The meltless Ti alloy powders can be consolidated into mill products, or used to manufacture near-net shape (NNS) components. A vision is outlined for creation of a new supply chain producing meltless Ti alloy powders, leading to the production and application of innovative turbine engine components. This paper highlights the benefits of meltless Ti alloys, details current assessment of meltless Ti alloy technology, and outlines some of the challenges to fully establish the new supply chain for aerospace applications.

Introduction

Since the beginning of Ti alloy production in the late 1940’s, Ti sponge has been produced by either the Kroll (magnesium reduction) or Hunter (sodium reduction) processes [1]. Alloying has been accomplished by blending the sponge with elemental and/or master alloys and the use of multiple melt steps. Over time, numerous other Ti reduction processes have been researched, however, none of these alternative approaches have been adopted in production for significant quantities of Ti [1]. In the last 10 years, numerous new Ti reduction processes have been demonstrated [2], with multiple processes being scaled at least to the pilot scale. These processes typically produce spongy powders, as opposed to the current reduction processes that produce a sponge cake. Another attribute that some of the new processes have is the potential to form alloys directly during reduction, leading to the possibility of clean, directly reduced, uniformly alloyed powder suitable for consolidation into mill products and NNS articles without ever melting during powder formation or consolidation, i.e. “meltless Ti”. Since no melting is involved, it is possible that new, novel Ti alloy compositions and microstructures can be developed that could not have been produced using current technology. It is clear that this new, meltless Ti technology will likely come from outside the current Ti industry [2], due at least in part to the substantial re-investments in existing technology recently described by the Ti industry [3].

Meltless Ti Industry Requirements

For a meltless Ti industry to evolve, several partially interrelated meltless Ti technology requirements need to be met, as outlined below:

1. Lower cost basis than current technology. The technology must compete successfully with existing materials, including coupling the new reduction with highly efficient consolidation processes for the spongy powders. This may drive application of the technology toward higher value alloys versus commercial purity (CP) Ti, higher value mill products such as bar, sheet and tube versus billet and plate, and higher value NNS articles. Like conventional Ti production, a significant fraction of the cost basis for the new technology is estimated to come from its energy consumption; however, a detailed process model for energy intensity per lb of meltless Ti-64 bar, indicates an advantage of 50% less energy usage versus the conventional process.
(2) Greener process than current Kroll plus melt-based technology including efficient recycling of byproducts. In the same process model referred to above, the lbs of generated CO₂ per lb of meltless Ti-64 bar indicate an advantage of 50% less CO₂ versus the conventional process.

(3) Complete separation of byproducts from powder to avoid the presence of intrinsic defects, such as residual salt or reductant metal, in the consolidated material.

(4) Management of intrinsic and extrinsic defects, such as air-borne contaminants or process equipment materials of construction contaminants, in consolidated material. An understanding of what size/types of defects can be seen during inspection, and the impact of these defects on mechanical properties is necessary.

(5) Development of industry-accepted specifications to promote widespread use.

(6) Significantly lower price point than current technology to ensure sufficient driving force for adoption of the new technology.

**Meltless Ti Demonstration**

GE Aviation has been involved in Meltless Ti technology since 2001, and had the opportunity to collaborate and investigate numerous aspects of multiple technologies. This included meltless Ti alloy reduction, separation of powder products, powder morphology effects related to consolidation, inspectability/impact of defects in mill products, and demonstration of microstructures and properties of consolidated mill products and finished articles from current alloys such as Ti-64 and novel alloys that cannot be produced by ingot metallurgy approaches. Two meltless Ti demonstrations of mill product are highlighted below.

**Meltless Ti-64 Mill Product and Component Demonstration**

Meltless Ti-64 powder was uniaxially pressed, loaded into an extrusion can and evacuated, hot isostatic pressed (HIP) using conventional Ti-64 process conditions, extruded into 1.75 cm diameter bar at elevated temperature with a ~9:1 extrusion ratio, and forged and machined into turbine engine compressor vanes as shown in Figure 1. Tensile testing of this bar material showed a 0.2% yield strength of 980 MPa and an ultimate tensile strength of 1050 MPa. Plastic elongation to failure was measured at about 5%, but the low ductility was related to the presence of small, ~50 μm-sized defects, introduced during powder transfer to the uniaxial compact press. After modifying the handling procedures, and producing additional bar material, plastic elongations in the 10-12% range were obtained.

Figure 1. Manufacturing sequence for meltless Ti-64 turbine engine vanes from (a) cold compacted pucks, (b) HIP can, (c) extruded bar, (d) forging, and (e) finished vane airfoil.

Figure 2 shows the ultrafine primary alpha grain size (approximately 3 μm) in the above bar after heat treatment at 955°C for 1 hour, air cool, followed by 705°C for 2 hours. This compares to a typical 5 μm primary alpha grain size in similar diameter, conventionally produced bar. (Note, for comparison, production Ti-64 billet would have a primary alpha grain size of ~15 μm.) Chemistry results from the heat treated meltless Ti-64 bar are also included in Figure 2. Although these chemistry results show Al and V to be slightly above the
AMS 4928 specification range, oxygen and other interstitial elements are within specification, and the residual levels (reductant metal and byproducts) are at very low levels. Also included in Figure 2 is a digital C-scan ultrasonic inspection image comparison with conventional Ti billet using identical sample sizes and inspection conditions. The inspection data clearly reveal the handling defects (~50μm in size, identified by arrows) against the almost zero background noise levels. The background noise level in conventional billet is sufficiently high such that if the same defects were present, they would likely not be detectable due to the higher background noise attributable to the microstructure.

The above results demonstrate the potential for manufacturing a meltless Ti-64 turbine engine component having a highly refined microstructure and high degree of inspectability.

**Meltless Ti Novel Alloy Demonstration**

The meltless Ti reduction process enables the possibility of novel alloys that cannot be produced with current melt-based technology. For example, boron additions to Ti alloys have been demonstrated to increase strength and moduli significantly [4], however, use of melt-based technology (either ingot or gas atomized powder) or blended elemental technology results in greater than 1μm TiB particles leading to detrimental effects on fatigue crack initiation. A meltless Ti-B alloy extruded bar was produced to validate that the meltless introduction of B would yield a much finer, more uniform distribution of TiB particles. Figure 3a shows a TEM micrograph from a meltless Ti-0.9 weight percent B alloy, with an ultrafine distribution of significantly less than 1 μm TiB particles in an approximately 2 μm primary alpha grain size matrix. Tensile data from this material, Figure 3b, showed a 0.2% yield strength of 1035 MPa, an ultimate tensile strength of 1170 MPa and a plastic elongation to failure of 7 percent, while the modulus was about 117 GPa. High-cycle fatigue (HCF) data obtained from this material is shown in Figure 3c, where it is compared to identically tested AMS 4921 Grade 4 CP Ti bar, and literature Ti-64 data [5]. The data show meltless Ti-0.9B material has a runout stress (10^7 cycles) 275 MPa greater than CP Ti, and 240 MPa greater than Ti-64 bar. This very significant increase in HCF strength for meltless Ti-0.9B is due primarily to a combination of the very fine primary alpha grain size and the presence of an ultrafine TiB dispersion in the matrix. The results described above demonstrate novel meltless Ti alloys can be produced, and that the periodic table should be re-examined for new alloying elements (or higher levels of alloy elements) that can be potentially added to meltless Ti alloys without the detrimental constraints of melting on alloying.
As outlined earlier, there is promise of a new meltless Ti industry with alloys produced at lower cost by more environmentally friendly processes, and using new alloys with improved properties. In order for this new industry to materialize and thrive, it will need to differentiate itself from current technology with a lower cost basis than traditional; as mentioned earlier, this will be easier in higher value mill products, such as alloy tube, sheet, and bar. Significant effort is currently being applied to different meltless Ti reduction technologies [1,2], however, similar emphasis needs to be placed on the development of highly efficient consolidation of the typically spongy alloy powders to produce mill products and NNS articles. For example, Figure 4 outlines two potential methods to efficiently produce a meltless Ti bar from spongy powder. The first approach consists of cold pressing the powder into a continuous preform, Figure 4a (re-drawn from [6]), canning and evacuating the preform, and hot extrusion, Figure 4b. The second approach includes continuously rolling the powder into ‘green’ bar (illustrated in Figure 4c) or sheet, cutting to length and stacking, canning and evacuating, and subsequent hot extrusion, Figure 4d.

Figure 4. Efficient consolidation approaches for meltless Ti alloy bar manufacture.

The emergence of meltless Ti is envisioned to lead to a significant expansion in the overall Ti market including in the aerospace industry. Meltless Ti reduction technology development is proceeding, however, the industry also needs to develop highly efficient methods for consolidation of the spongy meltless Ti powders into mill products and NNS articles. Several potential approaches for efficient consolidation to bar were suggested. Finally, it has been demonstrated that revolutionary, new meltless Ti alloys can be developed with extended alloy capabilities enabled by the elimination of melting.

References


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Outline

Benefits / Requirements
Powder Reduction / Consolidation
Meltless Ti-64
Meltless Novel Alloy
Component Demonstration
Opportunities
Meltless Ti Process Map

Today: Kroll (Melt)
- Kroll Reduction Ti Sponge
- AIV Alloy, Scrap, TiO2
- Melt: 2X VAR or HM+VAR
- Convert to Mill Product
- Ti64 Melted Billet

Tomorrow: Meltless Ti
- Consolidation
- Convert to Mill Product
- Ti64 Meltless Billet
- Ti64 Meltless NNS Part
  - > 50% Lower Part Cost

New Approach… Less Steps & Higher Yield

Pigment Industry
Less than 10% of TiCl₄ produced used for Ti Metal

New Technology
- Near net shape (NNS) to part

Ore TiO₂
Chlorination TiCl₄
Environmental Benefits

New Approach… >50% Less Energy / Emissions

• >50% ↓ energy
• >50% ↓ CO₂ & CO
• >50% ↓ SO₂ & NOₓ
Meltless Ti … Aerospace Capable?

Powder handling
Defects
Chemistry Control
Reduction Process vs Alloy Capability
Properties
Weldability
Price
Ti Reduction Patent Activity

Major Patent Activity … Last 10 years
Meltless Ti Processes

Meltless Ti Processes ... Global
Powder Reduction Status

Technology Entitlement

Many Processes ... 1 Nearing Commercialization
Meltless Ti Powder Morphology

Powder forms and Geometries

**Porous poor packing**
- sponge
- dendritic
- irregular
- ligamental

**Solid poor packing**
- angular
- tear drops
- flakes
- acicular
- aggregates
- rounded

**Solid well packing**
- cubes
- polygons
- spheres
- cylinders

packing density ~4% to ~65%

Meltless Powders … Challenge for Consolidation

*ref. R. German*
Consolidation of Meltless Ti Powder

Uniaxial Pre-consolidation Behavior

Oxygen Pickup during Consolidation

Pre-Consolidation Issues … Loads and Oxygen
# Meltless Ti-64 Demonstration

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<th>Element</th>
<th>#1 (wt%)</th>
<th>#2 (wt%)</th>
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<tbody>
<tr>
<td>Aluminum</td>
<td>6.96</td>
<td>6.96</td>
<td>5.5 – 6.75</td>
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<tr>
<td>Vanadium</td>
<td>4.61</td>
<td>4.63</td>
<td>3.5 - 4.5</td>
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<tr>
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<td>0.195</td>
<td>0.196</td>
<td>&lt;0.2</td>
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<tr>
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<td>0.004</td>
<td>0.003</td>
<td>&lt;0.05</td>
</tr>
<tr>
<td>Carbon</td>
<td>0.016</td>
<td>0.017</td>
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<td>Hydrogen</td>
<td>0.0035</td>
<td>0.0030</td>
<td>&lt;0.0125</td>
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<tr>
<td>Iron</td>
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<tr>
<td>By-Products</td>
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<td>&lt;0.001</td>
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Early Run … Close to AMS4928
Structure / Inspectability

As-extruded

1750F/1h AC

Meltless Ti-64 Bar

Ti-64 Billet

Fine Grain Size … Excellent Inspectability
Properties

Lower Ductility ... f(Extrinsic Defects)
# Meltless Novel Ti-B Alloy

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<tr>
<th>Element</th>
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<tr>
<td>Boron</td>
<td>0.89</td>
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<tr>
<td>Oxygen</td>
<td>0.645</td>
<td>&lt;0.4</td>
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<tr>
<td>Nitrogen</td>
<td>0.019</td>
<td>&lt;0.05</td>
</tr>
<tr>
<td>By-Products</td>
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<td>N/A</td>
</tr>
</tbody>
</table>

Fine Powder ... High Oxygen
Structure

Alpha-Ti hcp; \(a = 2.95\text{Å}, \ c = 4.69\text{Å}\);
TiB: Orthorhombic; \(a = 6.12\text{Å}, \ b = 3.06\text{Å}, \ c = 4.56\text{Å}\)

OR: \([11-20] \alpha \parallel [010] \text{TiB}, (0001) \alpha \parallel (001) \text{TiB}, (-1100) \alpha \parallel (100) \text{TiB}\)

Long edges of TiB plates \(\parallel (100) \parallel (-1100) \alpha\)

Ultrafine TiB … Coherent with Matrix
Excellent Strength/Fatigue … Despite High O
Consolidation / Forging

Press Round Pucks → Can Pucks + HIP → Hot Extrude → Bar Machine → Vane Forge → Vane Machine

Component Demo … No Manufacturing Issues
Implementation Needs

Additional needs

– Convert powder to mill products / NNS
  • Cleanliness
  • Economics

– Example bar consolidation route

Development Needs … Clean, Efficient Consolidation
Summary

Meltless Ti Alloy Powders Demonstrated

Supply Chain Developing

Consolidation Development Needs