Final CRADA Report for

CRADA No. ORNL05-0702

with

a Heavy Vehicle Component Manufacturer

Alloy Design and Thermomechanical Processing of a Beta Titanium Alloy for a Heavy Vehicle Application

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Abstract

With the strength of steel, but at half the weight, titanium has the potential to offer significant benefits in the weight reduction of heavy vehicle components while possibly improving performance. However, the cost of conventional titanium fabrication is a major barrier in implementation. New reduction technologies are now available that have the potential to create a paradigm shift in the way the United States uses titanium, and the economics associated with fabrication of titanium components. This CRADA project evaluated the potential to develop a heavy vehicle component from titanium powders. The project included alloy design, development of manufacturing practices, and modeling the economics associated with the new component. New Beta alloys were designed for this project to provide the required mechanical specifications while utilizing the benefits of the new fabrication approach. Manufacturing procedures were developed specific to the heavy vehicle component. Ageing and thermal treatment optimization was performed to provide the desired microstructures. The CRADA partner established fabrication practices and targeted capital investment required for fabricating the component out of titanium. Though initial results were promising, the full project was not executed due to termination of the effort by the CRADA partner and economic trends observed in the heavy vehicle market.

Introduction

The aircraft industry is currently the single largest market for Ti and Ti alloy products primarily because of the exceptional strength-to-weight ratio, elevated temperature performance, and corrosion resistance. The strong dependence of titanium on the aerospace industry has caused titanium production to be very cyclic. Titanium alloy plate costs have been as high as $40-50/lb, sheet costs have been as high as $100/lb, and lead times for product delivery from time of order can be as long as 18 months. Titanium usage outside the aerospace industry has strongly been limited by its higher cost relative to competing materials, primarily aluminum alloys and steels. Although titanium possesses an attractive set of properties; including high specific strength, corrosion resistance, damage tolerance, low Young’s modulus, high strength to weight ratio, and biocompatibility; cost limits applications to selected markets. Many automotive systems would benefit from the use of Ti products. Automotive exhaust systems could save as much as 50% of their current weight by integrating Ti parts [i]. Titanium valves and valve springs, connecting rods, suspension springs, wheels, drive shafts, underbody panels, side impact bars, and half shafts are just some of the automotive applications that could benefit from the use of Ti [ii, iii, iv].
A 2005 market comparison for the use of engineering materials showed that steel is the most widely used material, with 800 million tons used each year [v]. Aluminum, stainless steel, and copper are used at a rate of 22 million tons, 16 million tons, and 12 million tons, respectively. Titanium is much less widely used at 0.05 million tons per year. Government studies have identified that titanium enjoys only 1/20th of its potential usage [vi].

Technical problems preventing the integration of Ti, apart from cost of manufacturing, include wear resistance, a lower modulus than steel, and machining difficulties (titanium machines at 1/10th the speed of aluminum). Wear resistance can be addressed by coatings, reinforcements, and compositing as has been shown in preliminary work performed by Blau et al. While low modulus is a benefit in some applications, when required, it can be increased by reinforcing the part via compositing or with a second material. Machining difficulties can be reduced by the production of near-net-shape parts. The major problem not addressed by these process modifications is that titanium components cost substantially more than competing materials.

The high cost of sheet, plate and most other Ti and Ti alloy product forms is a result of the elaborate reduction process, the multiple melting steps, the secondary processing, and the machining to final component. For example, processing of Ti from ingot to 1” plate accounts for 47% of the total cost of the material [vii]. Thinner plate and sheet gauge sections increase the production costs, the amount of production waste, and the percentage of total production costs.

Current melting techniques include vacuum arc remelting (VAR), electron beam melting, and plasma arc single-melt processes. VAR is used to consolidate feedstocks, improve homogeneity and refine grain size. To VAR-cast an ingot, a direct current arc is struck between a Ti electrode and a base plate. The intense heat melts the electrode, and the Ti is cast into an ingot. VAR requires 20 hours and large-scale equipment.

Electron beam and plasma arc single-melt processes have the advantage over VAR in increased use of low cost scrap and improved removal of high-density and low-density inclusions. In these two processes, ingots are cast directly from melted feed sponge, alloy additions and scrap. But the cast ingot still requires post-processing, which accounts for approximately half of the total cost.

Although much time and effort has been invested in the development of these processing techniques, the cost of Ti still remains high because of the cost of raw materials, cost of post-processing, and yield losses. In an ongoing effort to reduce the cost of Ti and Ti alloy production, new solid state and melt technologies have been investigated by ORNL in producing plate, sheet, and rod. Vacuum Hot Pressing (VHP), high-density infrared (HDI) processing, roll compaction, and extrusions have been explored at ORNL. These methods have used DARPA Titanium Initiative “low cost” powders produced by the Armstrong method and provided by
International Titanium Powder (ITP). ITP has produced CP Ti and Ti alloys out of their pilot processing facility, and has initiated the development of a full scale production plant to produce CP Ti and Ti alloy powders. Consolidation studies are required to produce consolidated components, parts, and material.

**Objective**

The objective of this CRADA project was to develop a “low cost” Beta titanium alloy for weight reduction of a heavy vehicle component while utilizing titanium powder produced by new reduction technologies. The CRADA team was to evaluate if a heavy vehicle component could be fabricated from the new titanium powders, and if the economics and weight reduction were justified for total life cycle of the component. The CRADA partner currently produces the targeted component out of steel. ORNL is collaborating with the company to determine a new alloy composition, lower in price than current aerospace Ti alloy compositions, and that is geared towards the mechanical properties of the component.

**Approach**

The project was divided into four major tasks: Task 1: Component Design (performed by the CRADA partner); Task 2: Economical Modeling (performed by EHK Technologies with input from ORNL and the CRADA partner); Task 3, Alloy Design and Verification (performed by the full team); and Task 4: Process Development (performed by ORNL and the CRADA partner).

In component design, the CRADA partner developed the new geometry and specifications required to make the heavy vehicle component out of titanium. The effort included the development of new tooling and equipment required to fabricate and optimize the new titanium component. Items such as spring back, percent cold working, shot peening, and other engineering technical questions were evaluated as a result of this effort.

The economic model was constructed within the first 18 months of the project, and was to be modified as results were developed and the process was optimized. Due to the early termination of the project and the downsizing of the CRADA partner, the economic model was not fully utilized to determine the economics of the new component.

Alloy design was performed in two segments to improve efficiency of the project budget. ORNL initiated the project with casting the new series of Beta titanium alloys to evaluate mechanical and microstructural characteristics while establishing solution treatment protocol for the new alloys. The second subtask involved the blending of elemental powders with the new titanium powders, and evaluating the benefits and challenges of using a powder metallurgy approach. This two pronged approach had the added benefit of providing an alternative method of fabrication if economic or technical challenges could not be surmounted in one technology. Oak Ridge National Laboratory investigated the blending procedures for elemental or master alloy additions and then developed the process for extruding the powders into flat stock/rod as a preform for a vehicle component; alloy design was to take into consideration elements that may be able to be directly synthesized and alloyed in the new titanium reduction technologies, such as the Armstrong Process by International Titanium Powder, LLC (ITP).
In Task 4, ORNL evaluated homogenization (specifically for solid state processing), and then developed the consolidation processing parameters for solution treating, rolling, and ageing of the new titanium alloy. The CRADA team then evaluated microstructure, mechanical behavior, and chemistry of arc-melt and drop castings of the new alloys to compare with existing, expensive Beta titanium compositions. ORNL then demonstrated the ability to extrude rod using powder Ti precursor of the new alloy, and compared the data with extrusions of commercially pure titanium. The project was cut short before the heavy weight vehicle component could be fabricated from powder, but Beta Ti alloy components fabricated from conventional melt technologies was fabricated for the first time as a result of this project. The CRADA partner witnessed the value added by potentially replacing the steel component with a titanium alloy component if production costs could be minimized. The technology was not executed due to the economic conditions.

**Experimental Procedure**

The present scope of work was performed within four tasks as outlined in the approach. Each task was designed with the ultimate goal of producing a Ti alloy component that reduced the weight of a heavy truck component. For ease of discussion, the Experimental Procedure and the Results will be subdivided into the four tasks.

**Task 1: Component Design**

The identity of the component selected for this CRADA is proprietary. However, certain mechanical/structural considerations had to be taken into account to redesign the component so that a titanium alloy could be successfully implemented. Component design was performed by the CRADA partner with ORNL providing information on the potential processing routes and thermo-mechanical history that could influence the resulting product form and component design. Reports by the fabricator were provided to ORNL and kept on record.

**Task 2: Economic Model**

A preliminary economic model was constructed based on a “Greenfield” production approach. Specific economics that were taken into account include: labor, price of the titanium powder, price of elemental alloying powders, thermo-mechanical processing costs, machining, capital costs, and post ageing processing. The model was modified as the processing development matured. The economic model was not completed due to the abrupt ending of the project with the manufacturer.

**Task 3: Alloy Design and Verification**

The selected component would best utilize an alloy with the Beta (body centered cubic) crystal structure. Beta alloys are normally composed of Beta grains with precipitates of the Alpha
(hexagonal close packed) phase within the Beta grains and at grain boundaries. The application
of the selected component will operate at ambient temperatures, so there is no need to provide
high temperature strength, creep resistance or enhanced oxidation resistance, but Ti corrosion
resistance is a benefit. One priority objective of the alloy design is the achievement of a
comparable “low cost” alloy. Many of the conventional, commercial alloys contain significant
levels of expensive alloy additions. Since the CRADA project utilizes powder processing rather
than melt metallurgy, issues of melt segregation of elements, such as iron, are not of concern.
Therefore, lower cost Beta stabilizing elements can be used in the design of the composition for
the heavy vehicle component.

A method of alloy design was then utilized which takes advantage of established tendencies of
various elements to stabilize the Alpha or Beta phases in comparison to the benchmark elements
Mo (Beta stabilizer) and Al (Alpha stabilizer); these tendencies are known as the Mo Equivalence or Al Equivalence of each element. A Beta stability index for an alloy is then calculated as the sum of the Mo Equivalences of all it’s Beta stabilizing constituents, minus the sum of the Al Equivalences of all it’s Alpha stabilizing constituents; this is the Mo – Al Equivalence. This index was calculated for many commercial Beta alloys, and for numerous candidate compositions which would contain low levels of expensive elements, and could utilize inexpensive master alloy powders.

One conventional composition and two lower cost compositions were then selected for further
investigation. Transformation diagrams were determined by computer simulation for these three
compositions. The conventional composition and two new beta Ti compositions were then arc
melt and drop cast to provide material for preliminary processing. The intense heat of the arc
was used to melt the titanium and other constituent elements of the alloy. Elements were
thoroughly mixed in a copper crucible, and flipped four to five times in between arc melting
operations to improve homogeneity. Once mixed, the alloy button was cast in a water cooled ½”
x ½” x 3”copper mold. Chemical analysis was performed on the Ti alloy ingots; inert gas fusion
was used to determine oxygen and nitrogen weight percentages (ASTM E 1409-05) and metal
elements were analyzed using direct current plasma emission spectroscopy (ASTM E 1097-03).
Micrographs were made of the as-cast microstructure. Small samples (20 to 50 mg) were cut
from the castings to perform differential thermal analysis (DTA) measurements using a SDT Q600 from TA Instruments. A heating rate of 20°C/min was used, and the sample was allowed
to go a full cycle. A CP Ti sample cast from low cost ITP titanium was analyzed for
comparison. DTA analysis was performed to identify the beta transus.

Task 4: Process Development
A solid state or powder metallurgy (PM) processing technique that takes advantage of the new
low cost titanium powders was the objective. The blending of elemental or master alloy powders
to form the desired alloy, extrusion parameters and methodology, solution treating temperatures
and time intervals, rolling conditions, ageing temperature and time intervals, and post ageing
conditioning were all thermo-mechanical variables that were to be developed and identified for
the realization of the heavy vehicle titanium alloy component. However, identifying these
parameters on extruded samples of blended powders initially would be costly and time
prohibitive. Therefore, solution treating (ST), cold rolling (CR), and ageing (A) processes were
performed on drop castings to evaluate the candidate alloys in the required processed state since
cast microstructures will not provide the necessary mechanical properties for the application. Once an alloy composition exhibited the required microstructures, economics, and mechanical properties, extrusions of the alloy were initiated. Thermo-mechanical processing parameters were to be refined for the extruded condition. However, the project terminated before solution treating on extruded powder samples could be performed.

Process Development was performed in four individual efforts to rapidly develop the processing technology. The four stages included: blending of elemental powders (performed at ORNL), extrusion of low cost titanium powders, (performed at ORNL), development of ST, CR, and A (performed at ORNL), and post-ageing conditioning of the component (performed at the CRADA partner’s facilities). Procedures are identified for blending of elemental powders and development of solution treating, cold rolling, and ageing procedures in the following paragraphs. The extrusion procedures for low cost Ti powder were discussed in the FY2005 Annual Progress Report provided to the Department of Energy[viii]. Post-ageing conditioning is proprietary to the CRADA partner and will not be discussed.

**Blending of Elemental Powders**
The blending of powders to establish the alloy was performed using ITP Ti powder synthesized by the Armstrong process and elemental alloying powders. The initial objective was to blend the powders using attrition methods, and observe the degree of mixing or homogenization that occurred. The secondary objective was to minimize the level of oxygen pickup during blending operations. Once homogenization has been verified, high purity powders were utilized in extrusion of the alloyed bar stock. Two batches of Ti powder with elemental alloying powders, around 128 g for each batch, were milled. One batch was dry milled while the other was wet milled in ethanol to see if one method was more proficient at milling than the other. Zirconia media at a ball to powder weight ratio of 14 to 1 was used to blend the alloy and attrite the ITP Ti powder particles. Milling was performed for a duration of 4 hours. The dry milled bottle was pumped out and backfilled two times with argon. The milling rate was around 100 rps in a 1 liter high density polyethylene container. The material milled in ethanol was dried at 60C overnight and then oblique blended 1 h to mix up the powder. The blended powders were then compressed into 1.7 cm diameter disks by 3-6 mm thick at a pressure of 20,000 psi in air. In a similar cold compression process, ITP CP Ti powder, without alloying elements and without milling operations, was also pressed at various pressures to assess compaction versus pressure trends for the ITP powder morphology; the results were used to develop the correct pressures required to consolidate powder into the cans for extrusion.

SEM and EDX mapping was performed on the compressed alloy samples to observe homogeneity. Chemical analysis was also performed on the sample to assess the accuracy of the chemical composition; inert gas fusion was used to determine oxygen and nitrogen weight percentages (ASTM E 1409-05) and metal elements were analyzed using direct current plasma emission spectroscopy (ASTM E 1097-03).

**Extrusion of Blended Beta Ti Alloy Powders**
Blending of powders was performed in similar procedure as listed above in “Blending of Elemental Powders”. The blended powder of the primary beta developed alloy was pressed into two cans at an approximate density of 30% of theoretical. The can was evacuated and sealed by electron beam welding in vacuum. The can was then extruded after a 925°C preheat for 2 hours.
with an extrusion ratio of 6.75. The peak load on the extrusion press was 380 tons. After extrusion, the can was removed, and small size tensile bars in accordance with ASTM E9 were electrical discharge machined out of the bar stock. Surfaces were ground to remove oxidation. The samples were then tested for monotonic tensile properties in accordance with ASTM E9. Aging and process optimization were not performed due to the early termination of the project.

**Development of Solution Treating, Cold Rolling, and Ageing Procedures**

Development of solution treating (ST), cold rolling (CR), and ageing (A) procedures is critical for the resulting mechanical properties of a titanium alloy component. The primary developed alloy and the conventional beta Ti alloy were both studied. ST of the as-drop cast ingots was performed in a small circular halogen/T3 infrared furnace for 0.5 to 1 hr at temperatures above the beta transus in ultra high purity argon atmosphere. Cold rolling was performed on a 4-high WC roll mill. Multiple passes were performed on the ST samples; 3-5% reductions were taken every pass. Total cold rolling reductions were up to 50%. Ageing was performed in a tungsten element, vacuum furnace at moderate temperatures for 4 to 30 hours. Macro and micro hardness values were measured throughout the processing of both compositions. Hardness values and microstructures were compared.

**Results and Discussion**

**Task 1: Component Design**

Initial design of this component in titanium has been completed. It was selected to replace a steel vehicle component for a weight savings of 40% including allowance for geometric changes required to provide equal mechanical performance. The component is of a bar shape amenable to extrusion which has been selected as the primary process, as well as alternative process methods.

**Task 2: Economic Model**

A preliminary Greenfield model was produced for production of the titanium alloy component. However, after thorough discussion between all parties at the annual review meeting, the approach was modified to focus on utilization of existing commercial operations wherever possible. Restructuring of the model to accommodate this change was initiated, and an effort also begun to identify the potential facilities which would be used in volume production. A synergistic approach was required for the realization of the titanium component and the economic model since economics were of primary importance, new production capabilities were continuing to evolve, and the field of titanium solid state processing was a new and rapidly expanding field. The final economic model was not completed due to the early termination of the project.

**Task 3: Alloy Design and Verification**

As previously indicated, a primary “low cost” composition was selected for initial development. The low cost composition and the conventional beta Ti alloy were first cast into buttons and then...
successfully drop cast into square cross sectioned ingots. A secondary composition was also drop cast, but after down selection further work was not performed with the second alloy. At least three ingots were produced for each composition. Figure 3 is a button and drop casting of the new composition, a micrograph of the new composition, and a micrograph of the conventional composition.

The drop cast ingot and button of the conventional beta alloy and the new alloy were similar in color and appearance. The micrographs exhibited equiaxed grains with the new composition having a similar appearance to the conventional composition. The chemical analysis results were very good for preliminary trials. The conventional alloy contained one alloying element that was off by 1 weight %, and titanium composition was slightly high. The developed composition was closer to target with alloying elements within 0.3 weight percent of target values. Interstitial elements (e.g., oxygen, nitrogen) were very low for both compositions. The hardness of the as-cast ingots was the only significant difference observed between the two alloys; the hardness value of the new alloy was approximately 40% greater than the conventional alloy. The increase in hardness seemed to be solely based on the metal alloying additions since oxygen and nitrogen contents of both sets of ingots were extremely low.

Task 4: Process Development
Blending of Elemental Powders
Initial blending efforts focused on the required procedures to homogenize the powders. Preliminary chemical homogenization results were promising; SEM and EDX results exhibited relatively good uniformity to around 50 to 100 μm. Several of the initial powder particles were larger than optimal; this is the main reason chemical uniformity was not observed below 50 μm. However, fine alloying element powder particles exhibited good blending at even very low scales, as can be seen in figure 4. The chemical analysis results were within 0.6 weight percent for each alloying element. Oxygen and nitrogen values were high in the partially consolidated blended powders; this may be a result of initially high oxygen levels in the alloying powders used, and oxidation during the ball milling process. Values of zirconium, from the zirconia ball media, were below traceable values in the chemical analysis results.

Extrusion Procedures
Extrusions for various sizes, shapes, pressures, and compositions have been previously reported in Annual Reports. Fully dense as-extruded samples have exhibited mechanical, chemical, and
metallurgical properties similar to wrought Ti products. Photos, micrographs, and the resulting mechanical properties are provided in Figure 5 for commercially pure titanium.

Extruded bar stock of blended powders were successfully fabricated. The material was consolidated. The new beta alloy was tested for monotonic mechanical properties. Table 1 illustrates the tensile properties that were found for the two extruded bar, 6 samples each. Strength values and elongation in the as-extruded condition were very close to minimal values of Beta C for high strength ageing conditions. These results indicate that powder metallurgy approach would allow for the new low cost Beta alloy compositions and could eliminate costly solution and ageing heat treatments. Further studies in short anneal treatments could be useful in homogenizing mechanical properties. Final construction of a heavy vehicle component and commercial implementation/transfer were not performed due to the early termination of the project.

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<th>Sample</th>
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Development of Solution Treating, Cold Rolling, and Ageing Procedures
Castings of the new alloys made it possible to study the thermo mechanical process development required for the desired mechanical properties prior to extrusion of the blended powder approach. Therefore, solution treating, cold rolling, and ageing procedures were completed for drop cast specimens, but were not performed on extruded powder specimens. The main intent of the thermo-mechanical processing was to allow for proper alloy selection early in the project. Work
was performed on both arc melt castings at ORNL, and compared with VAR melted samples prepared from an outside commercial source using conventional techniques. The new alloy was processed in a similar fashion as the conventional material using the beta transus, acquired from DTA results, as a reference temperature. Cold rolling of the cast, conventional alloy was performed; a total reduction of 50% after multiple passes was possible. The cast and solution treated, new alloy was compared to this amount of cold work. However, the new composition is harder than the conventional alloy, and the total cold rolling reduction may not need to be at 50%; this agrees with the results from the extrusion data that rolling, solution treating, and ageing may not be necessary. Figure 6 shows the conventional alloy during and after processing with microstructures of the cast material compared to conventional VAR material.

Post-Ageing Procedures and Development of the Component
The CRADA partner has performed preliminary final processing on commercial Beta C plate material in order to begin understanding the effect of this processing on properties. The details of this processing are proprietary. However, the effort has resulted in a basic understanding of the relative effect of several process parameters on some critical material properties. This data will be useful in specification of these process parameters for powder processed material of the finally selected composition if commercialization of a titanium heavy vehicle component is reevaluated at a later date.

The CRADA partner also performed aging studies to begin an understanding of the effect of aging time and temperature on properties. Rockwell C hardness was measured after various aging times at four aging temperatures. Yield strength and hardness were then measured for two of the most promising aging conditions.

All partners in this project have also participated in gathering data and literature on the processing, heat treatments and properties of various Beta alloys for use in designing the thermomechanical processing and heat treatment of the powder processed alloys.
Conclusions
Although Ti and Ti alloys have attractive properties that would benefit a great number of engineering applications, their use is limited by the cost of the raw material and the processing that is necessary to make a usable product. But with the recent advancements in low-cost powder production at ITP, in conjunction with processing techniques developed at ORNL in collaboration with industry, the cost of Ti and Ti alloy products could be drastically reduced. Previous extrusions have proven the feasibility of utilizing low cost powder in new, innovative markets. The preliminary study of CP-Ti extrusions seems to produce mechanical properties similar to cast Ti. New beta alloys were targeted that could lower the cost of titanium alloys; enabling this class of material into new markets. Castings have shown promising microstructures and results while alloy blending practices for the new powders were successful. The new alloys were extruded, but full process optimization and comprehensive mechanical properties were not obtained due to early termination of the projects. Further processing development could lead to new “low cost” Ti alloy components for heavy vehicle usage if market trends allow.

Subject Inventions
No new intellectual property was generated under this CRADA since alloy compositions and manufacturing practices were not fully optimized and/or realized.

Commercialization Possibilities
Further optimization would be required before commercialization could be implemented.

vi B.E. Hurless et al. The AMPTIAC Quarterly, Volume 6, Number 2, pp. 3-9