NOVEL PROCESSING TO PRODUCE TI AND TI ALLOY POWDERS ON A CONTINUOUS BASIS

Authors: *J. C. Withers, J. P. Laughlin, Y. Elkadi, J. DeSilva, R. Storm, V. Shapovalov, D. Myers, R. O. Loutfy*

Materials & Electrochemical Research (MER) Corporation

ABSTRACT

The Kroll process that produces titanium is a multistage sporadic complex heterogeneous slow-speed, uncontrolled, labor intensive, high energy and cost intensive batch process that has defied well over a half century of investigations to simplify into a continuous process. Even with modern engineering current productivity is barely over 1 ton/day per reactor that produces an iron contaminated lumpy sponge product that is formed at the reactor wall interface which limits the capability for continuous operation. Serious environmental issues are prevalent in spite of controlled circulation of Mg, Cl_2, $MgCl_2$ and electrolysis of the $MgCl_2$. An innovative approach has been demonstrated to metallothermically produce titanium continuously in a powder morphology in a single reactor that in-situ produces the reductant metal and $TiCl_4$ without environmental issues. The feed to the reactor is a carbothermically refined TiO_2/ore that provides the source for the in-situ formed $TiCl_4$. The metal reductant can be magnesium or other alkali earth or alkali metals. The concurrent addition of other metal chlorides produces titanium alloy powders. The powders can be used for powder metallurgy processing for meltless manufacturing, as well as rapid manufacturing technology to produce components.

INTRODUCTION/BACKGROUND

It has been over 100 years since Hupperty[1] reported the deposition of titanium by fused salt electrolysis and over 70 years since Kroll[2] reported the magnesium reduction of $TiCl_4$ to produce titanium. The Kroll process is a series of batch steps by which the entire world's production of titanium is produced involving three major steps[3]:

1. The production of titanium tetrachloride ($TiCl_4$) by carbochlorination of the titanium feed (ore/TiO_2) using chlorine (Cl_2) gas and carbon (C) at approximately 1000°C, with subsequent purification of the $TiCl_4$
2. Magnesiothermic reduction of the purified $TiCl_4$ (a highly exothermic reaction ΔH 1000K = -433KJ) and, subsequent separation of the by-product $MgCl_2$ from the titanium sponge
3. Electrolysis of the molten salt, magnesium chloride ($MgCl_2$), obtained in the reduction process to regenerate the Mg reductant and the Cl_2 gas[3].

A technical drawback in the titanium industry is the low productivity of the Kroll production process due to its slow-speed/batch type processing that involves sporadic complex heterogeneous reactions that involve high energy which processing is quite labor intensive resulting in high cost[4]. Many variations of the magnesiothermic reduction have been attempted as reported in the literature[5-10]. A number of intensive research efforts and process developments have been undertaken to overcome some of the crucial issues in the Kroll process[11-17] that have included decreasing the energy consumption of the overall process and reducing cost[5]. In spite of the many studies reported on the Kroll process, there is no consensus on the mechanism of sponge formation and the reaction model[18-23].

The Kroll process is unsuitable for the development of a continuous reduction process because titanium deposits generated in the reduction process firmly adhere to the inner wall of the steel reaction container[24] as a porous aggregate known as "sponge". There have been no complete answers as to why titanium grows from the reactor walls and why the lumps stick/semi-sinter together that produces the sponge[25]. Due to the reaction on the vessel walls, iron contamination is unavoidable. An illustration of the Kroll reaction process is shown in Figure 1[26].

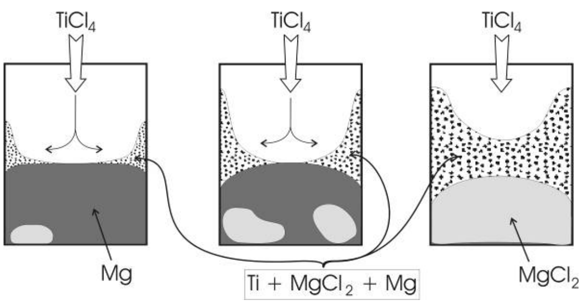


Figure 1: Schematic illustrations for the titanium growth in the Kroll process [26].

Due to the batch complex heterogeneous exothermic reactions occurring in the gas, liquid and solid phases, improvements in productivity speed, cost, and energy and environmental reduction appears difficult and unlikely[27]. However, chloride based processing has an essential advantage since it can produce high quality titanium by using an oxygen-free system[3, 24]. Titanium tetrachloride ($TiCl_4$) is an indispensable intermediate compound for the metallothermic reduction of titanium production and is the established common base to all titanium production throughout the world.

Although the entire world's titanium production is sponge from the Kroll process, there is a strong demand for Ti powder in a specific particle size range for powder metallurgy processing. Processes to produce titanium powder require a Ti-ingot or compact[28]. Since in the Kroll process of $TiCl_4$ fed to the top surface of the molten Mg, its product is sintered sponge as contrasted Ti powder. To exclude the extra cost for ingot making to directly produce titanium powder, investigations have been made to modify the Kroll process to yield powder instead of sponge[26]. One alternative process has been the floating of Mg on top of $MgCl_2$ and bubbling in the $TiCl_4$ below the surface of the Mg layer instead of above the molten Mg layer as traditional. This process produced spheroidal morphology Ti particles under 10μ in diameter[26] which is too small for powder metallurgy and are subject to high oxygen pick-up (even pyrophoric) when exposed to water washing or to the air after vacuum evaporation of the $MgCl_2$. There have been a variety of approaches investigated utilizing fused salt medium to mix the $TiCl_4$ and metal reductant that produces a very small Ti powder[24, 26, 29-39]. Very small and even pyrophoric titanium particles are the result of virtually all burner type reaction schemes where the metal reductant and $TiCl_4$ are mixed. To directly produce titanium particles in a user friendly size i.e. 25 – 40μ at the low end size upto 250μ on the high end size; reaction architecture that differs from the standard Kroll process of reaction at the reactor wall or burner type reactors that occurs in space is enabling. It is necessary to create a reaction architecture where there is ample supply of $TiCl_4$ and metal reductant to grow

Ti particles in the desired size range. There are several reaction architectures that utilize metallothermic processing which can produce not only the desired titanium particle size, but produce spheroidal particle morphology. It is also possible to add alloying chlorides with the $TiCl_4$ to directly produce alloy particles. For example, add vanadium chloride and aluminum chloride to produce Ti-6Al-4V particles. As stated above, this eliminates the necessity to melt CP Ti, alloy and produce an ingot before then reducing to an alloy powder.

APPROACHES TO PRODUCE TITANIUM POWDER CONTINUOUSLY

The first and basic step to the continuous production of titanium powder is the prevention of nucleation on a reactor wall surface such as in the standard batch Kroll retort. It is also a necessity to overcome the restrictions in a burner type mixing of the metal reductant and $TiCl_4$ that results in very small pyrophoric Ti powders. The reactor architecture must provide the metal reductant and $TiCl_4$ in a mixing arrangement that provides nucleation, time, and reactants concentrations to build-up particles >>25µ. The produced Ti particles and by-product metal reductant chloride must be removed from the reactor on a continuous basis, or at least intermediately that permits the reduction reaction producing the Ti particulate to be performed continuously. These reaction conditions are met if the metal reductant is continuously generated in a spacial state in a continuous supply of $TiCl_4$ which has been confirmed experimentally[40, 41]. The experimental techniques can include an electrolytic arrangement, a shower head arrangement, fountain spray arrangement and a spinning vortex arrangement to produce or deliver the metal reductant in a spacial state that produces a spherical droplet. The delivery of the metal reductant spherical droplets into an atmosphere high in $TiCl_4$ gas (that may also contain alloy metal chloride gases) with sufficient time to react in the spacial volume without contacting a surface results in producing spheroidal Ti or alloy particles in a size related to the original reductant metal droplet size. Reactor systems can be designed to continuously deliver the reductant metal in the spacial volume containing a continuous supply of $TiCl_4$, and as may be appropriate alloying metal chlorides, for the continuous production of titanium or alloy spheroidal particulate with the produced titanium and metal reductant chloride removed from the reactor that allows continuous operation. The continuous operation of producing alloy powder directly provides a basis to produce titanium much more cost effectively than the standard batch Kroll process even though the same chloride chemistry is being used.

SYSTEMS TO CONTINUOUSLY PRODUCE TITANIUM POWDER

An electrolytic system such as shown in Figure 2 can be used to practice the concept outlined above. In this illustration, magnesium is electrodeposited on a porous cathode that also feeds $TiCl_4$. The electrodepositing Mg forms droplets as it is deposited and becomes into contact with the $TiCl_4$ that performs the classic reaction to produce titanium particulate and $MgCl_2$ for a cyclic regeneration. The Ti particulate settles to the cell bottom and is harvested with subsequent vacuum evaporation to produce clean titanium analogous to standard Kroll sponge processing.

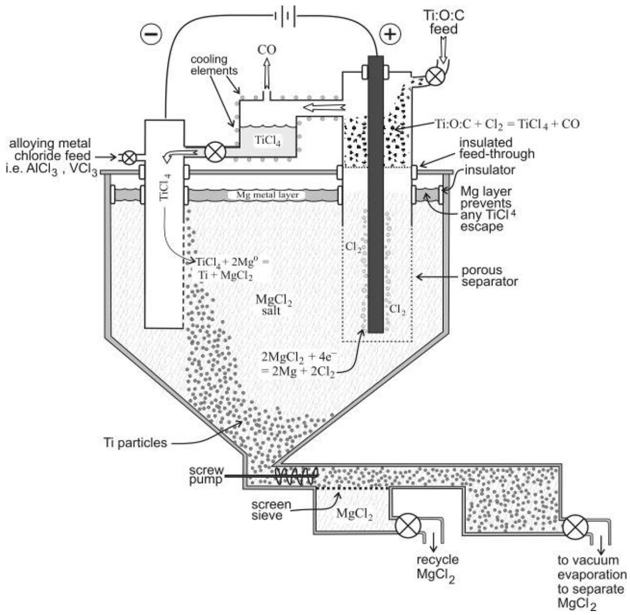


Figure 2: Schematic of Electrolytic Cell to Continuously Produce Ti/Alloy Particulate

In the electrolysis process that produces Mg, it is possible that competing electrolytic reactions reduces the $TiCl_4$ to $TiCl_2$ or the already produced Ti particles can reduce $TiCl_4$ which is not a problem as the Mg then reacts with $TiCl_2$ to produce Ti and $MgCl_2$.

In the processing arrangement shown in Figure 2 there is no separate handling of chlorine or magnesium, the reaction and the reaction components all are performed in-situ within the cell which is a significant environmental benefit. The cell is fed Ti:O:C which is produced by carbothermic reduction of ore/TiO_2 and the titanium particulate is extracted. The particle size is controlled through the electrolysis parameters that produces Mg droplets and the availability of $TiCl_x$ feed. An example of Ti particulate is shown in Figure 3.

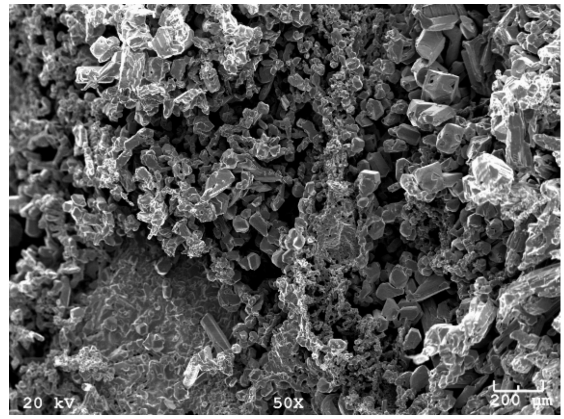


Figure 3: Ti powder produced by in-situ metallothermic reduction consisting of electrolytically produced Mg reducing $TiCl_4$.

Reducing metals other than magnesium such as calcium, sodium, potassium and lithium can be utilized.

An alternative system that separates the electrolytic cell that produces the reducing metal and the reduction reaction is illustrated in Figure 4. As shown, an alloy powder is produced, but pure titanium can also be produced. This process however results in producing particulate on the smaller size as shown in Figure 5.

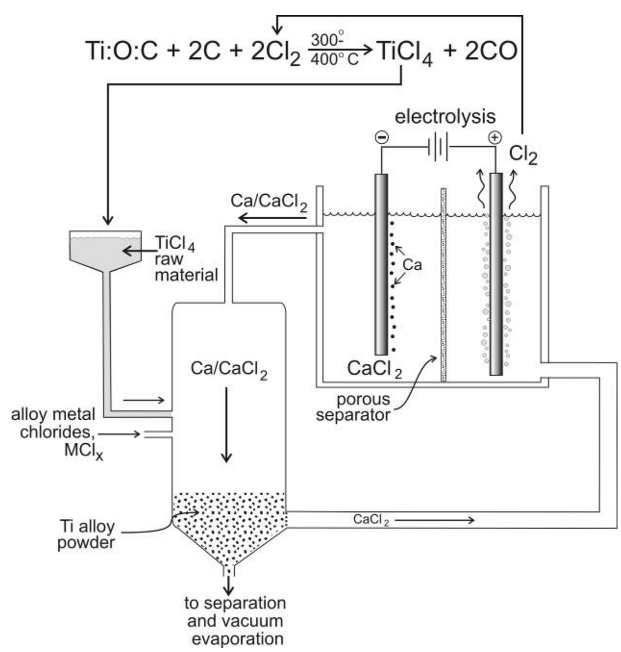


Figure 4: Conceptual Diagram for Continuously Producing Titanium Alloy Powder

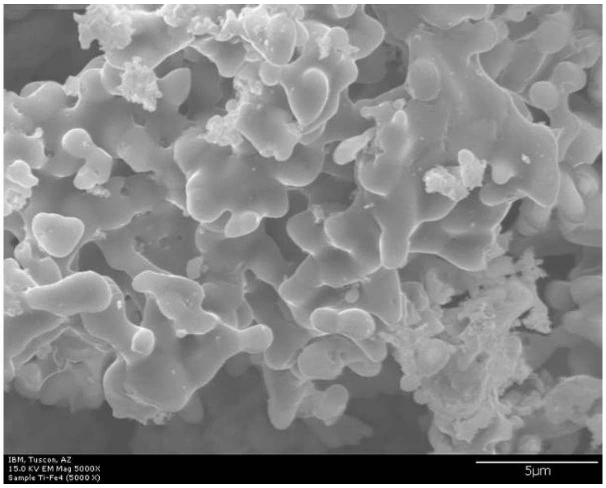


Figure 5: Titanium-Iron alloy particulate produced in a system as illustrated in Figure 4.

Another alternative system that separates the electrolytic cell from the reduction step that does not take place in a fused salt medium is illustrated in Figure 6.

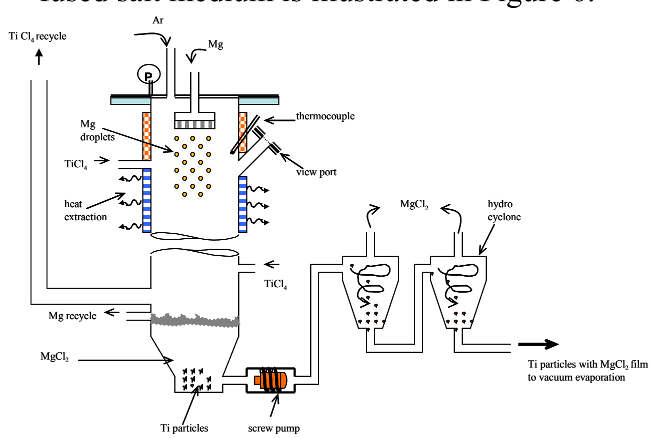


Figure 6: Illustration of a continuous system to produce spheroidal Ti particles from the reaction of Mg droplets and $TiCl_4$ vapor.

The reducing metal droplets are formed by passing the molten metal through a shower type head with very small droplets that fall into a vapor of $TiCl_4$. The reaction chamber has sufficient length that the reducing metal droplets and $TiCl_4$ react to produce spheroidal particle whose size is related to the reducing metal droplet size. Reaction chamber lengths are several feet to assure the reducing metal is completely reacted with $TiCl_4$ by the time it reaches the reactor bottom. Typical lengths depending on the droplet size is under approximately 8 feet. The reducing metal can be any of the alkaline earths or alkalis which Mg is illustrated in Figure 6. It is clear that if alloying element chlorides are added with the $TiCl_4$, an alloy particle will be produced.

Another reaction architecture that prevents nucleation on a wall or other surface and utilizes the basic chloride chemistry concept is illustrated in Figure 7[42]. Although this is a stirred fused salt reactor, the particulate that can be produced is much larger than has typically been reported from fused salt medium of under 10µ[26].

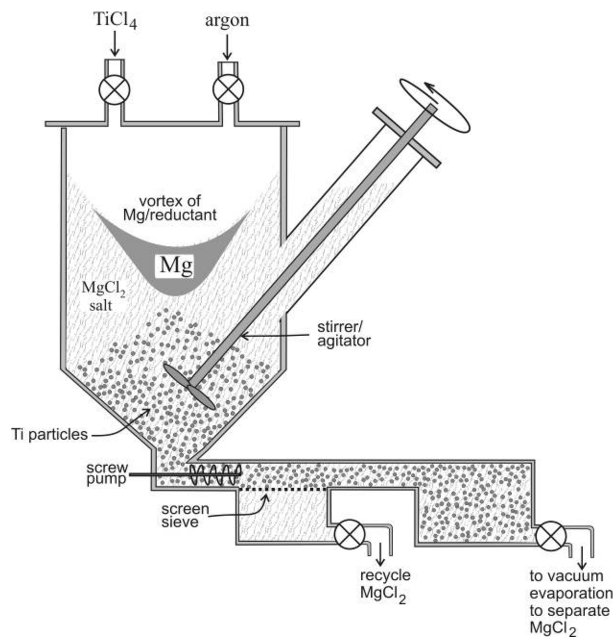
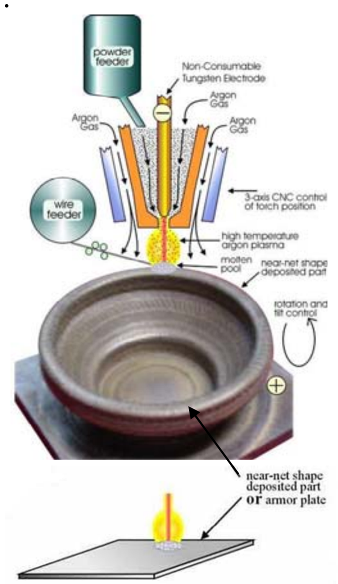


Figure 7: Illustration of a continuous system that eliminates wall nucleation to produce Ti particles instead of sponge[42].

In this case, the $TiCl_4$ and any alloying element chlorides provide a continuous supply of the reactant to a continuous supply of the reducing metal which provides sufficient time for larger particles to form before becoming sufficiently large that the particles settles through the reducing metal reservoir to the bottom of the reactor for harvesting the Ti particulate and reducing metal chloride. There are a variety of ways to stir or circulate the molten salt to create the vortex that isolates the reducing metal from a side wall which would initiate a nucleation reaction. Stirring through the bottom or at an angle through a sidewall are among the simplest methods. It is readily seen that through the continuous feeding of reducing metal and extraction of the produced titanium particulate and the reducing metal chloride, the production of titanium or alloy particulate is continuous.

RAPID MANUFACTURING UTILIZING LOW COST POWDER TO PRODUCE LOW COST PRODUCTS

The titanium powders shown in Figures 3, 5 and the powders that can be produced in the systems shown in Figures 6 and 7 can be utilized in a plasma transferred arc (PTA) rapid manufacturing process shown in Figure 8a and 8b to produce low cost titanium components such as shown in Figure 9.



(a)

(b)

Figure 8: (a) Schematic of plasma transferred arc (PTA) rapid additive manufacturing process and (b) the operational MER unit.

Figure 9: Example Titanium Components Produced by the Rapid Manufacturing Plasma Transferred Arc System

It is well known fabrication cost is approximately 50% of the cost of a product as shown in Figure 10[43] and well may be more where buy to fly ratios of 50:1 are not uncommon. The marrying the low cost powders with low cost PTA rapid manufacturing can produce really low cost products.

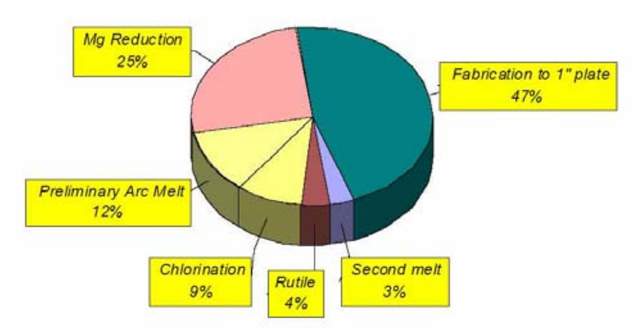


Figure 10: Relative Cost for Producing Titanium Products[43]

Of the additive rapid manufacturing processes such as laser or e-beam, the PTA is significantly more economical from all aspects of capital cost, operating cost and rate of production. The PTA system can use a wire or powder feed separately or concurrently. The operating cost of a PTA rapid manufacturing process can be (only a few dollars per pound) and with the low cost powder has the potential to meet the Army (cost) goal to produce armor plate. As shown in Figure 11, several of the pieces are armor, and in one case consist of TiB_2/Ti armor that provides a 30% weight savings over Ti64 armor plate to provide protection at the same threat level. The PTA system is capable of using Kroll fines as a feed as well as shredded scrap to produce low cost products.

CONCLUSION

The demonstration of generating a reducing metal in a spacial volume free of solid interfaces provides the capability to reduce $TiCl_4$ without or with alloying element chlorides to titanium/alloy particulate that can be spheroidal in morphology in a particle size for direct utilization in standard powder metallurgy processing. The removal of the produced titanium/alloy particulate and the reducing metal chloride intermediately or continuously, results in the ability to produce titanium/alloy on a continuous basis that overcomes the stigma of the batch labor intensive standard Kroll process. The continuous processing provides the opportunity to produce titanium particulate at a cost savings over the standard batch Kroll process. The direct production of alloy powder in a size directly useable in powder metallurgy processing provides the opportunity to produce such alloy powder at substantial savings over the standard process of melting sponge, alloying, producing an ingot and then reducing to alloy powder.

The CP produced powder can be utilized in a PTA rapid manufacturing process with co-fed Al-V powder to produce Ti64 net to near net shape components at a very low cost compared to the traditional manufacturing route. Similarly, alloy powder or shredded scrap can be utilized in the PTA to produce very low cost components.

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Novel Processing to Produce Ti and Ti Alloy Powders on a Continuous Basis

Authors: J.C. Withers, J.P. Laughlin, Y. Elkadi, J. DeSilva, R. Storm, V. Shapovalov, D. Myers, R.O. Loutfy

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Materials and Electrochemical Research Corporation

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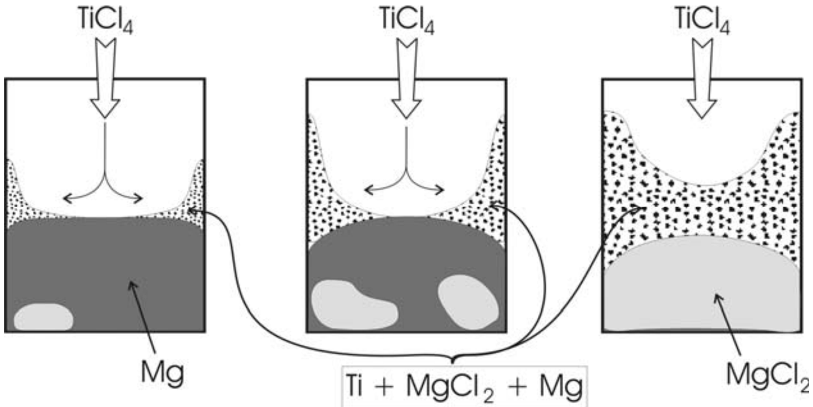
Dr. J. C. (Jim) Withers
jcwithers@mercorp.com
520.574.1980 ext. 113

The Kroll Process to Produce Titanium

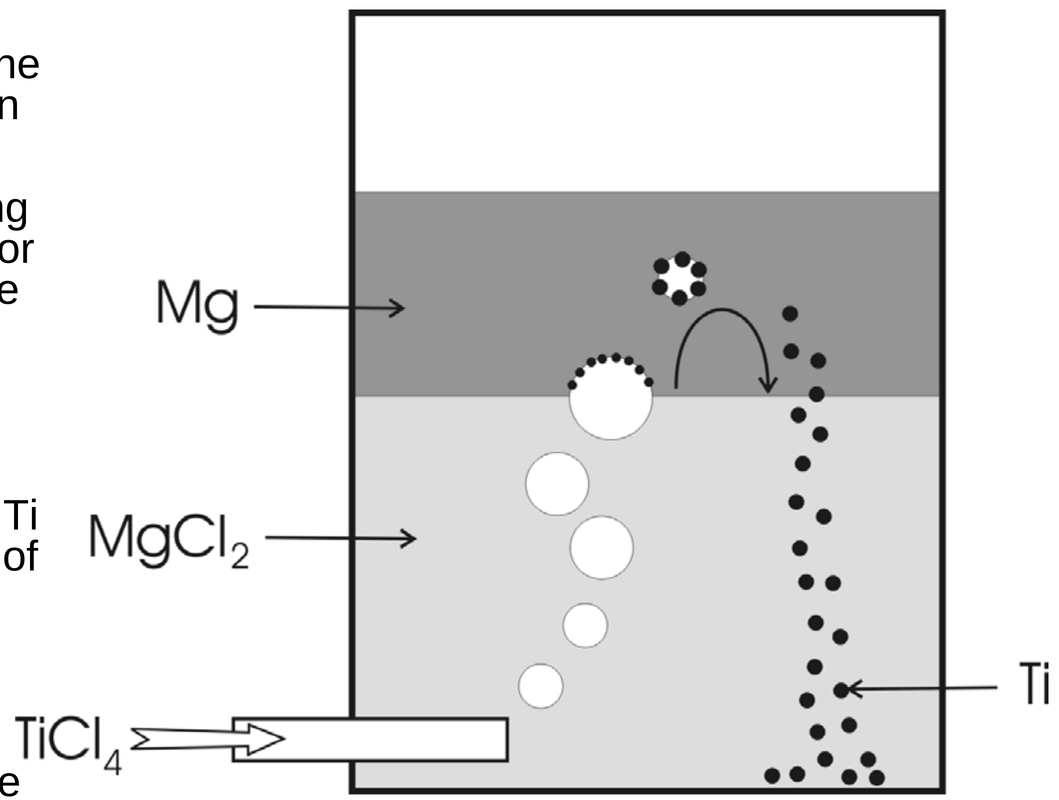


Schematic Illustrations for the Titanium Growth in the Kroll Process

- Nucleation to form titanium particulate primarily occurs at reactor wall – Mg interface
- The produced Ti particulate grows and becomes semi-sintered termed Sponge
- Due to titanium sponge firmly adhering to the reactor/retort wall and semi-sintering, the processing is unsuitable for continuous operation
- Due to nucleation and reaction on the vessel walls, iron contamination of that portion of the produce sponge is unavoidable
- The chloride base chemistry has demonstrated high quality low oxygen titanium can be produced
- Tickle/$TiCl_4$ is an indispensable chloride intermediate for metallothermic reduction to produce titanium

The Kroll Process to Produce Titanium (Continued)

- The slow-speed, batch, labor intensive Kroll process results in titanium sponge at high cost compared to most metals
- In spite of over 70 years of intensive diverse investigations, a consensus on the mechanism of a reaction model has been elusive
- Reversing the Kroll process of introducing the tickle/$TiCl_4$ in the bottom of the reactor in the salt produces only small particulate under 10µ that is subject to high oxygen pick-up even pyrophoric
- Introducing $TiCl_4$ into salts for metallothermic reduction results in fine particles due to the limited availability of Ti content in the $TiCl_4$ gas bubble and lack of reaction architecture to build up the fine particle
- For direct use in powder metallurgy, the particle should be spheroidal in morphology and 25 – 40µ on the low side and about 250µ on the high side



Schematic Illustrating the Titanium Particle Growth with Gas Introduced within the Salt

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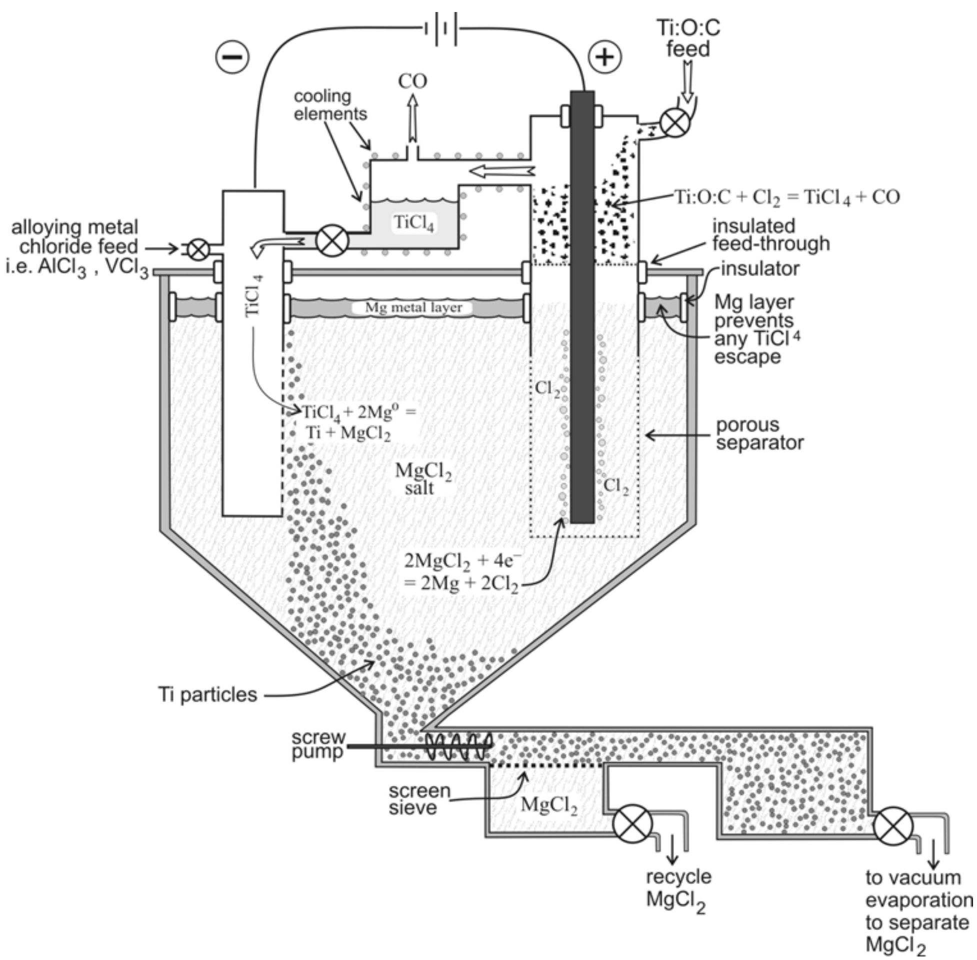
Chloride Chemistry in Reaction Architectures to Continuously Produce Titanium in Controlled Sized Large Particles

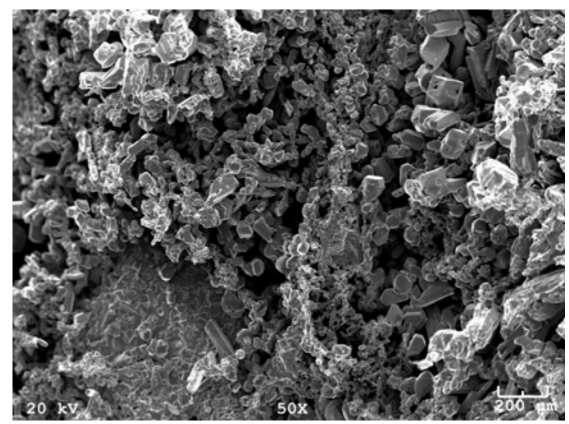
- Reactor design that prevents nucleation on or at reactor wall surface
- Reaction (metal reductant + $TiCl_4$) performed in a spacial state that provides mechanism to form spheroidal Ti particles
- Sufficient concentration of reactants and reaction time within a free space that allows large (25 – 250µ) particle sizes to be produced
- Titanium particle size produced is controlled by reductant metal (i.e. Mg, Ca, Na, K, etc.) spacial dominion size, when sufficient $TiCl_4$ is available to complete the reaction

Electrolytic Cell Reactor Architecture to Produce Titanium Powder

Schematic of Electrolytic Cell to Continuously Produce Ti/Alloy Particulate



- Entire chloride chemistry performed in-situ within a single electrolysis cell
- No separate handling of metal reductant (Mg or other), Cl_2 or $TiCl_4$
- Alloy powder can be produced if alloying metal chlorides are added into the $TiCl_4$ delivery stream
- Continuous production of titanium/alloy powder



Ti powder produced by in-situ metallothermic reduction consisting of electrolytically produced Mg reducing $TiCl_4$.

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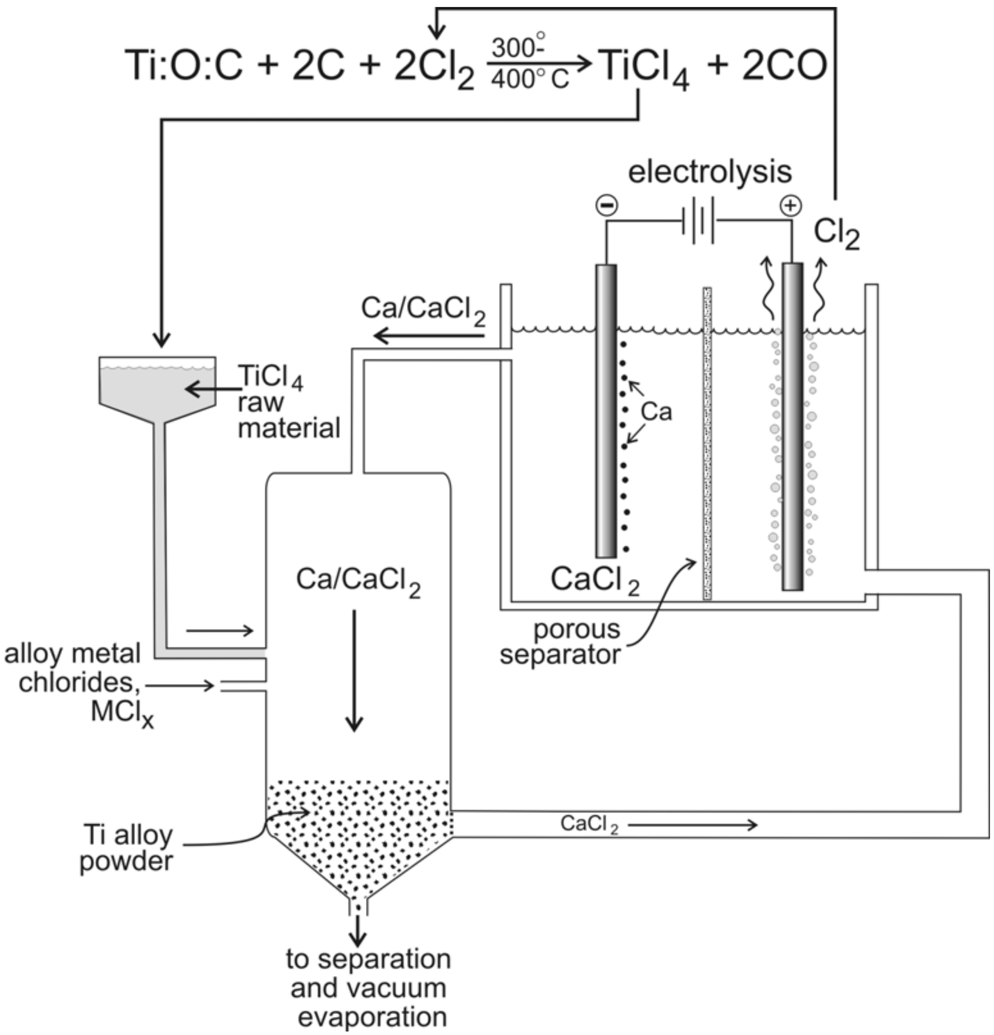
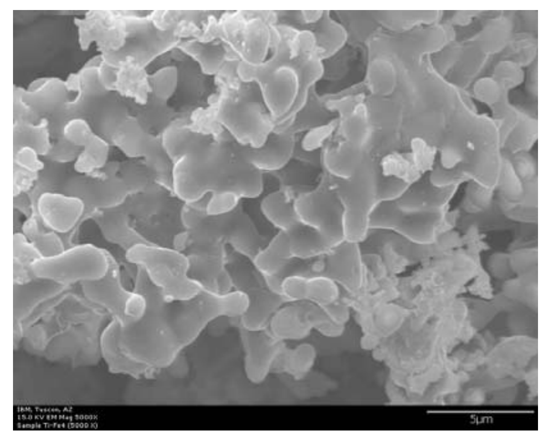
Alternate Electrolytic Cell Reactor Architecture to Produce Titanium Powder



Conceptual Diagram for Continuously Producing Titanium Alloy Powder

- Electrolytic cell producing the reductant metal (Ca or could be Mg) and reaction chamber of metallothermic reduction are separated but tied together in a continuous closed loop
- Can produce pure Ti or Ti alloy powder
- Ti particle size can be built up with continuous supply of $TiCl_4$/metal alloy chloride into the nucleated initial fine powder to produce larger particles before settling to bottom of the reactor.
- Continuous production of titanium/alloy powder



Titanium-Iron alloy particulate produced in a system as illustrated on the left.

Reactor Architecture to Produce Spherical Droplets of Reducing Metal to Produce Spheroidal Titanium Powder

- Reaction architecture is generating spherical reduction metal droplets in a specific size
- $TiCl_4$/alloying metal chloride is maintained as a saturated gas phase in the reaction space
- Sufficient height in the reactor to completely react the reduction metal droplet producing a controlled size spheroidal Ti particle
- Continuous production of titanium/alloy powder

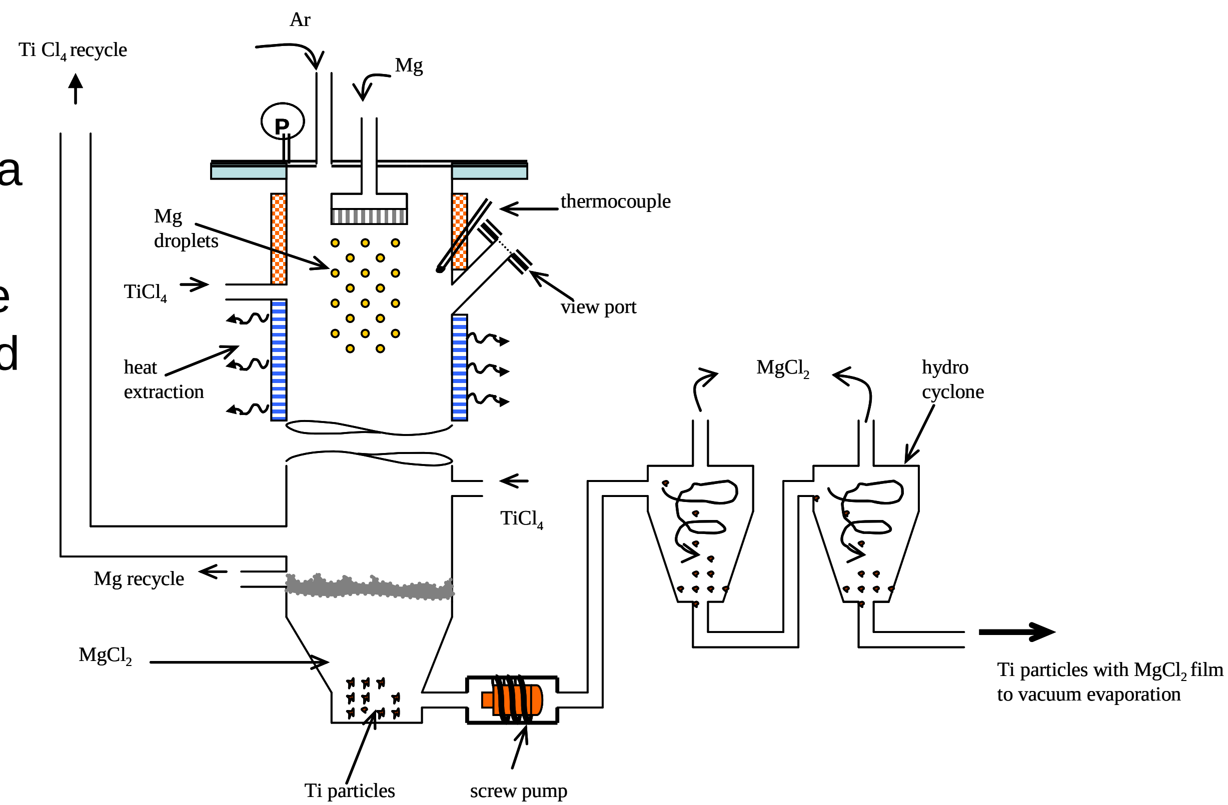


Illustration of a continuous system to produce spheroidal Ti particles from the reaction of Mg droplets and $TiCl_4$ vapor.

Reactor Architecture that Eliminates Nucleation at Reactor Wall – Mg Interface to Produce Titanium Powder

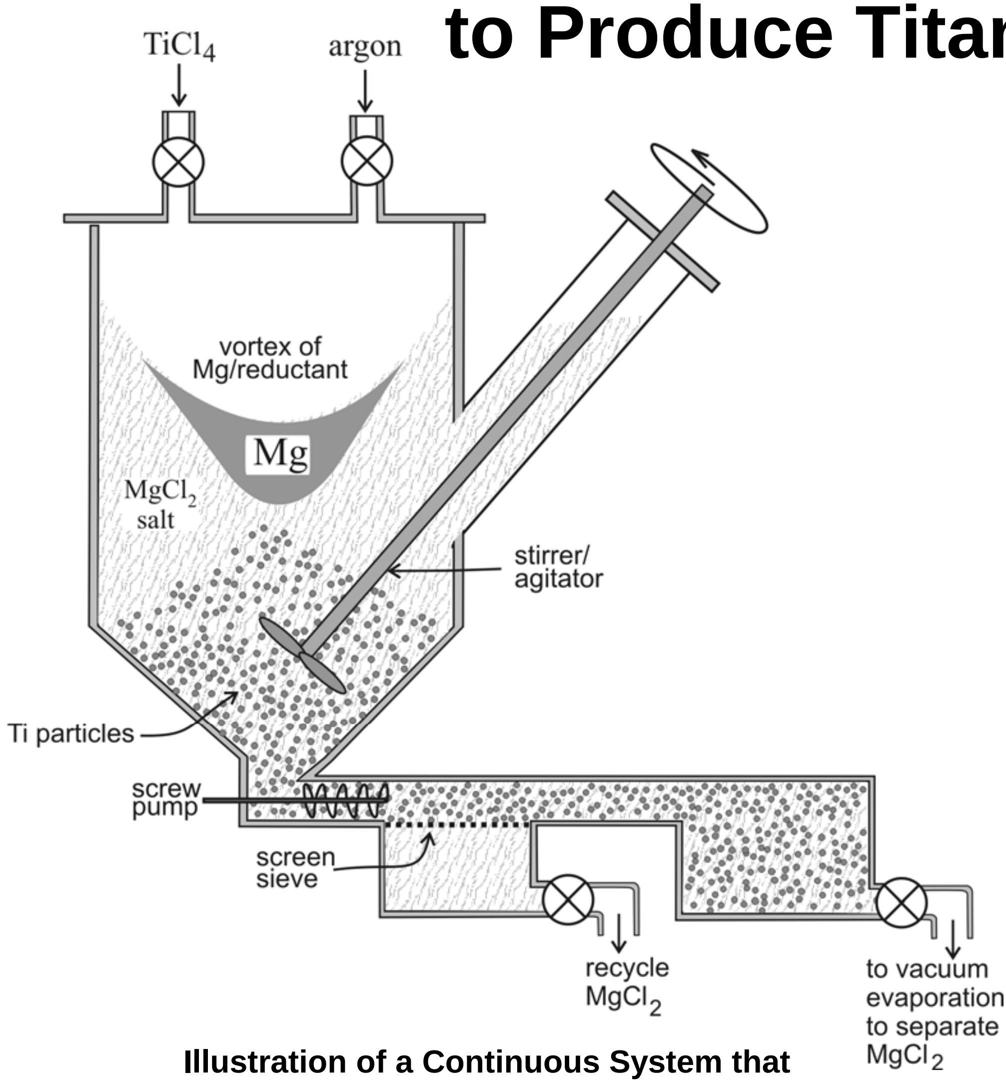


Illustration of a Continuous System that Eliminates Wall Nucleation to Produce Ti Particles instead of Sponge

- Same reaction architecture as Kroll reactor, but eliminates nucleation at the reactor wall that creates the sponge morphology, to produce Ti powder
- Continuous production of titanium/alloy powder

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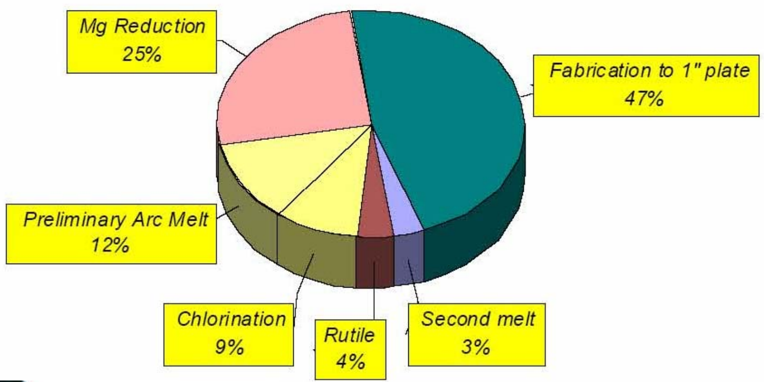
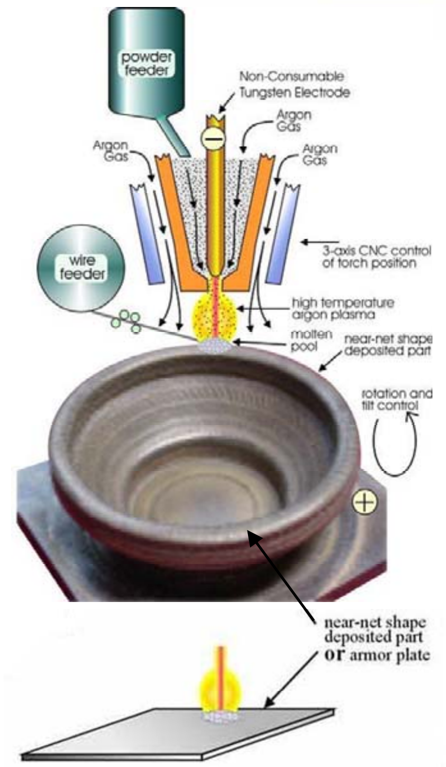
Utilization of Low Cost Ti Powder to Produce Low Cost Ti Products via Rapid Manufacturing





- Rapid low cost manufacturing can substantially reduce cost to manufacture Ti products to near net/net shape components
- If pure Ti powder is the feed, add alloying elements (i.e. Al-V) to produce alloy component

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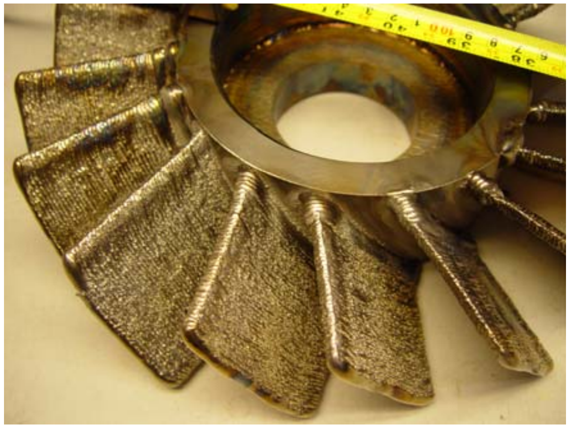
Utilization of Low Cost Ti Powder to Produce Low Cost Ti Products via Rapid Manufacturing (Continued)

- PTA rapid manufacturing process virtually eliminates traditional high buy-to-fly ratios
- The operating cost of PTA rapid manufacturing is only a few dollars per pound
- With low cost Ti powder can meet Army cost goal to produce armor plate
- A TiB_2/Ti armor produced by PTA processing can reduce weight by:

  50% over RHA Steel

  30% over standard Ti64 plate
- The mechanical properties of PTA Rapid manufactured material is the same as produced by traditional processing




Example Titanium Components Produced by the Rapid Manufacturing Plasma Transferred Arc System