Summary of Emerging Titanium Cost Reduction Technologies

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1.0 Summary of Findings

The purpose of this study was to investigate and summarize the development projects currently being carried out by organizations around the world on new technologies for production of titanium metal. Primary focus was on emerging technologies for reduction of titanium bearing oxides to titanium metal. Sixteen such reduction technology projects were identified and are described. While no claim is made that this list contains all such projects, it is believed to include a large majority of such efforts. Several additional technologies are reviewed which have recently been reported, and which may be of interest to vehicular applications. An initial section of this report provides a brief summary of the conventional technologies utilized currently for Ti production. This is included to provide a reference point for consideration of the implications of the emerging technologies for cost reduction. The emphasis on cost reduction technologies is on those with the potential to reduce the cost of final titanium products by very significant amounts on the order of 30%, 50% or more. This current study does not address the ongoing continuous improvement and innovation in present production technology which are expected to provide improvements on the order of 5% to perhaps 15%.

The ultimate commercial product of some of the emerging reduction technologies cannot be defined at this time. Some may be utilized to produce more than one product form; for example, a process may be optimized for production of either solid Ti or a granular or powder product. Nevertheless, the processes may be broadly grouped into those that will produce solid ingot, billet or slab, and those that will produce some form of sponge, granular or powder product. Figure 1 shows a general sequence of process steps for conventional production of titanium mill products using Vacuum Arc Melting. The Figure also shows the range of these process steps which would be replaced by various alternative technologies. The number of process steps replaced by the emerging technologies varies among the approaches, and in some cases has not been finally determined.

Production of a sponge product to be utilized as a replacement for Kroll sponge does not appear to have potential for large cost reduction. The processes which may provide sponge all use some form of halide electrolyte or metal reductant which would need to be separated from the product sponge. In addition, if the process does not utilize $TiCl_4$ for raw material purification, then another such process must be utilized. Any cost reduction from the current Na reduction of $TiCl_4$ could not be expected to yield final product cost reduction of more than 5 – 10%. Such a process could replace only up to a few steps in the conventional process route depicted in Figure 1.

Cold hearth melting technologies are described briefly in the Conventional Processing section of this report. These technologies are providing needed incremental improvement in the economics of production. Figure 1 shows that they replace many of the steps in conventional VAR processing, although for some applications VAR must still be utilized for final melt. In addition to fewer process steps, the quality of product may be improved when utilizing high scrap levels, which in itself is a cost reduction measure. Finally, these processes may be used to produce rectangular blooms or slabs, which require fewer hot working operations, with the accompanying cost savings.

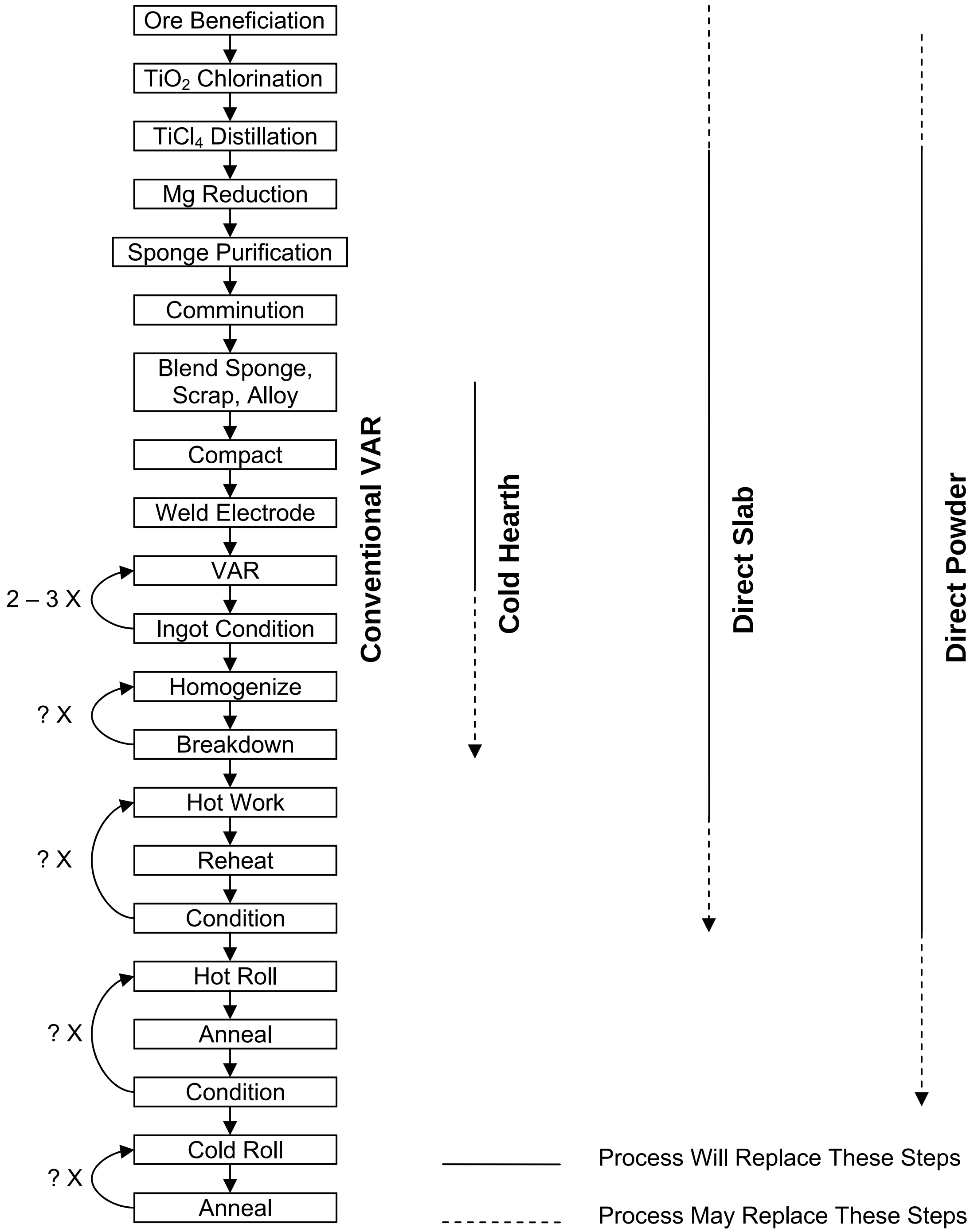


Fig. 1 Reduction in Process Steps By Emerging Reduction Technologies.

The group of emerging processes labeled "Direct Slab" in Figure 1 is those that either produces liquid, which can be cast into rectangular slab, or which directly produce solid slab. Depending on the process, these may replace several of the initial steps in the

Kroll Process. None of these processes are known to have round ingot as their target product, but are expected to produce rectangular bloom, billet or slab directly, in a single process sequence. In common with cold hearth, these processes would not require repeat cycles of any process step. The form of the final product will determine how far along the conventional process sequence these new technologies will provide replacement. General concerns with this class of process are: the degree of chemical homogeneity which is provided; the ability to provide complex, multi-element alloys within the chemistry tolerance required; the surface condition produced by some processes without the need for extensive conditioning.

The group of emerging technologies labeled "Direct Powder" in Figure 1 are those that have granules or powder as their target product. Not depicted in this Figure are any process steps that are required to purify the powder and further process it into useable form. This finished powder or granular material may be useable in several subsequent processes to produce final product. Titanium powder metallurgy is a very small industry due to both the high present cost of quality powder, the need for specialized facilities and processes to handle the reactive powder, and to contamination by residuals of binder removal. The new powder technologies can be expected to significantly lower powder cost, thus providing incentive to resolve the other issues. Powder or granules may also be utilized as feedstock for a variety of compaction and sintering processes with the objective of providing mill products such as sheet, strip, plate, bar, wire and forging preform. Very little work has been done, however, on utilization of powder for these downstream processes. Use of mixed CP titanium and elemental alloy blends for production of complex alloys has been demonstrated. The promise of a direct route to such finished products should provide incentive for development of these methods. Significant cost savings by replacement of a large number of process steps appears to be worth the effort. General concerns with this class of process include: the low level of experience in using powder to manufacture mill products; the reactivity of powder / granules during its use to make mill products.

As mentioned, sixteen development projects for new oxide reduction processes have been identified. Some are well known, while others have just been revealed. Table 1 lists these processes, the organization performing the development, and the general nature of the expected product. In some cases considerable detail has been obtained, while little has been released on some efforts. The Table presents the list in random order. The details of the processes which have been released are provided in the report. Some processes operate by reduction of a titanium halide. Many utilize some form of electrochemical reduction while still others rely on metallochemical or metallothermic reduction. References are provided in many cases for further study of the mechanisms. Many of the projects are in the very early stage of development so that optimization and scale up are many years in the future. Other efforts are reported to be either in the pilot stage or near to scale up. No opinions are expressed as to the likelihood of success of any of these processes. Sufficient insight has been gained, and progress demonstrated, however, to have confidence that some of the developments will succeed in commercialization.

No attempt was made to be as comprehensive in investigating other groups of emerging titanium technologies. There are numerous efforts at developing new alloys with reduced cost through utilization of low cost master alloys. Strong effort continues on development of titanium intermetallics. Solid freeform fabrication of titanium is now being commercialized, especially for use to add stock to structures in order to reduce machining. Titanium continues to find new applications in industrial, consumer and vehicular applications as well as potential new defense applications. As the promise of significant cost reduction is realized through the emerging technologies discussed, as well as others to be disclosed, applications and substitution of titanium for other metals can be expected to increase.

Table 1 Summary of Emerging Reduction Technologies

Name / Organization	Process	Product(s)
FFC / Cambridge Univ. / Quinetiq / TIMET	Electrolytic reduction of partially sintered TiO_2 electrode in molten $CaCl_2$	Powder Block
Armstrong / International Ti Powder	Liquid Na reduction of $TiCl_4$ vapor	Powder
MER Corp.	Anode reduction of TiO_2, transport through mixed halide electrolyte and deposition on cathode	Powder, Flake or Solid Slab
SRI International	Fluidized bed reduction of Ti halide	Powder, Granule
BHP Billiton	No details available	NA
Idaho Titanium Technologies	Hydrogen reduction of $TiCl_4$ plasma	Powder
GTT s.r.l. (Ginatta)	Electrolytic reduction of $TiCl_4$ vapor dissolved in molten electrolyte	Liquid Ti, either tapped or solidified as slab
OS (Ono / Suzuki; Kyoto Univ.)	Calciothermic reduction of TiO_2	Powder / sponge
Millenium Chemical	No details available	Powder
MIR Chem	I_2 reduction of TiO_2 in "shaking reactor"	Particles
CSIR (S. Africa)	H_2 reduction of $TiCl_4$	Sponge
Quebec Fe & Ti (Rio Tinto)	Electrolytic reduction of Ti slag	Ti Liquid
EMR / MSE (Univ. of Tokyo)	Electrolytic cell between TiO_2 and liquid Ca alloy reduces TiO_2	Highly porous Ti powder compact
Preform Reduction	Reduction of TiO_2 reduction by Ca	Ti powder compact
Vartech	Gaseous reduction of $TiCl_4$ vapor	Powder
Idaho Research Foundation	Mechanochemical Reduction of liquid $TiCl_4$	Powder

2.0 Introduction

Titanium is the ninth most abundant element, comprising 0.6% of the earth's crust. It is also the fourth most abundant structural material after aluminum (8.1%), iron (5.1%) and magnesium (2.1%). Of these four elements, only aluminum has a higher free energy for reduction of its oxide. Nevertheless, 1997 US titanium production, including scrap recycle, was only 48,000 metric tons, vs. 138,000 metric tons of Mg, 7.2 million metric tons of Al, and 99 million metric tons of steel. Inversely, prices ($/metric ton) for these metals in '97 were $9,656 (Ti sponge), $3,460 (Mg), $1,440 (Al) and $625 (Steel).

The reason for this discrepancy in pricing and production volume is primarily due to the high reactivity of titanium. Titanium has a great affinity for oxygen, nitrogen, carbon and hydrogen. Even though the free energy of formation of TiO_2 is less than that of Al_2O_3, no smelting process similar to that used for aluminum has been successful. The Kroll process and subsequent purification operations used for the majority of titanium production is energy, material and capital intensive, so that the sponge produced currently sells for $3.50 – 4.00 per pound. Since approximately half of titanium production is used in aerospace applications, and these are the most profitable applications, the requirements of this industry have dominated production technology. Stringent property requirements dictate very low levels of microstructural defects. Melt processing in either vacuum or inert atmosphere is therefore required. Double and even triple melting sequences are common. Mill processing, such as conversion of ingot by hot rolling and forging can only be carried out in air, so that multiple conditioning steps (oxide and surface defect removal) are required. Yield loss and the cost of these conditioning operations can contribute half of the cost of plate and bar products. Final prices for titanium mill products consequently range from a low of ~$8/lb to $20/lb and sometimes much higher, depending on form, specification, quantity, alloy and the state of the aerospace economic cycle.

Efforts to reduce the cost of titanium products have continued practically uninterrupted since the beginning of the industry. Progress has been made in improving the efficiency of the conventional process route, and in development of some process alternatives. None of these efforts, however, have provided pricing approaching that of the competing materials. In recent years, there has been an acceleration of the interest in alternative routes to titanium product. Most of this effort is directed at alternatives to the use of ingots cast from double or triple vacuum arc remelted Kroll process sponge. Objectives include providing either lower cost billet / slab, or production of high quality powder at low cost. Lower cost slab has the potential to reduce product cost by both reduction of the casting cost, and elimination of ingot breakdown steps. Efforts to produce low cost powder may be viewed either as providing an alternative to sponge in the ingot/slab casting process, or to provide raw material for alternative routes to mill product and for conventional powder metallurgy.

The objective of this report is to review the efforts to develop new technology for titanium production. A summary of conventional technology is provided, along with description of some of the current improvement efforts. Sixteen approaches to reduction of titanium oxides to pure and alloyed Ti have been found. These efforts are described in levels of detail which vary depending on the status of development, and the willingness of the developing organization to disclose information. Reluctance on the part of many firms is understandable considering the value of the intellectual property being developed and the competitive nature of the industry. Finally, some description is provided of technologies available or under development to convert the output of these new reduction processes into useable product.

Extensive effort has been expended in investigating the existence of the emerging technology efforts, and in obtaining meaningful details of the processes and products. While an attempt has been made to collect and to verify the information provided herein, no warranty can be provided that all of the information presented is entirely accurate. There are undoubtedly other activities which may have equal or superior promise for providing affordable titanium. Readers are invited to send any comments or additional information to the author.

This report may serve as an update for an earlier report that also focused on the opportunities for low cost titanium in heavy-duty vehicles.[1]

3.0 Conventional Processing

In order to understand the importance and cost reduction potential of the emerging reduction and processing technologies, it is instructive to review the conventional production methods and the sources of high cost. Figure 1 includes a view of the sequence of operations normally used in titanium mill products production. For simplicity, this sequence uses Vacuum Arc Remelting as the melt process. It should be recognized that one of the cold hearth melting technologies may be used instead of or in conjunction with VAR. Nevertheless, this long sequence of process steps is currently required to produce quality titanium mill products. As with the mill processing of most metals, an iterative sequence of reductions and reheats is required. More than some metals, titanium normally requires many hot work and homogenization steps in order to produce desired chemical and microstructural uniformity. Many of these steps involve ingot, bloom, slab or plate conditioning which is required to remove surface contamination and roughness, with a resulting high yield loss. Figure 2 is an estimate of the relative cost factors for production of one inch titanium alloy plate, and serves to illustrate the sources of high cost.

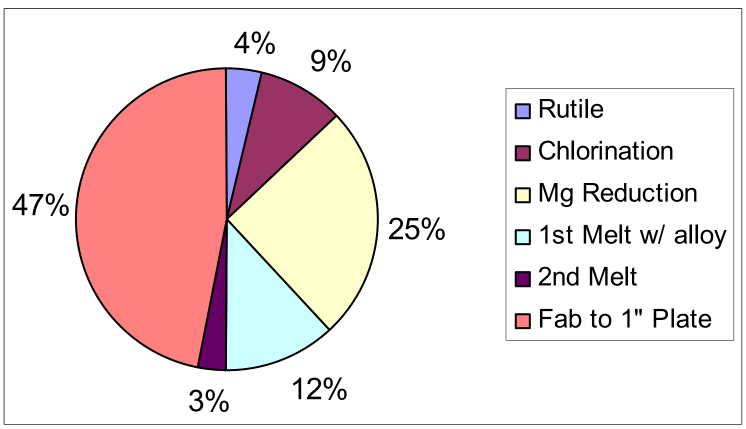


Fig.2 Relative Cost Factors for Conventional Mill Processing of 1” Ti Alloy Plate [2]

3.1 Titanium Raw Metal - Sponge

Titanium metal for the production of mill products (sheet, strip, plate, bar, wire), castings and forgings has been made by essentially the same process since the start of the industry in the mid 20th century. The vast majority of this metal is made using a multi-step process pioneered by Dr. Wilhelm J. Kroll in the 1930’s.[3, 4] Titanium originally comes from the ores Rutile (TiO_2; Anatase is a closely relative crystal structure), and Ilmenite ($FeTiO_3$). Ilmenite ores are used in Fe production, leaving a slag rich in TiO_2, which is normally upgraded for use in Ti production. Figure 3 is an overview of Dr. Kroll’s process as practiced by one manufacturer today.

The steps in this process are:

1. Chlorination of TiO_2 with coke, by the fluidized bed reaction:

$$TiO_2 + 2Cl_2 + C = TiCl_4 + CO_2$$

followed by distillation to purify the $TiCl_4$ of metallic impurities such as Fe, Cr, Ni, Mg, Mn.

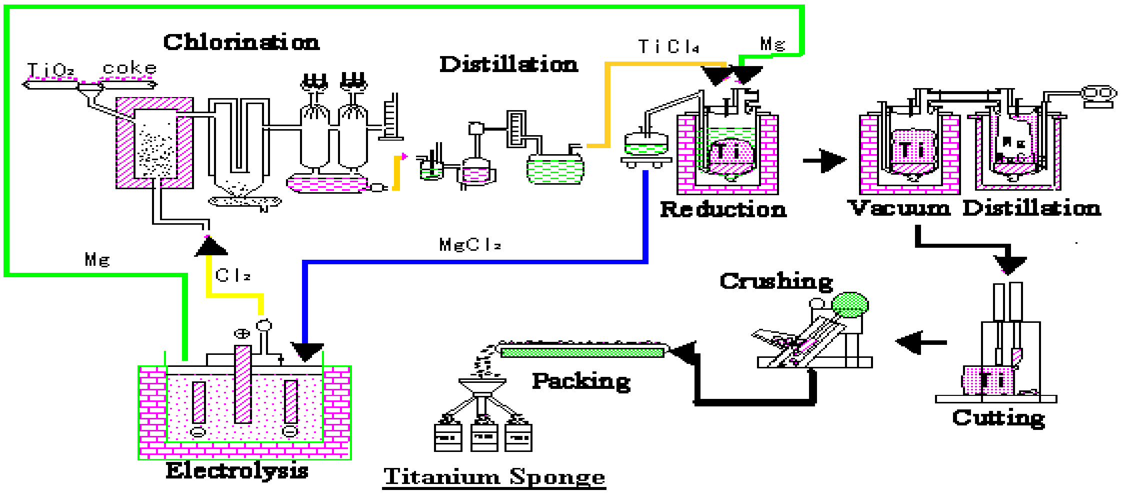


Fig. 3 Overview of Titanium Sponge Production [5]

2. Magnesium reduction of $TiCl_4$ in a sealed, inert gas filled retort according to the reaction: $TiCl_4 + 2Mg = Ti + 2MgCl_2$
   A furnace helps control the temperature of the exothermic reaction. Either solid Mg is melted, or liquid Mg is used, followed by controlled introduction of the $TiCl_4$. Molten $MgCl_2$ is tapped from the retort periodically. After consumption of the Mg and final draining of $MgCl_2$, remaining Mg and $MgCl_2$ must be removed.
3. Vacuum distillation is the most prevalent method of removing impurities from the sponge. Other processes which are or have been used include He gas sweep followed by acid leaching, or simple acid leaching. In most cases, the sponge on the outside of the mass, next to the pot wall, is either left in place or discarded as a means of absorbing the iron and associated metals leached from the pot.
4. Comminution of the sponge mass, either before or after purification, is carried out by a series of boring, shearing, crushing and screening steps.

Examples of the sponge produced by this process are shown in Figure 4. Sponge is a primary raw material used in the melting operations producing ingot or slab. It is available in various grades, with varying levels of impurities.

3.2 Melting, Ingot and Slab Casting

3.2.1 Vacuum Arc Remelting (VAR): The conventional, and most common method for producing titanium ingot is the vacuum arc remelting process, depicted in Figure 5. As shown, titanium and alloy elements are blended to the desired composition. This blend may contain levels of scrap Ti (revert), which has been carefully controlled for composition and contamination. The blended material is then compacted and the compacts assembled with additional scrap and a stub, and welded to form an electrode.

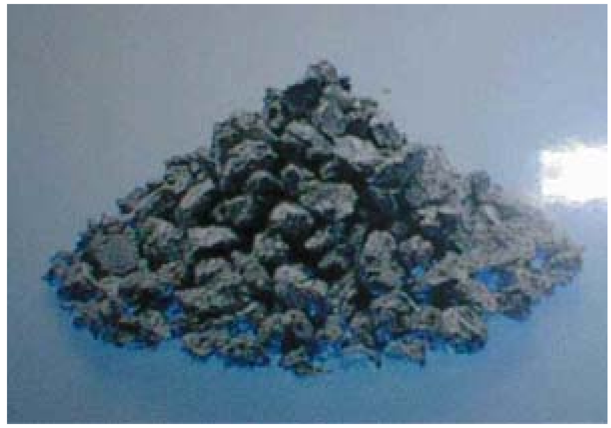
a)  b) 

Fig. 4 Ti sponge: a) before, and b) after crushing [6]

The VAR furnace consists of a water cooled copper crucible, vacuum system, electrode drive and control system. Some operations utilize high frequency electrical coils around the furnace to induce magnetic stirring in the melt for improved homogeneity. The ingot from this first melt is conditioned by grinding to remove surface defects and contamination, is inverted and welded to a stub. A second vacuum arc remelt is normally used to improve homogeneity and dissolution of inclusions. Very high reliability applications such as turbine rotor components may use a third VAR. Only round ingots are produced by VAR. The ingots are conditioned again prior to conversion by grinding or turning to remove contamination and surface defects that could act as stress concentrations or crack initiators during subsequent hot working.

Sources of high cost in these processes include the labor intensive electrode preparation, multiple melt sequence and the intermediate and final conditioning with its attendant yield loss.

3.2.2 Cold Hearth Melting: Cold hearth melting, as its name implies, utilizes a water cooled copper hearth to contain a “skull” of solidified titanium, which in turn contains a pool of molten metal. Figure 6 shows a simplified view of one configuration using a gas plasma as the heat source (Plasma Arc Remelting – PAM). Other configurations of the process may use an electron beam as the heat source (Electron Beam Cold Hearth Melting – EBCHM). The figure shows only one pool for addition of material and homogenization, whereas there are often multiple pools for these functions, with multiple plasma or electron beam guns for precisely monitored and controlled heat input. Advantages of cold hearth melting include improved capability for scrap melting, improved process control and the ability to cast rectangular slabs in addition to round ingots. Improved scrap utilization involves removal of high density inclusions by gravity settling and entrapment in the mushy zone at the bottom of the molten pool. Figure 7 shows the rectangular slab produced by one electron beam facility. Efforts are underway to utilize cold hearth melting in a single melt operation for less critical applications. For high stress and fatigue inducing applications such as engine rotors, VAR melting may be required after cold hearth melting.

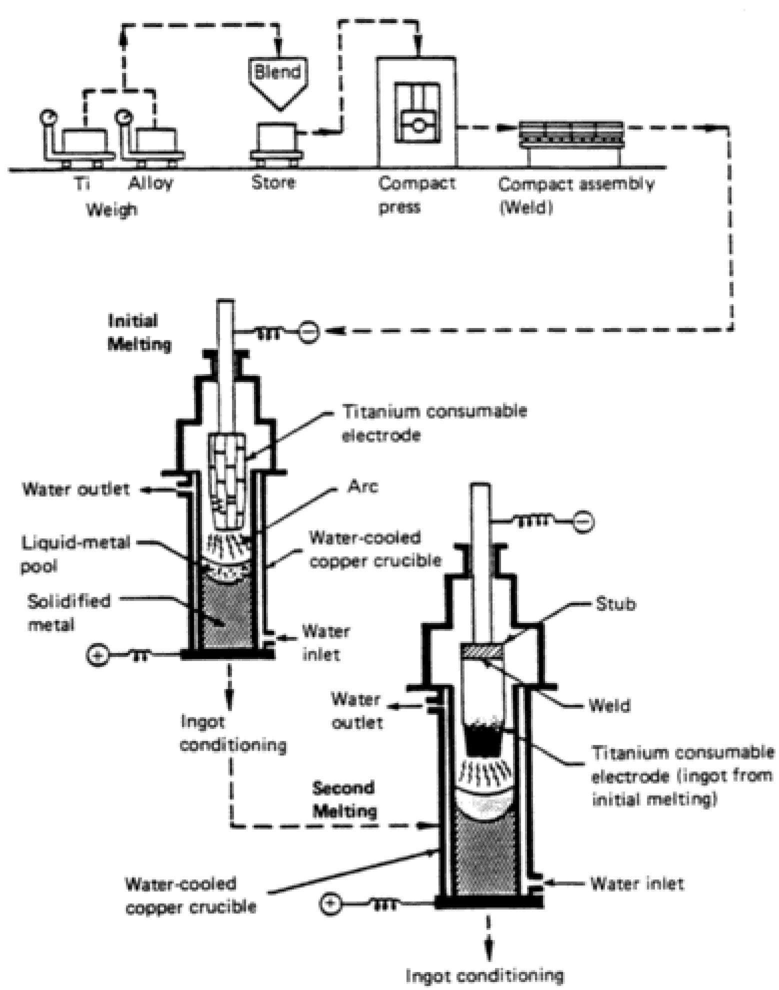


Fig. 5 Flow Diagram for Double Vacuum Arc Remelt Process for Titanium Ingot [7]

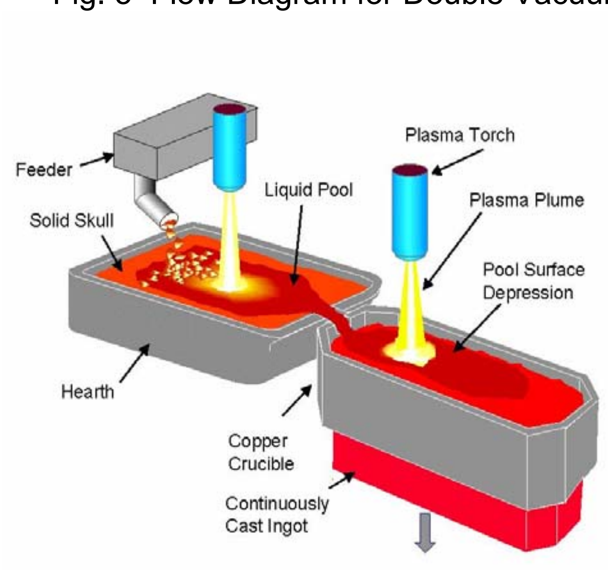


Fig. 6 Schematic of Galt Alloys Plasma Arc Melting Process [8]

Fig. 7 Slab produced by Antares Electron Beam Furnace [9]

3.2.3 Electroslag Remelting (ESR): ESR has been used for many years for production of tool steels, superalloys and heavy forging steel ingots. Like VAR, the process must start with a solid or fabricated electrode. Figure 8 shows one variant of an ESR furnace. The distinction of ESR from VAR is the use of a molten slag into which the electrode is dipped. As the bottom of the electrode melts, drops of liquid fall through the slag and into the molten metal pool at the bottom of the furnace. As the drops fall through the slag, they are refined, with removal of non-metallic impurities by chemical reaction with the slag. Solidification occurs essentially uni-directionally, eliminating central pipe, and providing improved homogeneity. Rectangular slabs are also now available from ESR furnaces.[10]

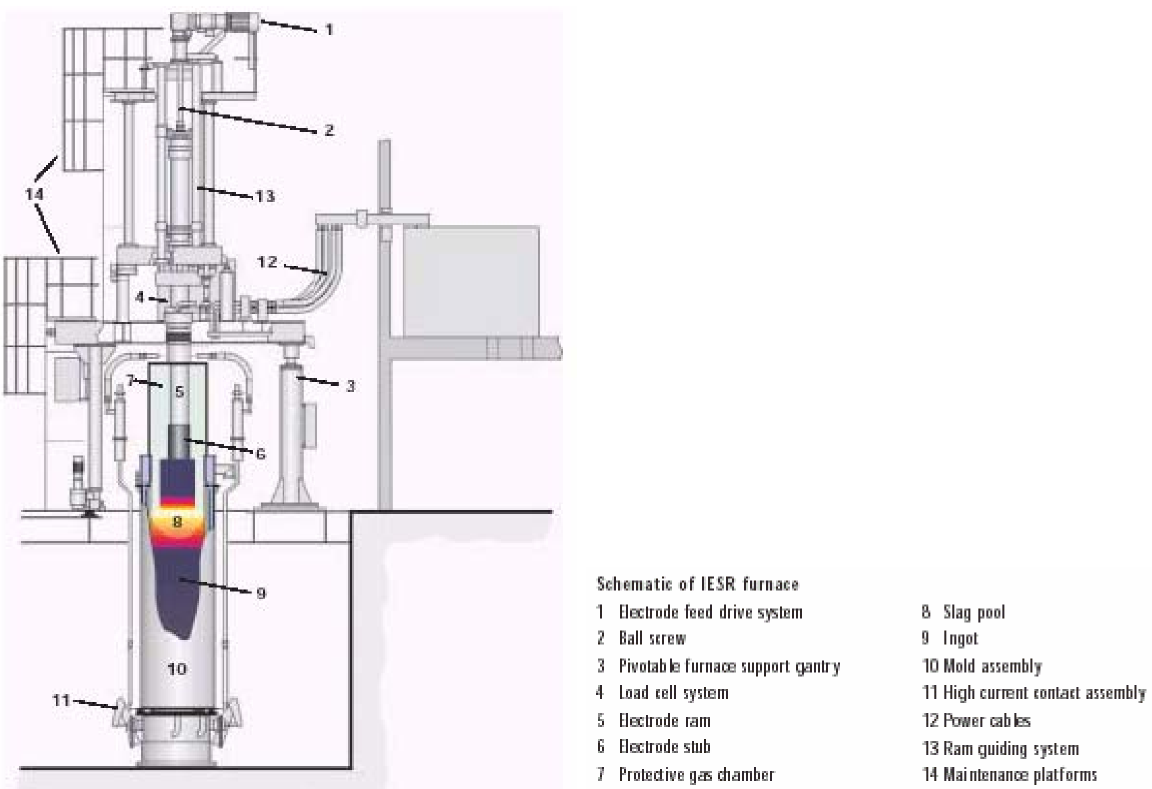


Fig. 8 Schematic of ALD Vacuum Technologies Electroslag Remelting Furnace with Inert Gas Atmosphere [11]

3.3 Mill Processing of Bar, Plate and Strip

When an ingot or slab is produced for use in mill products such as bar, plate and strip, it is processed through a sequence of operations such as is illustrated in Figure 1. The multiple breakdown, homogenization, reduction, reheat and conditioning iterations are complicated by the oxidation susceptibility of the material. Production of a hard and brittle oxygen stabilized alpha layer (alpha case), combined with surface defects requires frequent surface removal and trimming. These operations are costly and result in significant yield loss. These losses have been reduced to some extent in recent years by use of rectangular blooms and slabs rather than round ingot castings.

4.0 Emerging Reduction Technologies

It is apparent from the discussions above that if a process could be developed that would eliminate many of the process steps in conventional production of mill products, very large cost savings could be achieved. There have been a great variety of efforts over many years to achieve this goal, with near universal failure. In recent years, however, a variety of new approaches have been developed and effort has expanded, resulting in some promise of success. Sixteen current efforts at reduction of oxides to titanium metal or hydride have been identified, plus a new approach to cost reduction of the hydriding of scrap. The products of these processes range from liquid Ti which may be cast into rectangular slabs, to solid slab production, sponge like forms and powder. In most cases, current effort is focused on process development with a "CP" grade of material as the product. However, in most cases, claims of applicability to alloys have been made, or the expectation expressed. Little confirmation of flexibility to produce a wide variety of complex alloys directly has been provided. Fortunately, this is not likely to be a serious issue for processes producing powder as the ability to blend CP powder with master alloy powder and develop homogeneous structures has been repeatedly demonstrated. One unresolved issue for these powder production technologies is the treatment of the reaction product into powder of usable impurity level, particle size and morphology. For the slab producing technologies, the ability to produce an alloy slab of uniform thickness, chemical homogeneity and adequate surface smoothness to avoid excessive conditioning also remains to be demonstrated.

An initial attempt was made to categorize the identified technologies according to type of expected product. The variety of product forms, however, precluded this classification. Also, since there is no standard nomenclature for each process, an alphabetical listing was also impractical. The following list is therefore presented in a purely random order. Position in the sequence has no relation to any factor such as development status, size of the effort, development organization or any relationships of the author to any organization.

4.1 FFC / Cambridge Process: Cambridge Univ., QinetiQ, British Titanium, TIMET

Process Description: This process is most easily understood as the electrolytic reduction of solid TiO_2 which is immersed in a molten CaCl electrolyte. Figures 9 and 10 show the overall process and the reduction cell schematically. A TiO_2 powder is formed by conventional ceramic processing into a rectangular sintered cathode incorporating a conducting wire. This cathode is then immersed in the electrolyte with a graphite anode. Reportedly[12], removal of a small amount of oxygen from the electrically insulating rutile phase converts it into the highly conducting Magnelli phase (TiO_{2-x}). Continued electrolysis removes oxygen from the cathode, where it dissolves in the electrolyte and is then removed as O_2, CO or CO_2 at the anode. At the voltages used, no calcium is deposited. Process times are between 24 and 48 hours, with resulting oxygen levels below 1000ppm and N_2 of 5 – 20ppm. Longer processing times allow lower O_2 levels. Simultaneous reduction of several oxides has reportedly allowed production of Ti-6Al-4V alloy.

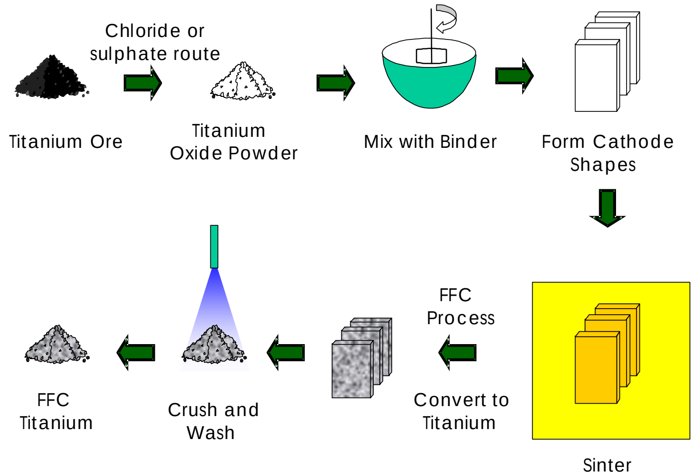


Fig. 9 Schematic Description of the FFC-Cambridge Process[13]

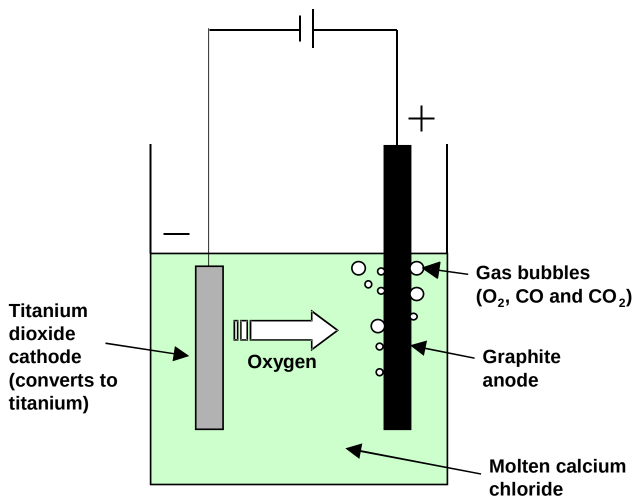


Fig. 10 Schematic Description of the FFC-Cambridge Process Reduction Step[13]

Status: A consortium consisting of TIMET, Cambridge University and QinetiQ, with additional team members Boeing and U.C. Berkeley has been awarded a contract from DARPA to develop and commercialize this process. QinetiQ has several 1kg cells operating and claims to be capable of producing powders of virtually any alloy. Powder size is approximately 100micron. Announced plans are to scale to commercial quantities in 2004.

Concerns: There has been considerable delay in moving to larger scale cells. In addition, little powder has been made available to outside entities in spite of announced intention to do so. Concern has been expressed over the electrolysis and chemistry fundamentals of the process. The cost of manufacture of the TiO_2 electrode, including cost of the TiO_2 itself, remains a concern, as does the cost of reduction of the reduced mass to usable powder. The long process time required to reduce the electrode to Ti metal also limits the potential for low cost.

4.2 Armstrong Process: International Titanium Powder

Process Description: This process produces Ti by the reduction of $TiCl_4$ with sodium, as does the Hunter process. However, ITP has devised a nearly continuous process in contrast to the batch mode of Hunter. A schematic of this process is shown in Figure 11. There are several key points which must be understood. The reaction is continuous and takes place in the “Ti Reactor.” Liquid sodium is pumped through a cylindrical chamber containing a centerline second tube. $TiCl_4$ vapor is injected into the sodium stream through this inner tube/nozzle. Reaction occurs immediately downstream of the nozzle, with Ti powder being carried out in the excess Na stream. Ti, Na and NaCl are separated by filtration, distillation and washing. The powder produced has a purity level near to that of commercially pure Grade 1, including a Cl content of less than 50 ppm. The pilot plant with a full scale reactor has achieved oxygen levels less than 1000ppm. By simultaneous reduction of other metal chlorides, it is possible to produce alloy powders..

Figure 11 shows several recycle steps either in the present small scale and pilot plant, or envisioned for a future integrated plant. The Camano cost study[14] also investigated this process and concluded that the “most probable” scenario produced Ti powder at near the present “cost” (should have stated “price”) of sponge ($3.54/lb). An “Optimistic” scenario, which includes recycling of NaCl and an integral $TiCl_4$ reactor would produce powder at production cost of $2.15/lb. This latter scenario assumes that $TiCl_4$ can be produced at a cost below its purchase price from outside vendors. These cost estimates, however, do not include the profit or SG&A required by a business enterprise. On the other hand, production of a quality Ti powder at this price level is a great improvement over the current cost of Ti powder, which may be in the range of $20 –40/lb.

Status: ITP is in the process of running a pilot plant to refine operating parameters and separation techniques in a continuous mode. The Ti reactor is capable of operating at ~2 million pounds per year rate for one hour. Scale up beyond that level would involve larger tankage and multiple reactors of the same design. Economies of scale would likely come from integrating some of the auxiliary processes. Product from pilot production runs has been thoroughly tested and analyzed to characterize its quality, morphology, and particle size distribution. Downstream melt processors have tested and verified good performance of ITP powder samples. ITP is working with powder processors on both process and powder improvements to optimize the use of the company’s powder in their processes.

Concerns: This process has been demonstrated to produce useable powder, and is close to commercialization. Remaining issues include demonstrating equipment durability, optimization of the separation equipment, and determining the capital cost of a fully integrated plant. Development of the processes to be used for producing the particle size and morphology required for product applications must be completed, and their cost determined.

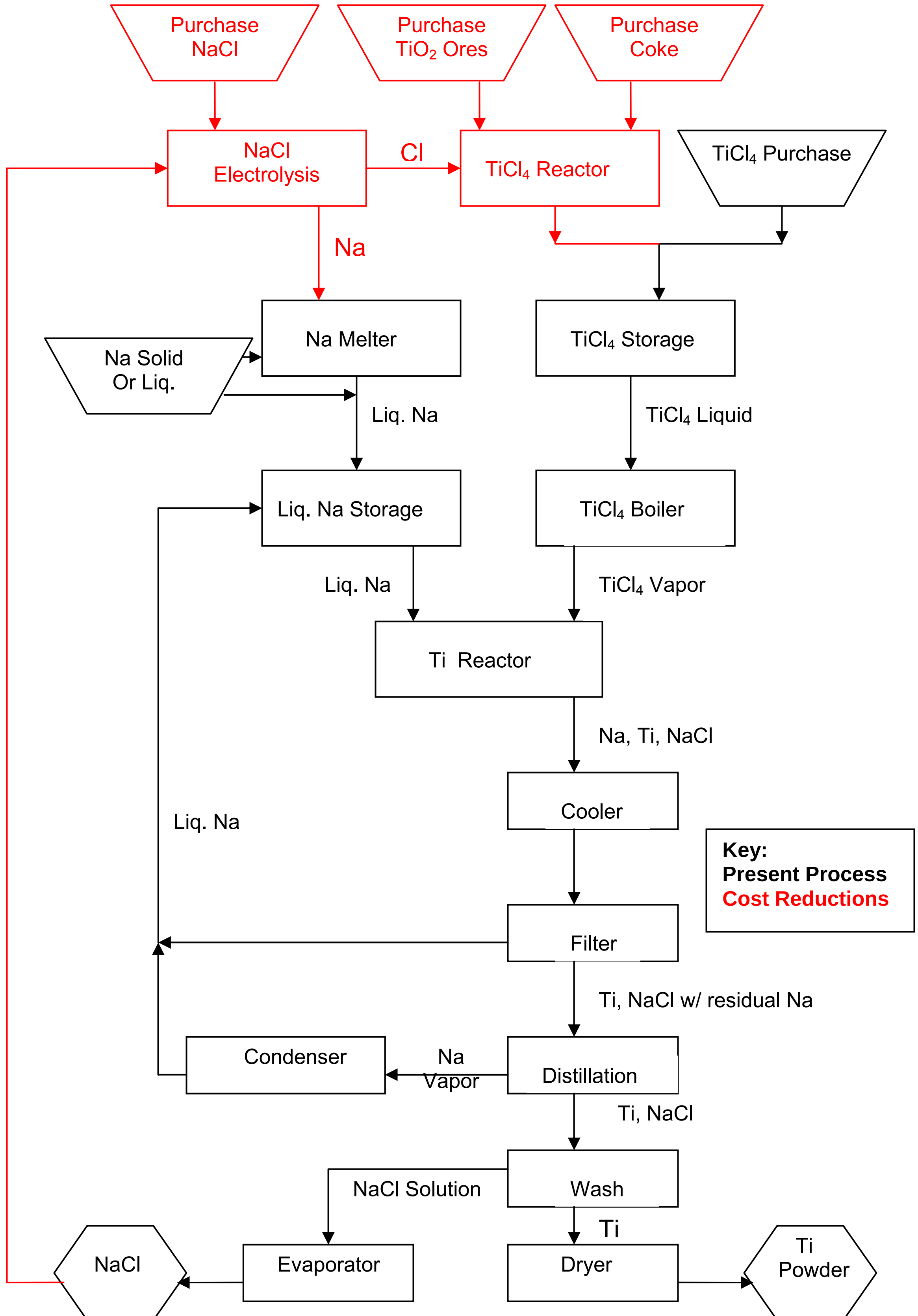


Fig.11 Armstrong / ITP Process Flow Diagram [15]

4.3 MER Corporation

Process Description: MER has developed an electrolytic reduction process which is significantly different than others. This process utilizes an anode comprised of TiO_2 plus a reducing agent, and an electrolyte of possibly mixed fused halides. Related background of the anode technology, applied to Mg and Al is provided in a set of expired patents. [16] A schematic of the process is shown in Figure 12[17]. TiO_2 or Rutile powders are mixed with carbonaceous material and binder, molded into electrode form and heat treated to form a composite anode. Ilmenite could be used for iron containing alloys if the other impurities could be tolerated. The composite anode contains a reduced TiO_2 compound as Ti_xO_y-C. Ti+3 ions are released into the electrolyte, are further reduced and deposit as Ti solid on the cathode. A CO / CO_2 mixture is released at the anode. The Ti can be deposited as powder, flake or a solid deposit as shown in Figure13. The form of the deposit is determined by salt composition and operating conditions. Powder with particle size from 1 to 125 micrometer has been produced, and larger particles are believed possible. The mean particle size can be controlled by process conditions, but the achievable particle size distribution for any one mean size has not been determined. An alternative to producing powder is the direct production of solid form (Fig.13, c)). The density of this solid form has not been reported. However, after some intermediate treatment, could conceivably be subsequently worked by conventional mill processes, avoiding the melting and ingot breakdown steps.
Status: MER has been awarded a DARPA contract for development of the process. One objective of this project is to produce billet with 300 – 500 ppm oxygen, suitable for mill processing. Current cell size or production rate is not known. Other team members in this contract are not known.
Concerns: While impurities are reported to be low, analytical confirmation is necessary. Processing cost needs to be determined. Consistent production of any particular product form remains to be demonstrated. Deposition of solid deposits with density, uniformity and configuration suitable for mill processing needs to be confirmed. As with most of the emerging processes, scale up, product acceptability and economics need to be developed.

4.4 SRI International [18]

Process Description: This process utilizes a high temperature fluidized bed to convert $TiCl_4$ and other metal chlorides to Ti or alloy which is deposited on a particulate substrate of the same material. [18] Particle size is flexible from microns to over 1mm diameter. Particle size distribution is reported to be narrow, and appears to depend on the particulate substrate feedstock. One experimental run of this process during laboratory exploration experiments, using Al_2O_3 particles as a substrate is shown schematically in Figure14. Product of a different run using Si microspheres as substrate is shown in Figure15. Eventual production of Ti powder would use any Ti or Ti alloy particulate substrate. This substrate would be produced by crushing about 1% of the product to smaller size and fed back into the reactor. Feasibility of alloy production has been demonstrated. Recent effort has focused on equilibrium calculations required to define the optimum experimental conditions for deposition of Ti and Ti-Al-V alloys.

Control of impurities is expected to be excellent, with purity analysis being investigated. Alloy of broad chemistry, including many metals may reportedly be produced.

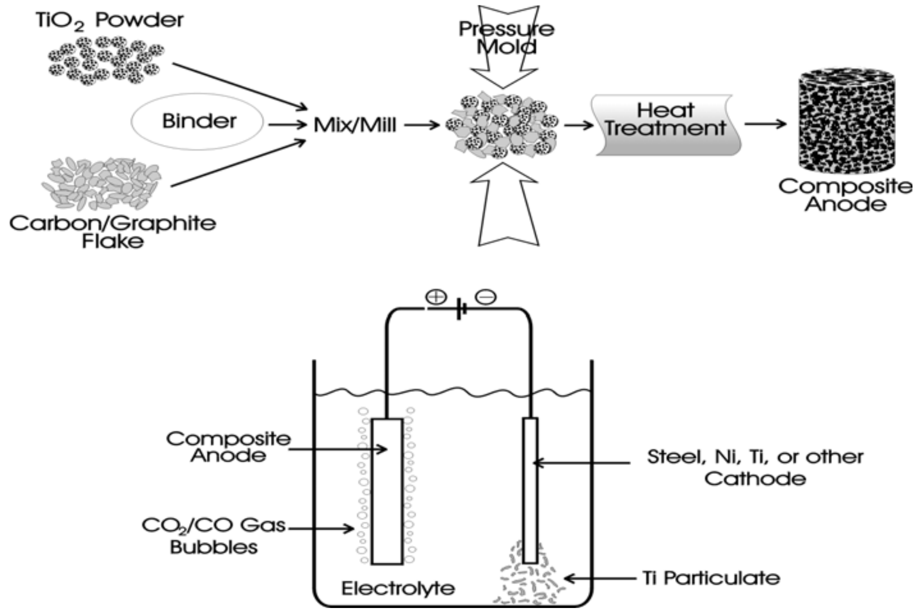
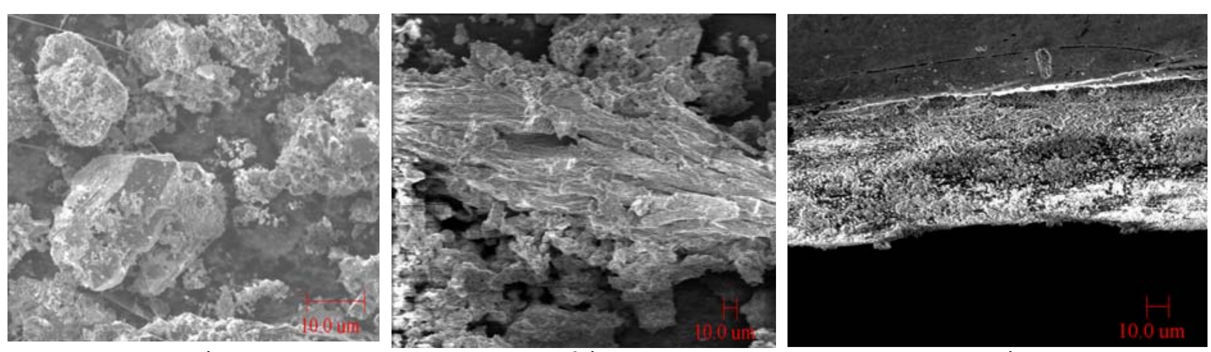


Fig. 12 Schematic of the MER Composite Anode Process[17]



a) b) c)

Fig.13. Titanium deposits in, a) Particulate, b) Flake, and c) Continuous form, produced by various salt compositions and operating conditions of the MER Process[17]

Status: The process is in early lab stage of investigation. SRI has been awarded a DARPA contract for development. Near term efforts will focus on: study of composition and microstructure of metal powders, rates of growth, and extent of reaction; purity (O, C, N) analysis; continued work exploring agglomeration in larger, taller beds; study of products recycling and/or disposal; reactor design; cost; production of powders for testing. At present there are no other participants on this team. However, discussions are being held with major players. Bench scale effort is scheduled for 2004, with pilot plant construction and operation during 2004-2006, and industrial production beginning in 2006.

Concerns: The economics of production in view of the need for particulate substrate, use of $TiCl_4$, and the unknown energy efficiency vs. deposition rate must be determined.

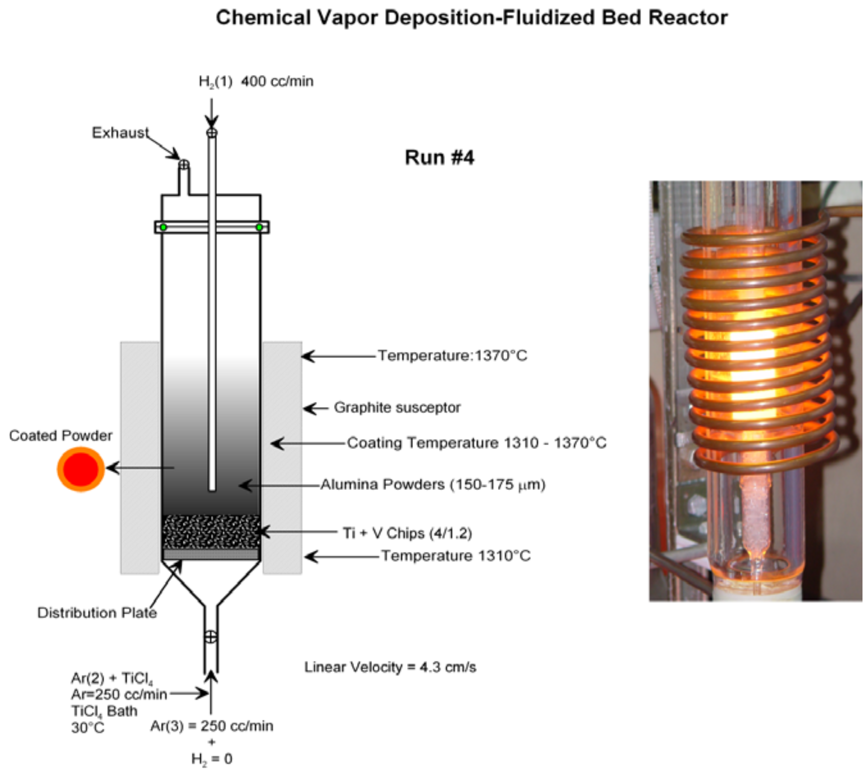


Fig. 14 Schematic of SRI International Ti Powder Production Process.

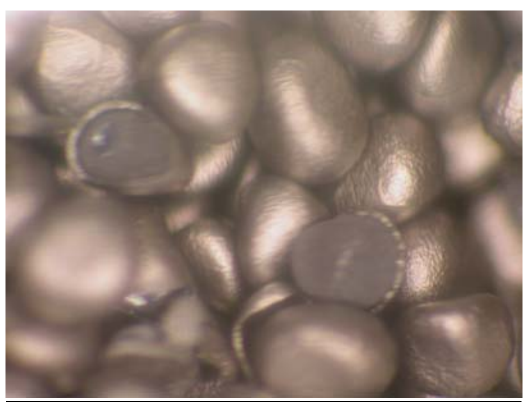


Fig. 15 Ti-Si-V alloy deposited on 23 μm Si spheres with the SRI International Process.

4.5 BHP Billiton [19]

Process Description: BHP Billiton is the world's largest diversified resources company. They are an industry leader or are in near industry leader positions in major commodity businesses, including aluminium, energy coal and metallurgical coal, copper, ferro-alloys, iron ore and titanium minerals, and have substantial interests in oil, gas, liquefied natural gas, nickel, diamonds and silver. This includes extensive expertise in mineral sands extraction, beneficiation, and in steel, aluminum and copper production. Little has been publicly released on the BHPB process development. Their process is announced to be based on the electrolytic reduction of TiO_2 in a $CaCl_2$ based bath.

They are expending significant effort on understanding the fundamentals of the process. Their aim is to achieve commercial production by 2009.
Status: Present scale of this process is designed to prove feasibility and understanding of process fundamentals. Small quantities of titanium metal have been produced. A 1kg/hr sub-pilot scale facility is currently being built which will prove the feasibility of their production concept, provide additional process fundamental understanding and provide engineering data for design of a production facility. It will also provide sufficient material to fully develop the auxiliary processes including fabrication. The sub-pilot facility is expected to be completed by early 2004.
Concerns: As with all of the electrolytic processes in molten salt, initial concerns involve the ability to achieve very low levels of chloride. Economic achievement of adequate oxygen levels is also to be demonstrated. Also as with all of the emerging reduction technologies, the economics of the overall process remains to be demonstrated. The BHP Billiton approach is understood to differ substantially from other EDO processes and the 2009 target date is believed to be feasible.

4.6 Idaho Titanium Technologies – Titanium Hydride Powder [20]

Process Description: ITT is the licensee for Ti applications from Plasma Quench Technologies Inc. the patent holder of the basic technology which was spun out of INEL in 1994. This process involves the thermal dissociation and reduction of $TiCl_4$. To accomplish this, it passes $TiCl_4$ through an electric arc in a vacuum chamber, which heats the vapor to over 4000°K forming a plasma. A stream of hydrogen carries the gas through a Delaval nozzle, where it expands and cools. The combined effect of rapid cooling (quenching), the reducing effect of hydrogen and formation of HCl prevent back reaction of the Ti and Cl. A very fine hydride powder is therefore produced by the basic reaction.
Status: ITT has been working on a NIST ATP grant (10/1/01-9/31/04) to develop the technology to increase this particle size to the range of 50-300micrometers, where it can be used in more conventional powder metallurgy based processing. As of this report, success has been achieved in producing spherical, apparently non-porous, powder in the 1 – 10 micrometer range with "narrow" particle size distribution. The developers believe particle size can be manipulated as desired. No impurity analysis is available. However, due to the use of a closed system and inclusion of hydrogen in the process, oxygen is claimed to be very low. Likewise chlorides are believed to be very low. Remaining chlorides are expected to be removed in subsequent vacuum sintering. No consideration has been given to production of alloy powders. Powder is expected to be available for testing by early Fall 2003. The new technology maintains the continuous mode of process operation, and the ability to start and stop production at will. Simultaneous to addressing the particle size issue, the reactor durability and energy efficiency have been improved. Current production rate capability is 40lb. / hr.

Cost factors which are claimed for this process include simple and therefore low cost equipment and low labor content. Prior to the current improvements, Camanoe [14]estimated the "process cost" at mid-value, or "likely current" scenario, cost of ~$3.26 / lb., just below the recent world market price for sponge. To this must be added normal

business costs, which normally add up to 40% to manufacturing costs. The effect of recent process changes on cost is unknown.
Concerns: The product of this process, being a hydride, may have advantages in powder metallurgy manufacturing of some discrete components; it will be of limited use, however, without dehydriding, for production of general mill products. Use of $TiCl_4$, while providing a purification method, also places a cost burden on the process, which some other technologies are seeking to eliminate. While previous serious concern about small particle size has apparently been successfully addressed, the goals of the NIST program for 50-300micrometers is consistent with industry need, and needs to be demonstrated. Likewise, careful analysis of impurity levels is required to meet commercial needs. To achieve minimum cost, the HCl product of the reaction must be economically recycled or sold.

4.7 Ginatta

Process Description: Dr. Ginatta developed the fundamentals of this process as a thesis at Colorado School of Mines, and has continued development through several different production concepts. In the 1980's methods were developed to electrolytically produce solid Ti deposited on cathodes which were periodically removed, thereby providing a continuous process. [21, 22] This technology was supported in part by and licensed to RMI from 1985 to 1991. It reportedly reached production of 70 tons / year in 1985. [23] Engineering issues related to multivalency and liquid metal production resulted in high production cost. In 1992, these issues and a market downturn caused RMI to withdraw from the project. Various issues with this earlier technology were addressed by Ginatta, resulting in a new concept which produces Ti liquid. [24-26] This is an electrolytic process, in which $TiCl_4$ vapor is injected into a halide electrolyte where it is absorbed. A "multilayer cathodic interphase" separates the molten Ti cathode from the electrolyte. This multilayer phase consists of ions of K, Ca, Ti, Cl, F and some elemental K and Ca. The layers contain various oxidation states of the species, with the bottom layer producing liquid Ti, which falls to the molten pool. Ti is contained by a water cooled Cu crucible, so that a frozen layer of Ti at the bottom and slag around the electrolyte provide insulation and protection from halides. Reportedly, solid scrap Ti and alloying elements can be introduced either through solution in the $TiCl_4$, or by solid metering via a screw feeder. The solid Ti layer may be allowed to grow either within a fixed cell geometry, or by using a movable hearth. In either case, the hearth is lowered and the solidified slab and electrolyte may be removed, as shown in Figure 16. Start up time for a subsequent batch is reported to be only 6 minutes. [27] Another claim of this technology allows liquid Ti to be tapped from the reaction vessel into a separate chamber. In this configuration, one could envision its use to provide liquid metal for castings, or to feed a succession of slab or billet molds. In such case, it could be considered a continuous process.

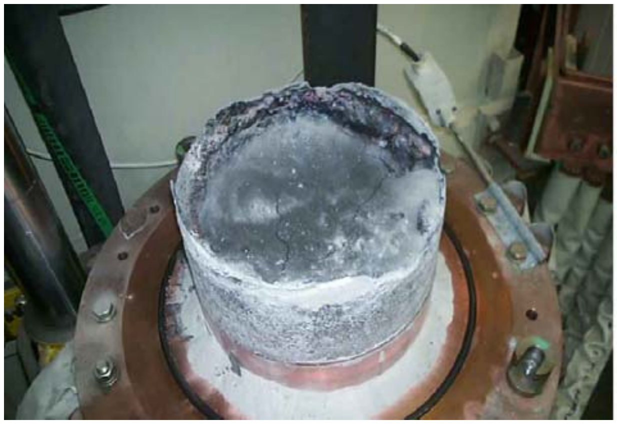


Fig. 16 Solidified Electrolyte and Ti Cathode from Ginatta Process. [26]

Status: The current pilot plant produces 250mm diameter ingot. Production of slabs 1 x 4 x 0.5 meter is being considered.
Concerns: The process is quite complex, so is not likely to be duplicated by others. The engineering issues which forced closure of the earlier effort are reported to be not as severe in the current high temperature cell. Confirmation of the product quality would be advisable. The use of $TiCl_4$ is a limiting factor on cost reduction if its production is not integrated into the process. Likewise, Cl gas must either be disposed, sold or recycled. No cost study appears to be available. If operated in the batch mode, start up and shut down costs would add to production cost. If operated in the liquid feed mode, the ability to start and stop the liquid stream needs to be demonstrated. The ability to add alloy elements or scrap and achieve a uniform solid appears very difficult; in a liquid feed mode, a steady state composition may be achievable, but this too must be demonstrated.

4.8 OS Process

Process Description: Professors Suzuki and Ono of Kyoto University have investigated the details of the calciothermic reduction of TiO_2 and developed a process for Ti production that is proceeding toward commercialization. In their most recent publications[28-32] they provide details of the mechanism of reduction of TiO_2 in Ca / CaO / $CaCl_2$ solution baths. A schematic diagram of an experimental setup for practicing the process is shown in Figure 17. At 1173ºK, $CaCl_2$ can dissolve 3.9 mole % Ca, but about 20 mole % CaO. Electrolysis is carried out above the decomposition voltage of CaO, but below that of $CaCl_2$. In this process, Ca^{+2} is reduced to Ca at the cathode, and O_2 is produced at the anode, combining with C to form CO / CO_2. It was found that when TiO_2 particles are in contact with the cathode, low oxygen Ti can be produced, whereas if the particles are electrically isolated, only suboxides are produced. This behavior is attributed, at least in part, to the high concentration of Ca on the cathode. 2000 ppm oxygen was achieved in 3 hours, 420 ppm oxygen was achieved in 24 hrs, and less than 100 ppm was achievable, at presumably longer times. Product of the reduction is lightly sintered granules. Optimum bath composition was found to be a

CaO composition in the range 1-6 mole %; higher CaO contents were found to slow reduction due to slower dissolution of the CaO reaction product. A cell design for continuous production of Ti has been proposed, and the possible use of inert anodes discussed. Formation of powdery carbon at startup of the bath was described, and attributed to initial Ca reduction of dissolved CO_2. After startup, carbon is deposited only at the anode.

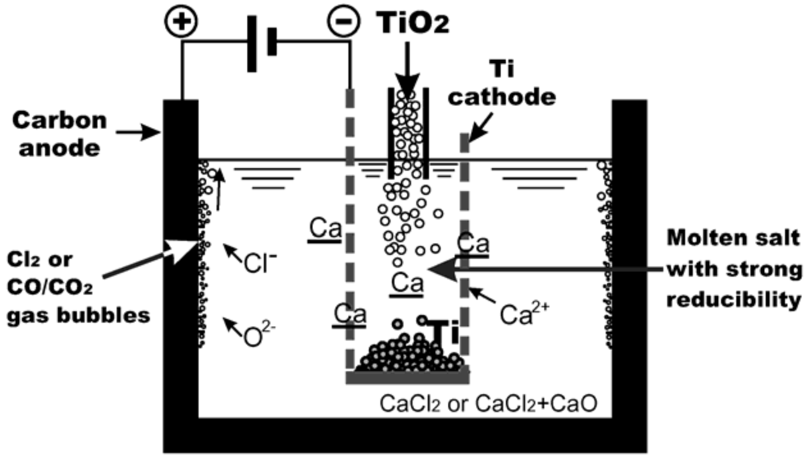


Fig. 17 Schematic of OS Calciothermic Process for TiO_2 Reduction. [29]

Status: Industrial application of the process has begun in collaboration with a Japanese aluminum smelting company. However, it is reported to require solution of many issues before quality product is available on a large scale. Operation of a “mud” covered bath in air has succeeded in preserving the reduced Ca. Ti production in this bath is being addressed.

Concerns: The production of low oxygen Ti in reasonable times and moderate cost with this process appears feasible. However, separation of the Ti product from the bath constituents, and purification to very low Cl level is still being addressed. In addition, the processing of the Ti lump into usable form, other than as melt process feed has also not been addressed. These operations could add considerably to cost. Finally, the reduction has been discussed as a purely calciothermic process. However, the possibility of involvement of electrolytic reduction of suboxides produced by the calciothermic process has not been addressed.

4.9 Millennium Chemical [33]

Process Description: Millennium Chemical is the world's second-largest producer of titanium dioxide (TiO2) and the largest merchant seller of titanium tetrachloride (TiCl4) in North America and Europe. As such, they have a strong position in intermediate feedstock for both TiCl4 and TiO2 based Ti metal production processes. Millennium is investigating options that allow it to vertically integrate from its position into a producer of titanium metal products. They are developing a process that produces Ti and Ti alloy powder.

Status: No details of the process or its current status are available. No announcement is expected before about mid 2004.

Concerns: It is difficult to formulate concerns until more is known of the process.

4.10 MIR-Chem

Process Description: Little is published on the collaboration of MIR-Chem and the University of Bremen. According to a July 2003 presentation at the Ti-2003 Conference in Hamburg[34], this group is developing a process based on the equation:

$$TiO_2 + 2\ I_2 + 2\ CO = TiI_2 + 2\ CO_2$$

The process is carried out on titania granules held in a “shaking reactor,” where oscillating patterns of particles, similar in appearance to moiré patterns, occur. This quasiperiodic pattern, termed a Faraday hydrodynamic instability, is the parametric excitation of surface waves via vertical oscillations of a flat bottomed-container filled with liquid. As a result, patterns appear on the surface, and in the case of a slurry, induces the pattern in the particles. High energy impact between particles provides the energy required for the reaction to proceed. The reaction mechanism is also termed a “tribo-chemical reaction.” In the formation of TiI_2, the process dwell time is on the order of four days. Following this reaction, TiI_2 is thermally dissociated to Ti and I_2, and the iodine recycled. More details may be available in the printed version of this paper.
Status: Unknown
Concerns: The dwell time of this process may appear very long for achievement of low cost. No data is available at present on product characteristics or projected cost.

4.11 CSIR [35]

Process Description: South Africa is one of the primary suppliers of titanium ores. As such, it has a strong interest in promoting use of titanium, and in increasing the added value of its minerals. CSIR is a South African science council operating as a market-oriented contract and consortium research partner to its clients and stakeholders. It has developed the fundamentals of a process to produce Ti from $TiCl_4$ and hydrogen as a reductant. Pure Ti in sponge form is planned as the product. Preliminary cost estimates indicate pricing competitive with the minimum production costs of Ti sponge via existing Kroll plants.
Status: Proof of concept experimental work has been completed and preliminary patents filed. CSIR is also planning a consortium to develop titanium metal technology, and are anticipating approval of funding for the first phase of that development.
Concerns: It is too early in development to understand the concerns that should be addressed. Further understanding of the product and cost will be necessary to determine if the product will compete with Kroll sponge or the other new emerging technologies.

4.12 Quebec (Rio Tinto) Iron and Titanium

Process Description: Québec Iron and Titanium (QIT) with mining and smelting operations in Québec, Canada is recognized as a world producer of titania slags (Sorelslag, and UGS). QIT along with Richards Bay Minerals (RBM) with operations in South Africa insure a leading position of their parent company RIO TINTO in the TiO2 business. They have recently filed an International Patent Application[36] for the electrolytic conversion of titanium slag to Ti metal. The concept is shown from the patent application in Figure 18. The product of the process is liquid Ti, which may be cast into ingots, billets or molds. There are several variations of this concept, with different electrolytes, anodes and methods of operation. However, the primary concept consists of pouring molten salt electrolyte, such as CaF_2 into the chamber, then pouring in molten titanium slag which is allowed to settle below the electrolyte, followed by electrolysis. Solid electrolyte, slag and metal forms a self-lining protective skull on the walls and floor of the cell. This skull is a key feature of the process which solves the containment issue for such a corrosive combination melt. This practice is used in their large Electric Arc Furnaces (EAF) for smelting ilmenite. The electrolysis may be carried out in one or two steps. In the two step process, the first electrolysis step purifies the slag by removal of less reactive species such as Fe, Cr, Mn, V etc. Droplets form at the electrolyte / slag cathode interface, and due to density difference, fall to the chamber floor. This metal mixture collects and is removed through a tap hole. After this reaction is complete, the second step, operated at a higher temperature, electrolyzes the Ti from the slag, which also collects at the chamber floor and is removed through the tap hole. If the process is performed in only one electrolysis step a mixture of titania slag (Sorelslag) and upgraded titania slag (UGS) is used insuring that the total iron content is sufficiently low (1.4 wt.% FeO) to avoid requiring its removal. Otherwise, operation of the process is as described above. Molten titania slag can be supplied continuously to the chamber either by connecting the electrolyzer to an operating EAF without exposing the molten titania slag to the atmosphere or by feeding solid titania slag to the melt during continuous operation. Since many new low cost alloys for automotive and other markets have substantial iron content, this Fe level is no longer an issue. Other metal oxides may be added to the melt to obtain various alloys. For example, alumina and vanadium pentoxide may be added in order to obtain ASTM grade 5 (Ti-6Al-4V). Quality of titanium ingots is measured using the standard used to qualify titanium ingots (e.g., ASTM B265) as a main reference.

<u>Status</u>: Considerable work has apparently been done on the QIT process. Since data is included in the application for a variety of cell designs and operating modes, the final process configuration is based on multiple iterations of design concepts. No information is available, however, on current production capacity or plans for commercialization.

<u>Concerns</u>: As with other emerging processes, repeated demonstration of compositional control and other quality measures will be necessary. No cost analysis of the process is available.

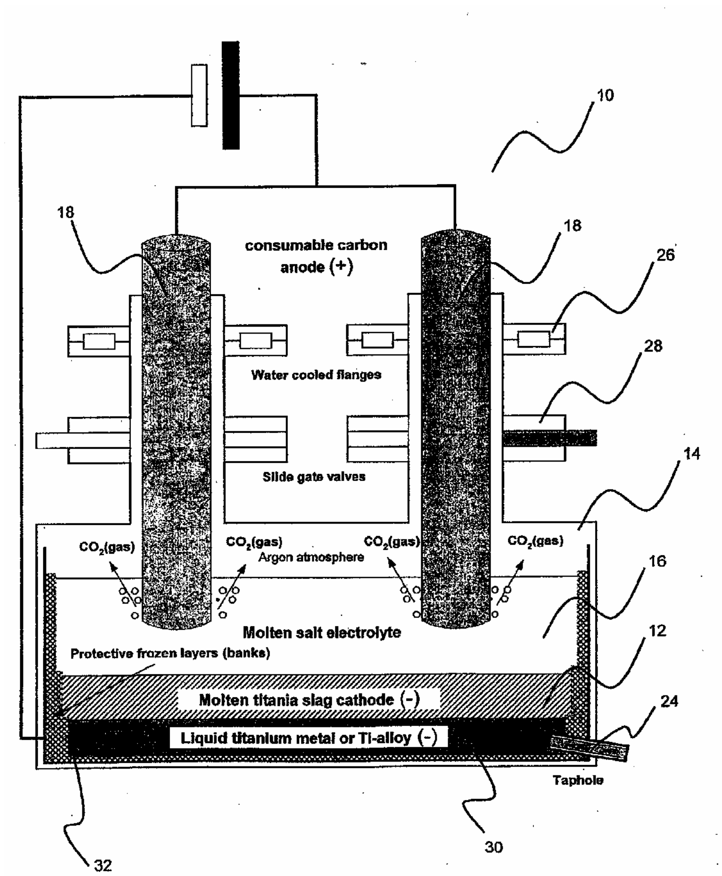


Fig. 18 Basic Concept of QIT Electrolytic Ti Production[36]

4.13 EMR/MSE Process (University of Tokyo): Electronically Mediated Reaction / Molten Salt Electrolysis [37]

(Prof. T. Okabe is a graduate of Kyoto University, where he studied under Prof. Ono [see OS Process]. His own group at Tokyo now studies electrochemical and metallothermic processing. He has developed two new processes for TiO_2 reduction.)

Process Description: This process is shown schematically in Figure 19. TiO_2 powder or a preform is placed in a holder shown on the left side. A Ca + 18 mass % Ni alloy is placed in the bottom of the reactor, and a carbon anode is provided. During the reduction step, no current is provided to the carbon anode, but an electrochemical cell forms between the TiO_2 cathode and the Ca alloy "reductant." During this phase of the process cycle, TiO_2 is reduced and Ca ions are formed. Pure titanium is reported to be produced. Results on trials simulating the left (reduction) side of the cell have been conducted and produced titanium with impurity levels on the order of 0.15 – 0.2 wt.% Ca, 0.2 – 0.5 Fe, .04 - .16 Ni and .35 - .65 O_2. Process times were on the order of 2 to 4 hours. Microstructure of the Ti produced is shown in Figure 20. Interestingly, only about 5% of the charge necessary to reduce the TiO_2 present actually passed through the circuit. The mechanism of the process is under discussion.

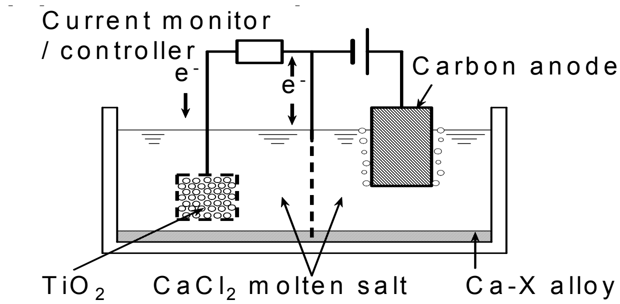


Fig. 19 Schematic of Okabe EMR / MSE Process [37]

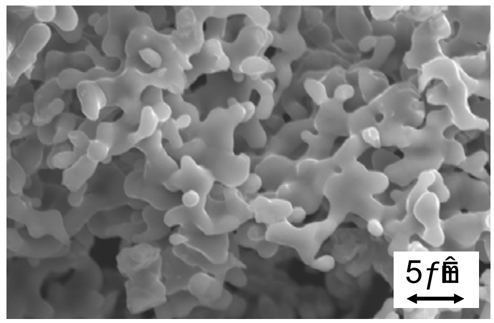


Fig. 20 Titanium Produced by the EMR / MSE Process [37]

The right side of the cell in Figure 19 is conceived as being operated at different times than the reduction occurring on the left side. At such times, Ca ions are expected to be reduced and the Ca alloy replenished.

Status: The process is in early stages of development. Reaction mechanisms are not determined.

Concerns: Until the current mechanism uncertainties are resolved and more work is done on the complete process, no assessment may be made.

4.14 Preform Reduction Process (University of Tokyo) [38]

Process Description: This process, also under development by Prof. Okabe, is shown schematically in Figure 21. TiO_2 and a flux of either CaO or $CaCl_2$ are formed into a preform and held with minimal contact in the space above a bath of molten Ca metal. The vapor (and flux?) react with the TiO_2, leaving Ti and CaO. Leaching and washing of the product produce titanium such as shown in Figure 22. Other temperature, fluxes and flux / TiO_2 ratios produced different powder size and morphologies. Ca content of the final product has not been sufficiently reported. Oxygen content is on the order of 2800 ppm. The mechanism of this process is under investigation.
Status: This process is in the early stages of development.
Concerns: Achievable oxygen and Ca contents need to be determined. Since the reaction is highly exothermic, adequate temperature control may make scale up of the process difficult. Concentration of reaction products and their recycle could be costly. No overall cost estimate is available.

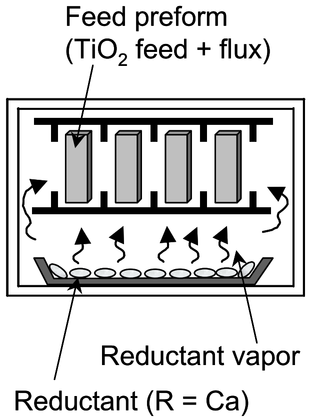
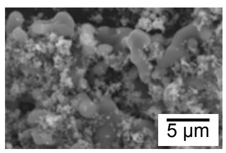


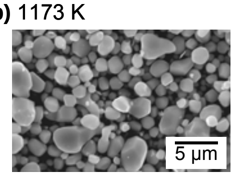
Fig. 21 Schematic of Okabe Preform Reduction Process [38]

(a) 1073 K



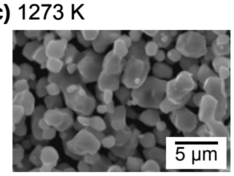
EDS analysis (mass%)				
Ti	Ca	O	Fe	Ni
82.85	9.19	7.63	0.15	0. 18

(b) 1173 K



EDS analysis (mass%)		
Ti	Fe	Ni
99.85	0.04	0.11

(c) 1273 K



EDS analysis (mass%)		
Ti	Fe	Ni
99.88	0.10	0.02

Fig. 22 Ti Powder Produced by Okabe Preform Reduction Process in 6 hrs and Ca/Ti Ratio of 0.5 [38]

4.15 Vartech, Inc. [39]

Vartech has received a Missile Defense Agency SBIR contract to develop a vapor phase process to produce titanium powder. The process uses $TiCl_4$ vapor and a gaseous reducing agent reacted in an inert atmosphere. The key objective of the project is to make powders at a "cost" of $3 – 5 / lb in large quantities. The process is in early stages of development and no additional details are available. It is therefore not possible to further describe the process, its status or concerns.

4.16 Northwest Inst. for Non-Ferrous Metals (NIN) – China [40]

Process Description: NIN is working to reduce the cost of hydride / dehydride powder made either from sponge, ingot or scrap. This effort is therefore not a new reduction technology, but is included here as an additional, or perhaps complimentary cost reduction process. Two approaches are being pursued. In the first, process efficiency

efforts such as fast crushing, automatic grading, gas protection and fast hydriding are being developed. The second effort is in developing a "motive HDH" process in which the material being hydrided is simultaneously being attrited to break up the 20 – 30 μm diffusion layer and thus speed the process. Combination of these approaches is expected to reduce process cost from ~$12.5 – 16.3/kg down to ~$2.4 – 2.9/kg.
Status: Pilot facilities are scheduled to be tested
Concerns: There is some concern with methods of raw material preparation. Success of the other new Ti reduction technologies could either make this process redundant, or could be viewed as candidates for application of the technology for comminution of their process product.

4.17 Idaho Research Foundation [41]

Process Description: This process has been termed "Mechanochemical Processing," since the reaction is energized by the mechanical energy of particle impingement by milling media, rather than by thermal energy. Powders such as magnesium or calcium metal, or their hydrides are placed in a milling apparatus such as ball, rod or attrition mill along with $TiCl_4$ liquid. Milling reportedly promotes the solid state chemical reaction. Calcium hydride is preferred as the product is Ti hydride. Patent data shows that Al and V chlorides may also be utilized to produce alloy powder.
Status: The concept has been demonstrated and the process is in the research stage.
Concerns: Use of $TiCl_4$ and other chlorides, and metallic or hydride reductant may lead to high cost. The ability to scale up to large quantity production would need to be demonstrated.

5.0 Developing Alloy and Product Technologies

A great variety of activities are taking place worldwide on many subjects related to reducing the cost of processing titanium, developing lower cost alloys and in applications technology. Only a few such activities will be discussed here, and the discussion will focus mainly on those topics of interest to vehicular and industrial applications. Discussion is limited to providing only a few of the key points available at the present time. Several recent or upcoming publications are of interest to those in this field. [42-45]

5.1 Alloy Development

- Comparison of ingot and PM routes to production of a Ti-10V-2Fe-3Al alloy showed that while UTS was similar, tensile elongation was higher for the PM approach, and tensile property variability was reduced by 40-60%. This finding has important implications for establishment of design allowables. [46]
- A particulate reinforced alloy, Ti-6Al-4V-2Mo-1Fe+10vol.% TiB_2, has been developed for engine valve applications. Processing uses hydride Ti powder, master alloys and boride particles. High matrix-boride coherence, in part due to close CTE match, produces double the fatigue strength of ordinary Ti alloys, with low wear and increased elastic constant. Valve use is limited to intake position due to temperature limits. ~0.5 million valves have been used in the Toyota Alzeta; high cost has limited further use. [47]
- A new type of alloy, termed "Gum Metal," has been developed with the following characteristics, and as illustrated in Figure 23:[48]
  - Extremely low elastic modulus with extremely high strength
  - Super-elasticity, capable of enormous elastic deformation exceeding 2.5%, displaying non-linear elastic deformation behavior (Hooke's Law does not hold true).
  - Super-plasticity that allows cold working of 99.9% or more without work-hardening.

  Composition is Ti + 25 mole % (Ta, Nb, V) + (Zr, Hf, O) and fabrication is via compaction of elemental powders. Applications include automotive springs, seals, diaphragms, medical and consumer products.

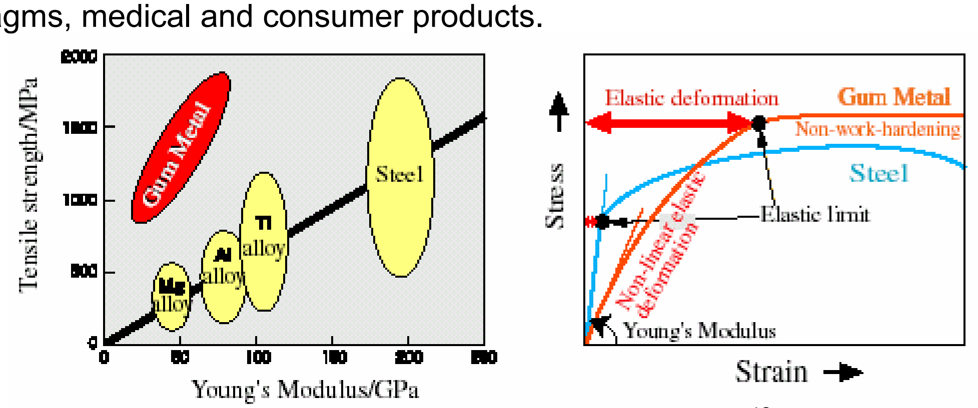


Fig. 23 Characteristics of Gum Metal[48]

- A new alloy for improved oxidation resistance and reduced cost has been developed for exhaust system applications.[49] Composition was optimized at 1.5% Al on a CP Grade 1 base. Objectives included workability equal to Gr 2, with high temperature strength and heat resistance greater than Gr 2. Ultimate and 0.2% yield strength are greater than Gr 2, with 0.2% proof stress equal to 304 stainless steel below 400ºC. Oxidation is only 2/3 that of Gr 2 at 700ºC. Strip has been made into welded tube, with less property degradation than Gr 2. For heat exchanger applications, it has hydrogen resistance superior to Gr 2.
- A low cost α/βalloy with improved machinability and properties equivalent to Ti-6Al-4V has been developed. Cost was reduced by design of the alloy to utilize scrap and low cost master alloy in a single electron beam melt process. Target applications include automotive forgings, armor and land based structures. [50]

5.2 Powder Consolidation

A majority of the emerging reduction technologies described in Section 4 are designed to produce powder. This powder may be usable in the existing titanium powder metallurgy industry. PM, however, represents only a few percent of the overall titanium industry. The reason is only partially explained by the high cost of current quality powders. Very few PM companies provide titanium parts. Explanations for this have included the high cost of powder, lack of familiarity by designers, inadequate sintering facilities and binder systems that result in high interstitial content in finished parts. Reference 1 describes activities by ADMA and Dynamet Technologies to develop powder based titanium business, which has been increasing. A recent review[51] of approaches such as these provides additional insight. It has also been reported that Advanced Forming Technology, a unit of Precision Castparts, is preparing to provide commercial titanium PM products.[52] Availability of new, high quality, lower cost powders can be expected to have a positive influence on the Ti PM industry, but the other factors are likely to restrict the rate of growth.

Little work has been done on methods of using powder to develop alternative routes to products such as bar, wire, sheet, plate and forgings. However, there appears to be considerable promise for significant cost reduction by process routes that avoid the costs of conventional melt and mill processing. Figure 2, above showed an estimate of the relative contributions of process steps to the cost of plate, and Figure 1 showed the reduction in process steps conceivable with the emerging direct reduction powder processes. The process arrow for direct powder, however, includes processes yet to be developed for consolidation and forming of powders into plate or sheet which can be rolled and heat treated to the desired end product. Even less attention has been given to use of these powders to reduce the cost of extrusions and forgings.

Work in the 1950's and 1960's at du Pont Company[53-57] demonstrated the feasibility of producing titanium plate, sheet and bar from powder. That work was abandoned, however, when welding was attempted on the resulting product without success. It was concluded that in order for this set of processes to be viable, chloride levels in the powder would need to be below 0.005% (50ppm), whereas available powders had chloride in the range of 0.01 to 0.05%.[58] du Pont also found that successful compaction required irregularly shaped particles rather than the spherical particles preferred by molding processes. Table 2 provides a summary of some of this work.

Table 2. Summary of Ti Alloy Pressing Procedures from duPont Patents.

Blend (m=mesh; 6-4=Note)	**Initial Forming (tsi=tons/in^2)**	**Partial Homogeniz.**	**Intermed. Reduction**	**Anneal**	**Final Cold Reduction**	**Final Homogeniz.**
.85 -60m Ti; .15 -270m 6-4	Direct to 0.060" strip	15min @1200ºC	To 0.010"	30min @1030ºC	To 0.003"	900ºC, 1hr + 600ºC, 1hr
.9 -60m Ti; .1 6-4	5x5x.35" @ 12.5 tsi; 73% dense	20min @1025ºC 77% dense	45-65% Reduction	30min @1065ºC	To 0.030" No porosity	1100ºC, 4hr
.9 -60m Ti; .1 6-4	Direct to 0.011" strip	15min @1000ºC 85% dense	To 0.002"	30min @ 1010ºC	To 0.001"	850ºC, 30min.
.9 -60m Ti; .1 -200m 6-4	2" dia. X 4" isopress @ 25tsi	15min @ 1065ºC	Heated 600ºC Argon; Extruded 4:1			1200ºC 1hr
.9 -60m Ti; .1 -200m 6-4	5x5x1" 12 tsi 73% dense	30min @ 1030ºC	1" sq. x 5" bar cold forged to .6"sq x 10" @ 20 to 60tsi 100% dense	30min @ 1000ºC	2 bars cold rolled & 2 @600ºC to 0.3" dia rods	1200ºC 15min
.730 -60+200 Ti; -20+325 m .13V, .11Cr, .03Al	Direct to 0.027" strip	15min @ 1300ºC Ar; induction 89% dense	Cold rolled on 2-high mill to 0.020"	15min @ 650ºC	Cold rolled on 4-high to 0.010"	1300ºC 15min Beta
282pts Ti - 60m; 6pts -60m Cr; 6pts-100m Fe 6pts-100mMo	Direct to 0.008-0.010" strip	15min @1200ºC He 90% dense	Cold rolled to 0.005-0.007" 100% dense	15min @ 1200ºC He	Cold rolled to 0.001"	450ºC 1hr

Note: 6-4 is 60%Al, 40%V master alloy powder

This work reportedly produced product of comparable microstructure and properties to conventionally processed alloys, other than the high chloride content. Use of the elemental or master alloy blends to produce completely homogenized alloy was established.

5.3 Solid Freeform Fabrication

The subject of solid freeform fabrication has received great attention, with application to a wide variety of materials, and with many processes. A few of the activities applying these techniques to titanium are as follows:

- Electron Beam Melting: An electron beam, controlled by a CAD file, scans the surface of a powder bed, fusing the material only in the area of the desired part. Layers of powder are sequentially spread and fused as above to create a 3D structure, as shown in Figure 24.[59, 60]
- A similar process, but with the use of a laser as the heat source has been used successfully by Trumpf to produce complex shapes such as that shown in Figure

25.[61] Figure 26 shows the principle of an AeroMet Laser Additive Manufacturing unit.

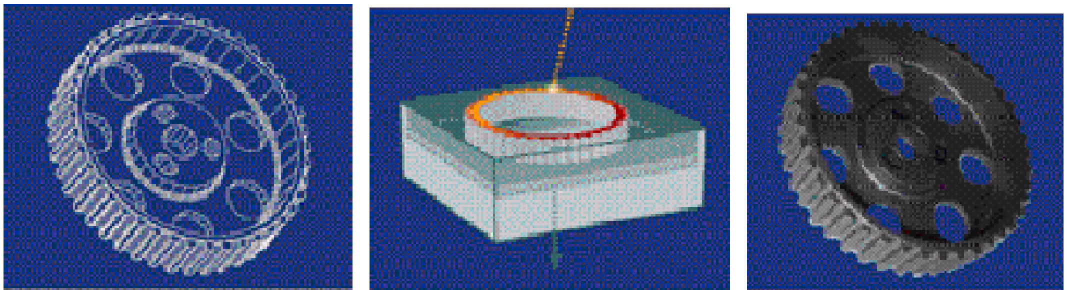


Fig. 24 STL File, E Beam Process and Finished SFF Part [59]

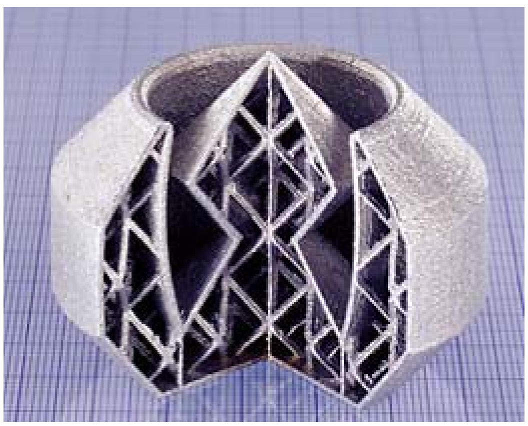


Fig. 25 Complex Ti-6-4 Parts by Trumpf Laser Melting Technology [61]

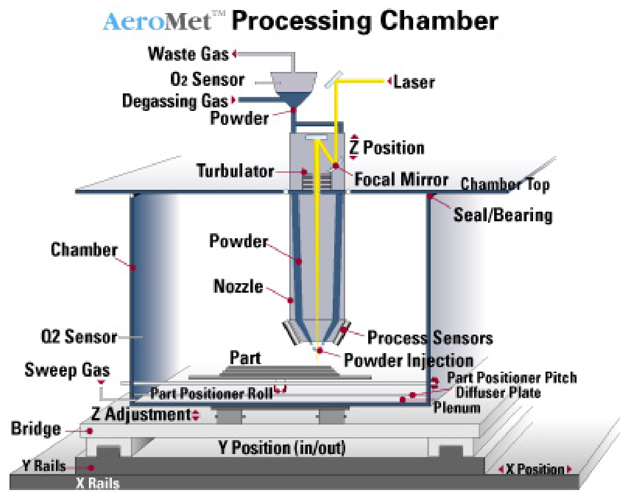


Fig. 26 AeroMet Laser Additive Manufacturing Using Powder [62]

- Laser Precision Metal Deposition uses flat wire fed into a melt pool formed using a laser as shown in Figure 27. [63, 64] This process is used to build up portions of a structure on a substrate in order to avoid extensive machining away of unwanted material. The process is entering production in aerospace manufacturing.
- The Plasma Transferred Arc (PTA) process is being developed and demonstrated for production of titanium components by MER Corp.[65]. The process is shown schematically in Figure 28, along with a titanium deposit. Plasma transferred arc was selected as a heat source for expected advantages in deposit purity, capital and operating cost, and build rate. Tensile properties in PTA deposits comparable to cast, wrought and laser deposited Ti-6Al-4V alloy have been demonstrated. Ti has been deposited on steel directly and with Ta, Nb, V or Ni intermediate layers. Ti-6Al-4V-WC cermet has also been deposited on Ti-6Al-4V to provide wear resistant surfaces on Ti alloy structures. Near net shape preforms have been used to produce a variety of components.

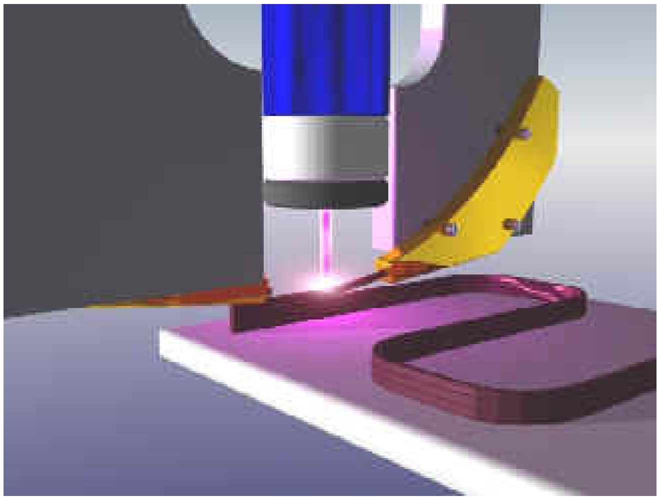
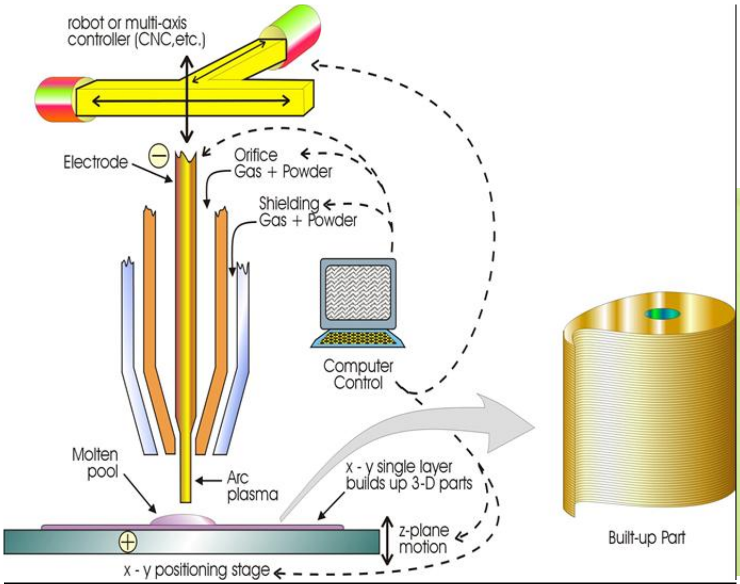


Fig. 27 H&R Technologies' Laser Flat Wire Deposition [64]



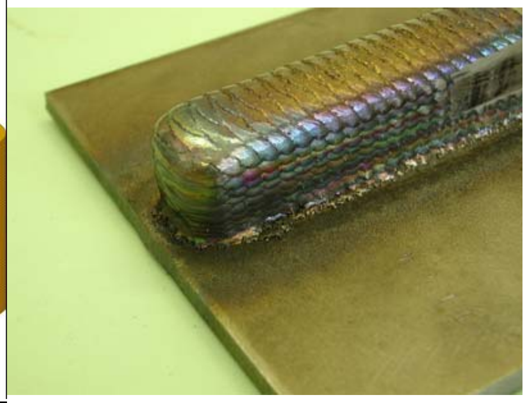


Fig. 28 Schematic of MER Corp. Plasma Transferred Arc SFF Fabrication Process and Deposited Ti-6Al-4V Alloy Preform.[65]

5.4 Applications

A list of new applications for titanium would be lengthy enough to deserve a dedicated study. No attempt has been made at such a comprehensive list, other than for heavy duty vehicles[1]. However, a few technology and applications papers from the Ti-2003 Conference are of interest to ground vehicles.

- γ-TiAl turbocharger rotor has been used in the 1999 Mitsubishi Lancer Evolution VI, RS version used in racing applications. [66, 67]
- Laser deposition is also being used to fabricate performs for titanium forgings. [68]

- A pilot plant has been commissioned for production of γ-TiAl engine valves. The process uses cold wall crucible induction melting and centrifugal casting with 50 valves / mold. Molds use Nb inserts. The system has capacity for 600,000 parts / year using one operator. Over 200 casting trials have been performed on 10 different valve types for 5 automotive companies. Engine testing is planned. [69, 70]
- TiAl turbocharger rotors, using Howmet XD45, are being considered for mass production Daimler-Chrysler autos beginning in ‘05/’06, providing development and cost efforts are successful. [71]

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