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Development of Titanium Processing Technology in the USA

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An overview of the progress in titanium processing technology in the USA is presented. Specific topics discussed include extraction processes for powder and sponge, single melt technology, non-melt processing for mill products, processes for making shaped components (casting, superplastic forming, extrusion, flowforming, powder metallurgy, free form fabrication and powder injection molding), welding, and process modeling.

Keyword: sponge, powder, single melt, non-melt processing, shaped component, casting, superplastic forming, extrusion, hot stretch forming, hot roll forming, flowforming, powder metallurgy, free form fabrication, powder injection molding, hybrid welding, friction stir welding, process modeling

1. Introduction

In the last few years the world's titanium market has grown rapidly. In Asia the fastest growth is in titanium's industrial applications, while in the USA most of the growth is driven by the needs of defense and aerospace, such as the new wide body aircraft Airbus A380 and Boeing 787. The industrial and defense/aerospace applications may require different grades of titanium, but they have the need for lower cost in common. For this reason titanium technology has focused more on the development of lower cost processing techniques rather than the new alloy development for performance enhancement. This paper surveys the progress of processing technology achieved in the USA over the last several years.

2. Production of Titanium Sponge and Powder

Near term market demand for titanium is addressed by increasing the production capacity for the standard Kroll sponge. On the other hand, significant efforts are being devoted to develop new extraction processes for making low cost sponge, and for making titanium powder suitable for making net shape products from titanium.

2.1 Capacity Expansion of Kroll Sponge

The producers of titanium sponge in the USA are all increasing capacity. Timet will increase its sponge production capacity by 4000 MT (40 percent) annually[1)] and Allegheny Technologies, Inc. (ATI) has restarted its sponge production facility in Albany, Oregon[2,3)]. This facility should produce about 9,000 MT annually soon, to be supplemented by a new sponge facility in Rowley, Utah, with an 11,000 MT annual capacity, for a total capacity of at least 20,000 MT per year. DuPont[4)] will supply all the necessary $TiCl_4$ for this new facility. The other major titanium producer in the USA, RTI International Metals, Inc. (RTI), will focus on high quality mill products and fabrication of titanium shapes, using purchased sponge.

2.2 New Sponge/Powder Processes

In 2004, DARPA (Defense Advanced Research Projects Agency) has started development of four promising processes to make titanium sponge or powder, by four competing teams. Table 1 summarizes the four processes[5-10)]. In addition, DuPont has established a 136-MT per year plan[11)] to make titanium powder, with a production process developed by Honeywell Electronic Materials that improves the percentage of powder in the final product compared to that of the Hunter process.

Table 1. Summary of DARPA funded new sponge/powder making processes.

Process	*FFC Cambridge*	*Composite anode*	*Armstrong*	*SRI*
Team Leader	Timet	MER Corporation	International Titanium Powder (ITP)	SRI International
Process Description	Electrolytic reduction of TiO_2 in $CaCl_2$ electrolyte	Electrolytic reduction of TiO_2-C composite anode in molten salt	Chemical reduction of $TiCl_4$ by Na	Hydrogen reduction of $TiCl_4$ in a fluidized bed
Product Form and Chemistry	Sponge Pure Ti and alloy	Powder Pure Ti and alloy	Powder Pure Ti and alloy	Particle Pure Ti and alloy
Current Status	No activity	Transition from laboratory scale to pilot plant. Building a 227 kg/day reduction cell. Teaming with DuPont.	Pilot plant capacity 100 kg-300 kg per week. End of 2007, 2000 MT annual capacity production plant. Product being evaluated for both melt and non-melt routes.	Still in lab scale

ADMA Products, Inc., has worked with its East Europe partner to develop two versions of modified Kroll process[12)]. One version is to add NaCl during Kroll processing to increase the percentage of powder product, and the other version is to add hydrogen for making titanium hydride powders.

The most important characteristic of these new sponge/powder making processes is that they can make products at a cost that is similar to, or even lower than, the cost of Kroll sponge. This characteristic provides three opportunities for these products to become commercially viable. First, if the cost of these products is actually lower than the cost of Kroll sponge, these products can be used as a sponge substitute for the conventional melting processes. Secondly, powders can be used to make titanium mill products by non-melt processing routes. The success of this approach can be a major landscape change for titanium mill products. More details about this approach are discussed in Section 4.2. Third, powders can be used to make shaped components by the conventional die pressing + sintering processes.

3. Melting

To meet the growing demand, all three major titanium producers in the USA (Timet, ATI, and RTI) are increasing their production capacity. All three also try to lower their costs by moving to single melt processes.

3.1 Higher Production Capacity

By the first quarter of 2008[13)], Timet should have finished a new 8,500 MT electron beam melting (EBM) furnace. ATI intends to build two new plasma arc melting (PAM) furnaces, and build six more vacuum arc remelting (VAR) furnaces[3)], and RTI will build one PAM and one VAR furnace[4)]. In addition, the bar and coiled rod producer, Perryman Company, will establish its own melting capability by building an EBM and a VAR[14)] furnace.

3.2 Development of Single Melt Processes

Making titanium mill products with the conventional VAR process is thought to be more expensive than with the various single melt processes. These have the benefits of reducing both input material and production processing cost. Development and implementation of the single melt processes has, therefore, been very active. For example, the new AMS 6945 specification applies specifically to Ti-6Al-4V flat products (sheet and plate) processed from single melt ingots or slabs produced by either EBM or PAM furnaces. Single-melt processes have already produced several thousand tons of plates and other products[15-19)]. In U.S. Air Force Metals Affordability Initiative, MMPDS-2 Design Allowables for static properties were generated and approved for all three suppliers[20)]. In addition, the equivalency of fatigue and fracture properties was also demonstrated, approved and implemented.

One development of single melt processes is the addition of a minor amount (~0.1wt%) of boron to refine the as-cast grain structure of the ingot/slab. Figure 1 shows the effect on single melt PAM ingots/slabs of the Ti-6Al-4V alloy with a uniformly distributed fine grain structure that improves the material's hot workability, reduces the tendency of the work piece to crack, and lowers the loss from machining and conditioning[21,22)]. Two issues to be concerned with for this technology are boron segregation in the large size ingots and the control of the boron containing scrap. This technology is still in the development stage.

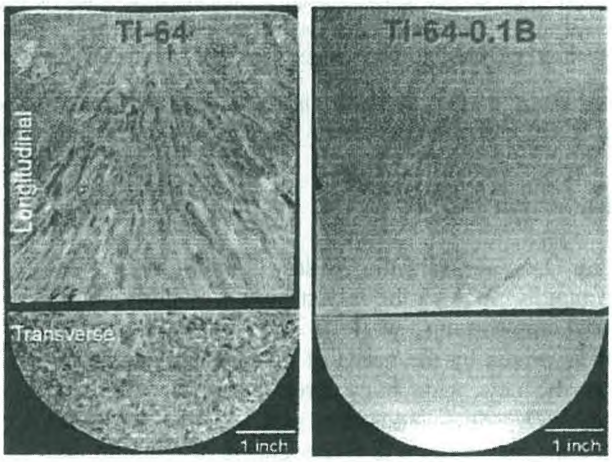

Figure 1. Micrographs of 127 mm diameter Ti-6Al-4V PAM cast ingots illustrating dramatic grain refinement produced by trace boron addition[22)].

4. Hot Forming

Advances in hot forming include lowering costs by producing mill products through direct rolling and extruding of the as-cast PAM single melt ingot and non-melt processing of powders. Another important activity is using process modeling to optimize the parameters of forging and rolling processes efficiently, see Section 7.

4.1 Direct Rolling and Extruding As-Cast PAM Single Melt Ingots

The billet needed to make rolled bars and extruded shapes is much smaller than the ingot made by the conventional VAR process. Therefore, the VAR ingot must be hot worked to become a smaller billet by various intermediate open die forgings before the last one can be rotary forged to a size that can be machined to the desired size. Each step in this series of processes takes time, demands inventory, loses material, and costs money. The PAM single melt process can cast small diameter ingots that only need a single machining step before they are in the right shape to make the final rolled bar or extruded shape resulting in a significant cost saving. Figure 2 compares the number of processing steps in the conventional VAR process with the newer PAM single melt process.

To date it has been found that this approach is very promising. For example, Yu showed that the microstructure and mechanical properties are essentially the same for Ti-6Al-4V and Beta C^{TM} bars rolled from conventional VAR billets[5,23)] as from as-cast PAM single melt ingots. As-cast PAM single melt ingots were also used to make extruded shapes and square/round tubes.

4.2 Non-Melt Processing for Mill Products

Titanium powders are typically used to make shaped components by die pressing plus sintering and cold isostatic pressing (CIPing) plus sintering routes. However, recent developments of new sponge/powder processes appear to make the availability of large quantity of low cost titanium powders become a reality. This resulted in an increased interest in making mill products from non-melt processing of titanium powders[12,24-26]. The most commonly studied non-melt processes are:

- Cold die pressing + sintering + rolling;
- CIPing + sintering + rolling;
- Hot isostatic pressing (HIPing) + rolling;
- Direct powder cold rolling + sintering + rolling;
- Vacuum hot pressing; and
- Extruding + rolling.

Products made from these processes include sheet, plate, bar, billet and shapes. Figure 3 is a schematic of what is involved in direct powder cold rolling followed by sintering and then hot rolling.

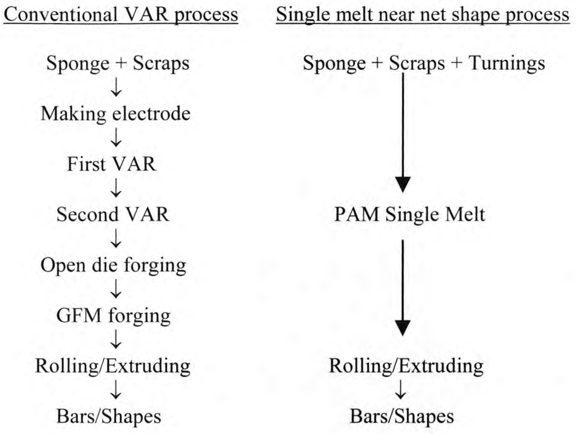

Figure 2. Comparison of processing steps for conventional VAR process and PAM single melt near net shape process for making rolled bars or extruded shapes.

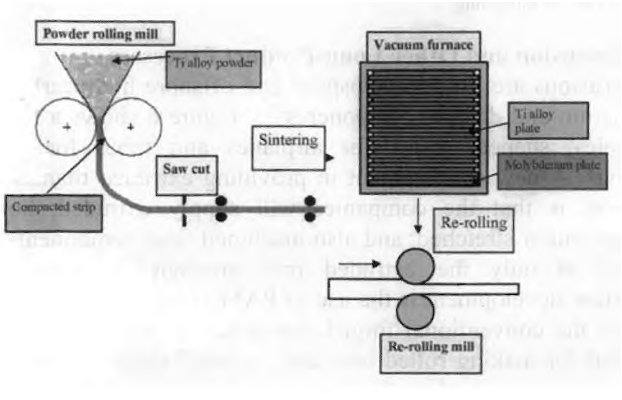

Figure 3. Direct powder rolling process for non-melt processing Ti-6Al-4V sheet and foil[12].

Making mill products by non-melt processing of titanium powders has the advantages of near-net shape processing, decreasing material yield loss, and less total processing steps. All of these advantages can potentially result in significant cost savings. However, several hurdles need to be overcome before the non-melt processed titanium mill products can be considered as commercially viable. These include of course the cost of the powder, but also the quality of the powder and that of the resulting product, notably its microstructure, defects, and the mechanical properties that result. In addition, there is as yet no industrial infrastructure for mass production, and it may be difficult to handle large amounts of fine titanium powder safely and without contamination. At this time, non-melt processing of titanium mill products is transitioning from a pilot plant scale to commercial production[12].

While it has been shown that the microstructure and tensile properties of non-melt processing products are comparable to the conventional wrought products[24,25], the open literature[27,28] contains only limited information on dynamic properties such as fatigue and stress rupture. In addition, there is no information about the types and characteristics of defects that may be found in these products. Also, it is not yet known whether they perform well in practice since only a few of the products made from the non-melt processed material have been sold commercially.

5. Shaped Components

In the USA, the biggest titanium market is aerospace. This industry wants to reduce a component's buy-to-fly ratio, so that processing that allows near net shape production receives increasing attention. The next sections discuss the various possibilities.

5.1 Castings

The sales volume of titanium castings is increasing despite the higher price of the material itself. The increase occurs in the traditional titanium markets, aerospace, and even more in non-traditional areas such as components for armed vehicles and artillery[29]. In addition, rammed graphite castings are now being used for the chemical process industry and military vehicle[30]. A new development in investment castings is the production of two thin wall (0.762-1.016 mm) titanium parts[29] for the Boeing C-17 that has been possible by careful optimization of casting and post-casting operations.

Microwave heating is common in home and restaurant kitchens, and is already applied on an industrial scale to heat treating and sintering. Melting industrial materials with microwaves has been demonstrated since the 1990s on Al, Pt, B, Ni, Cu, and stainless steel. The Department of Energy recently awarded a contract to Technikon[31] aimed at lowering the cost of castings through microwave melting and Digital Mold Printing. Their results to date suggest that microwave melting has a potential to reduce the cost for casting titanium.

In microwave melting, the metal is placed in a crucible, made from a ceramic that absorbs the microwaves and can withstand heat cycling. This crucible is inside another ceramic casket that is heat-insulating, but lets the microwaves through. The crucible and ceramic casket are placed in a microwave oven, which can be evacuated, and refilled to exclude oxygen as needed. During melting, the microwaves first heat up the crucible without much heat loss to the outside casket. Once the metal reaches to about 50 to 75% of the melting temperature, the metal's electrical resistance is high enough that it absorbs the microwaves directly. Once the metal has melted, the crucible may be removed for pouring, or the metal can be poured into a mold within the microwave oven itself.

The advantages of microwave melting are: (a) clean melts without induction field and the resulting turbulence in the melt, (b) with proper choice of materials, no wetting of the ceramic crucible; for titanium this implies minimal loss of material to the hard, brittle and oxygen-enriched layer that can form on the surface of hot titanium, the 'alpha case' shown in Figure 4, (c) 60% to 80% lower energy use compared to the currently used VAR Skull Melting and Induction Skull Melting, (d) increased safety because water cooling is not needed in the furnace, and (e) high molten metal superheat (more than 167°C) which is important for making sound castings.

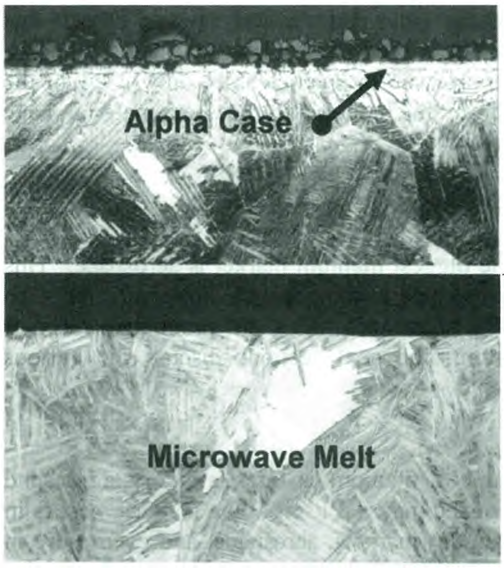

Figure 4. Alpha case on the surface of titanium from conventional melting and microwave melting[31)].

5.2 Superplastic Forming

The traditional use of superplastic forming (SPF) is high value aircraft components, but recently SPF has been applied in making components for buildings, cookware and automobiles[32)]. Several major developments in the last few years have tried to reduce the cost of SPF components.

Boeing tries to lower the cost of SPF components by using sheet with a fine grain structur[32)]. For a conventional Ti-6Al-4V sheet, the grain size is about 4 μm to 10 μm, but recent advances in titanium sheet production have reduced the grain size to around 1 μm. This fine grain material flows at much lower stress than the conventional grade Ti-6Al-4V, so that SPF can occur at 775°C, more than 10% lower than the 900°C needed for the traditional material. The result is increased operator safety and comfort, reduced maintenance of tooling and forming presses, and longer tool and equipment life. The lower forming temperature also minimizes the alpha case, and, thereby, minimizes subsequent chemical processing to remove it. Boeing is also trying to make large pieces for commercial aircraft[33)] using SPF followed by diffusion bonding (DB). Previously, Boeing had used SPF together with DB only to make components for military aircraft.

Hi-Tech Welding and Forming, Inc., developed a new SPF process, 2nd Generation^{TM} SPF^{TM}, to form large SPF parts in a vacuum environment[34)]. The company uses its proprietary engineering modeling software and specially designed tools to make large size, complex shape and low cost SPF components for aerospace, military and marine applications. Advantages of the 2nd Generation^{TM} SPF^{TM} include: a) ultra clean surface due to the vacuum environment, (b) about half the thinning which enables the use of thinner gage sheet to save material, (c) superior dimensional tolerance and repeatability reducing tool cost, (d) virtually no size limitations, and (e) capable of more complex shapes (Figure 5). The company also uses EB welding to weld two sheets together for making a large size component.

Figure 5. Ti-6Al-4V fresh water tank for Boeing Business Jet/737, deep integral ribs for stiffening[34)].

5.3 Extrusion and Other Long-Product Processes

Extrusions are used in aerospace and offshore hydrocarbon production and drilling components[35)]. Figure 6 shows a few examples, shaped beams for airplanes and tubes for oil drilling. A new development in providing extruded titanium products is that the companies will supply extruded, hot formed and/or stretched, and also machined final components, instead of only the extruded mill products[36,37)]. Another important development is the use of PAM single melt ingot to replace the conventional forged fine grain billet as the input material for making rolled bars and extruded shapes/tubes[5,23)]. This approach can reduce the input material cost significantly.

The growing use of composites in transport aircraft has led to the greater use of titanium long shapes and required product shapes like curved "L" and "T" that are not traditionally made from titanium[38)]. RTI is establishing a production facility of Hot Stretch Forming to form curved long parts from extruded shapes[39)]. Stretch forming at elevated temperatures has the ability to stretch a contour into a titanium long part while simultaneously controlling the dimensional stability and residual stresses[38,39)]. The process involves clamping the part in jaws on each end, heating the part, fitting the part around a die, prestretching the part with a specified stress, and pulling the part over the die when stretch is done. Examples of parts made this way are shown in Figure 7. Boeing is also working on Hot Roll Forming which has a potential to limit material waste by using plates or, potentially, round billets, to produce a near-net shape[37)]. The forming process is typically below the beta transition temperature. One limitation for Hot Roll Forming is that it is difficult to form a thick plate into desired shapes.

Figure 6. Examples of extruded shapes[36)].

Figure 7. Curved long parts made from hot stretch forming of extruded shapes[39)].

5.4 Flowforming

Flowforming is a net-shape cold-forming process that can make high precision cylindrical tubular components[40)]. Figure 8 shows some examples. To shape the part, a cylindrical preform is fitted over a rotating mandrel. Compression is applied to the outside diameter of the preform by a set of three hydraulically driven, CNC-controlled rollers. The desired geometry is achieved by compressing the preform above its yield strength so that it deforms plastically. As the preform's wall becomes thinner, the material lengthens and forms as dictated by the rotating mandrel. Typically, the preform is rolled out to four or five times its original length.

Figure 8. Flowforming produces extremely straight and concentric parts[40)].

The characteristics of the flowforming process are: (a) cold work can increase the material's strength and hardness, i.e., strain hardening, (b) dimensional accuracy is better than hot-forming processes and often good enough to eliminate the need for secondary finish machining operations such as turning, grinding, and honing, (c) parts are extremely round, straight, and concentric, often eliminating the need for straightening and balancing, (d) thin walls with varying wall thicknesses can be formed regardless of the diameter size, (e) the surface is very smooth, (f) as-formed parts may meet the required mechanical properties, eliminating the heat treatment and associated distortion, (g) grain size is significantly finer than that of the extruded material, and (h) grains become long and flat, and the "elongated pancake" shapes give a stronger texture.

Materials that can be flow-formed include Al alloys, Zr, Ni-base alloys, and titanium alloys. Applications include airframe, jet engine, oil exploration, bicycle, nuclear waste treatment, and military vehicle components. For flowforming of titanium alloys, it used to require cylindrical tubulars with an $\alpha+\beta$ structure as the input material. New developments have shown that tubulars with a β structure can also be used as the input material.

5.5 Powder Metallurgy

Traditionally, powder metallurgy makes shaped components by pressing powder in a die, followed by sintering. However, powder metallurgy with titanium has been limited by the expense of titanium powder, so that the anticipation of large quantities of low cost titanium powder made by the new powder making processes provides a renewed incentive to develop powder metallurgy for titanium. Dynamet Technology, Inc., has used both die pressing and CIPing followed by sintering to make shaped components[26)] from the pure metal, and also metal matrix composites. For example, Dynamet's CermeTi® metal matrix composite, titanium reinforced with titanium carbide and titanium boride, offers improved wear resistance, a higher modulus, and strength at elevated temperature useful in both commercial and military applications.

Oak Ridge National Laboratory[24] and ADMA Products, Inc.[41,42], are also working on making titanium parts with powder metallurgy. Oak Ridge's work focuses on powder made by ITP (International Titanium Powder, LLC), whereas ADMA works with several other types of powders, including titanium hydride. In general, this technology is still in the development stage and the commercial market is rather small.

5.6 Free Form Fabrication

Free Form Fabrication (FFF) is a metal additive manufacturing process; instead of removing material by machining or grinding, the process involves adding metal until the final shape is achieved[43-47]. The result is a fully dense structure that corresponds directly to a 3-dimensional CAD solid model. The method entails melting the powder or wire material with a power source that can be a laser, an electron beam, or a plasma, in the appropriate environment (argon for a laser or a plasma, vacuum for an electron beam). The part is constructed layer by layer under the control of software that monitors a variety of parameters to ensure geometric and mechanical integrity. Figure 9 is a schematic of the FFF process using the plasma transferred arc as the power source. Currently, there are nine different processes that are under development[43].

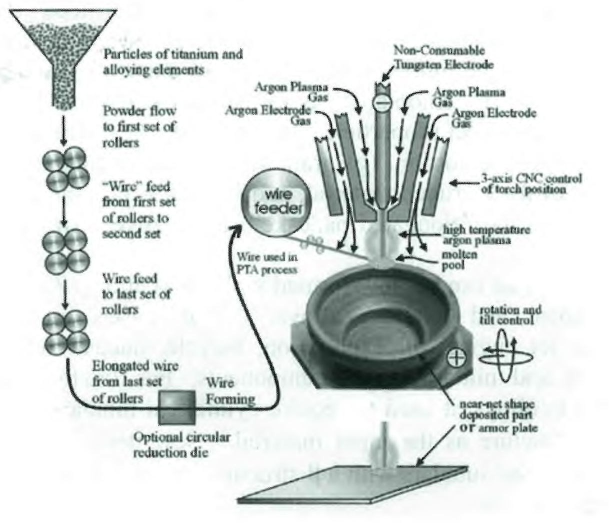

Figure 9. Schematic of plasma transferred arc (PTA) solid free form fabrication system (SFFF)[47].

FFF is particularly suitable in four cases: (a) rapid prototyping, (b) fabricating structures and equipment, (c) repairing and refurbishin[45], and (d) making hybrid structures. Solidification porosities that may form during deposition of the melted material on the work piece are typically eliminated by HIPing, after which the mechanical properties of FFF components are comparable to or even better than those of wrought products[45]. Boeing has conducted a significant amount of work on this technology, and specification AMS 4999 has been modified to apply for FFF component fabrication[43]. However, the technology is still in the development stage and waiting for transition to commercial production. The primary impediments to FFF technology are the costs: the expense of the powder or wire, the low productivity (deposition rate ranges from 0.2 to 20 kg/hr), and the additional cost associated with HIPing. One of the technical concerns is the control of distortion.

5.7 Powder Injection Molding

Similar to plastic injection molding, titanium powder injection molding (PIM) injects a mixture of fine titanium powder and hot liquid plastic into a metal mold or die[48]. This gives a 'green' part. Subsequently, the plastic binder is removed and the metal part is sintered in vacuum to high density. PIM is a cost effective way to produce small (normally below 400 g) parts with complex shapes. The process is currently used in Japan for commercial production, but not yet in the USA. For cosmetic items, where the mechanical properties of the material are not important, the preferred metal in PIM is CP titanium, while structural parts are also made from titanium alloys.

Two major issues must be addressed in titanium PIM parts. First, the process demands fine titanium powder with a low oxygen content. Fine powder not only costs more, but is also more chemically reactive because of its higher surface area, so that its oxygen content and tendency for ignition hazard are also higher. The new powder making processes would hopefully solve this problem. Second, the binders should not react chemically with titanium. However, conventional polymer binders depolymerize around about 260°C, where titanium already reacts with the decay products and takes up contaminants. Pacific Northwest National Laboratory (PNNL) reported a titanium PIM process that can potentially speed up production time and reduce the cost of making titanium PIM parts[49,50]. It uses a proprietary naphthalene binder that melts at 81°C, and minimizes contamination with oxygen by reducing oxygen impurities with titanium hydride powder that then becomes titanium metal at a temperature of about 350°C or higher.

6. Welding

Titanium is commonly welded by a gas-protected tungsten arc (GTAW)[51]. However, sheets thinner than 1 mm cannot be welded by GTAW because of excessive distortion, and welding sections thicker than 7 mm with GTAW is less economical due to low productivity. The same problems plague gas metal arc welding (GMAW) and pulsed gas metal arc welding (GMAW-p). A potential solution is laser plus GMAW hybrid welding. For welding thin sheets, a low power laser can stabilize the GMAW-p arc to result in a high welding speed and lower distortion. GMAW hybrid welding of thick section titanium needs a high power laser that increases the weld's penetration depth, as shown in Figure 10.

Friction stir welding (FSW) is a solid state welding process that uses a non-consumable rotating tool to heat and plasticize the materials in contact to form a weld. FSW has been used to weld all wrought aluminum alloys. The original application for FSW was the welding of long lengths of material in the aerospace, shipbuilding and railway industries. The application of FSW to titanium for aerospace, Army, and Navy has been investigated extensively in the last several years[52,53]. The advantages of FSW include: (a) no porosity that may arise in fusion welding, (b) low distortion and residual stresses, (c) good mechanical properties at weld line, (d) forming hybrid components by joining dissimilar materials, and (e) lower cost. Boeing is using FSW to weld several small sheets into a large sheet which is then used to make a large SPF component. Linear friction welding (LFW) is another friction welding process that has been developed for aerospace applications54). LFW uses a linear motion to generate friction heat for welding and is an established production process for the manufacturing of blisks of jet engines. Boeing is investigating a range of concepts for the production of machining preforms by LFW.

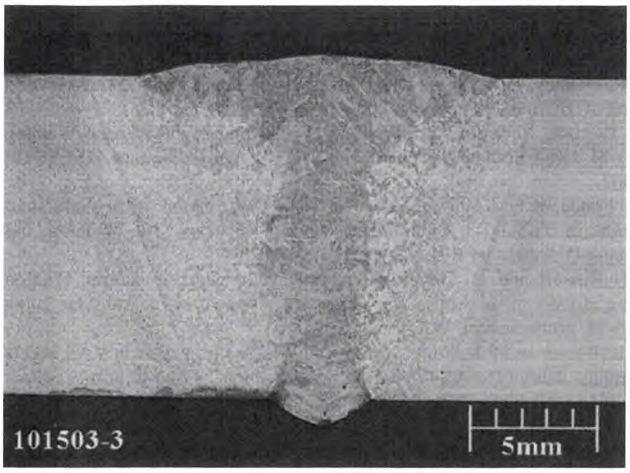

Figure 10. Laser-GMAW hybrid on titanium thick plate51).

7. Process Modeling

Process modeling has long been recognized as an effective tool to understand the process dynamics, and to optimize the process parameters needed for defect-free products. Earlier models could only address macroscopic issues such as heat transfer and electromagnetic field penetration for VAR ingots, heat transfer, fluid flow and mold filling for investment castings, and heat transfer, stress/strain distribution and die filling for closed die forgings. Nowadays some models even attempt to simulate ingot chemistry distribution, and to predict component microstructures and mechanical properties. For example, one new development for VAR modeling is the prediction of the distribution of oxygen 55). For investment casting, Howmet tries to couple the predicted grain size with mechanical properties29). However, most process modeling still addresses principally thermomechanical processing56-63) albeit with more modern approaches. These include: (a) finite element models (FEM) to predict temperature and stress/strain, (b) modeling of the thermodynamics, to predict the beta transus, phase proportion, phase chemistry, partition coefficient, and phase boundary, (c) following the evolution of the microstructure to predict beta grain, grain boundary alpha, primary alpha and lamellar alpha growth,
(d) modeling the evolution of texture during rolling and forging, and (e) trying to predict the mechanical property relating to processing, chemistry and microstructure by neural network modeling. Many of these modeling tools are being actively used in production for closed die forging, open die forging, rolling and extrusion processes.

8. Summary

Significant progress in titanium processing technology has been made in the last four years. As expected, technologies that are directly related to the current production processes or equipment have made the most progress. These technologies include single melt, SPF with fine grain sheet and vacuum environment, thin wall investment casting, extrusions, and process modeling. Still, there are several technologies which are either close to becoming commercial production or are already in small quantity production. These technologies are ITP Armstrong and DuPont processes for making powder, non-melt processing for mill products and shaped components, free form fabrication, flowforming, hot stretch forming and friction stir welding. Finally, several other technologies such as some of the new extraction processes for making sponge and powder, as-cast PAM ingot for directly rolled bars and extruded shapes, and powder injection molding are still in the development stage.

The titanium market in the USA is expected to continue to grow in the next several years. The demand of robust processing technology to reduce the cost of titanium remains high. Future titanium development efforts will continue focusing in this direction.

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