Critical Review

TITANIUM POWDER METALLURGY AND COMPOSITES

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Introduction

Powder metallurgy is increasingly being chosen as a processing route for high performance applications in advanced aerospace and industrial systems. Both the high cost of titanium and the high cost of machining these alloys have motivated development of P/M titanium technology. Unlike the comparable development in the field of P/M nickel base superalloys large scale commercial acceptance of titanium P/M has not yet been accomplished. Several reasons could be given - too small proven market, too little involvement from the end user's side. The consequence has been widespread uncertainty about the actual potential for the technology. It cannot be overlooked, however, that development efforts in this field have substantially increased in the past few years.

Briefly, here are some of the advantages that have been claimed for the use of powder metallurgy in conjunction with titanium:
- capability of making more complex shapes
- cost reduction
- isotropic mechanical properties
- controlled fatigue properties due to control of flaw size
- improved mechanical properties in large sections

The field of titanium P/M has been reviewed recently by Froes et al. [1]. It was therefore considered appropriate to condense the information available further and to contrast the existing devel-
opments to each other and - whenever possible - give a critical outlook.

Titanium P/M can be divided into conventional fine particle metallurgy (elemental and prealloyed approach) and coarse particle metallurgy (prealloyed powder approach).

Fine particle metallurgy

It has been recognized at an early stage that one of the lowest cost powders is the sponge powder which is also needed in the arc-melting of titanium alloy ingots. Titanium sponge fines can be made with almost the same impurity level as cast & wrought products, exceptions being interstitials, Na and Cl. The Cl-contamination cannot be avoided as long as the Kroll process sponge fines are used as titanium source. The Cl-content can be reduced to 0.05% by admixing low Cl-content electrolytic titanium powder (0.02% Cl) \cite{2}, although lower Cl-contents have been listed \cite{3,4}. Titanium alloys are made by blending elemental titanium powder with a master alloy of Al/V. The compaction techniques that are used are either uniaxial cold compaction or cold isostatic compaction. Sintering is carried out in vacuum at temperatures well above the β-transus (1100-1300°C). The sintered compacts reach densities up to 99% of T.D. Further consolidation and hot-working has been attempted by HIP or hot forging. Table 1 compares the effect of 3 different processing routes on mechanical properties of Ti6Al4V-powder from various sources. While it is difficult to fully assess the influence of varying impurity content various consequences of processing can be seen: (a) significant advances have been made in the press- and sintering technique \cite{2}. (b) A further reduction of residual porosity to a level of 0.1% by closed die forging at high pressures contains a significant potential for strength and ductility improvement \cite{4}, which has not yet been reached by HIP pressing at relatively modest pressures. It can be speculated that isothermal forging or hot-die forging at considerably higher pressures than HIP could further help to close the residual porosity. One would also expect that the fatigue strength would further increase to a
Table 1  Mechanical properties of Ti6Al4V via the elemental approach

<table>
<thead>
<tr>
<th>Ti-Sponge Powder (mesh size); Impurity level in consolidated product</th>
<th>Processing steps</th>
<th>Density (% T.D.)</th>
<th>UTS (MPa)</th>
<th>Yield Strength (MPa)</th>
<th>Elong. %</th>
<th>RA %</th>
</tr>
</thead>
<tbody>
<tr>
<td>powder size unknown; ~old</td>
<td>Cold Pressing (% 420 MPa); Sintering in vacuum (1232°C/3hrs)</td>
<td>94</td>
<td>754.3</td>
<td>643.3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>0.2 O₂</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.01 N₂</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.06 Na</td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>&lt;10 ppm Cl</td>
<td>a) forged*</td>
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<tr>
<td>Source A (ref. 3)</td>
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<td></td>
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<td></td>
<td></td>
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<tr>
<td>0.24 O₂</td>
<td></td>
<td></td>
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<td></td>
<td></td>
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<tr>
<td>0.016 N₂</td>
<td></td>
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<td></td>
<td></td>
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<tr>
<td>0.002 H₂</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>0.1 Na</td>
<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>0.12 Cl</td>
<td></td>
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<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Source B (ref. 2)</td>
<td>CP (620 MPa)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>- 100 mesh powder size;</td>
<td>S (unspecified temperature)</td>
<td>99</td>
<td>960.5</td>
<td>884.6</td>
<td>11.5</td>
<td>22.8</td>
</tr>
<tr>
<td>0.24 O₂</td>
<td></td>
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<td>0.016 N₂</td>
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<tr>
<td>0.12 Cl</td>
<td></td>
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<td></td>
</tr>
<tr>
<td>Source B (ref. 2)</td>
<td>CP (500 MPa)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>- 60 mesh powder size; impurities in powder:</td>
<td>S (%1300°C/1hr); forging**</td>
<td>99.9</td>
<td>1135</td>
<td>-</td>
<td>8</td>
<td>-</td>
</tr>
<tr>
<td>0.095 O₂</td>
<td></td>
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<tr>
<td>0.0035 N₂</td>
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<tr>
<td>0.014 H₂</td>
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<tr>
<td>Na ? Cl ?</td>
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<td>(ref. 4)</td>
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<tr>
<td>AMS 4928 - specification</td>
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<td></td>
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<tr>
<td></td>
<td></td>
<td>100</td>
<td>896.4</td>
<td>827.4</td>
<td>10</td>
<td>25</td>
</tr>
</tbody>
</table>

* Cond. 1 927 C forge, reduction 5:1
Cond. 2 927 C forge, reduction 2.5:1 + 968 solut. treat. + 732°C + 2 hr
Cond. 3 1024 C forge, reduction 5:1

** Cond. 4 900 C forge, reduction 1.1:1
Cond. 5 1000 C forge, reduction 1.1:1 + 0.5 hr 925°C/FC + 4 hr 500°C
Cond. 6 900 C forge, reduction 1.1:1 + 0.5 hr 925°C/WQ + 4 hr 500°C
level of forgings, well above the fatigue strength of castings. Fatigue crack growth rates are lowest for the as-sintered microstructures (transformed $\beta$). Hot-working in the $\alpha+\beta$-region would tend to slightly increase these rates. Fracture toughness values, $K_{IC}$, are still low and need to be improved further.

One of the main advantages of the elemental approach is the low cost of the starting powder and the considerable savings in the energy cost usually needed to convert sponge to billet by the arc-melting process. There is also considerably less capital-outlay needed for cold compacting equipment. Complex shapes have been demonstrated such as impellers |3a|. The most advanced application is a connecting link for the P&W FA-100 engine |2| (Figure 1). It is expected that the elemental powder approach will be considered also for more critically stressed parts.

Coarse Particle Metallurgy

Following the success of the P/M nickel-base superalloys the coarse-prealloyed-particle approach was developed also for titanium alloys. Because of the high yield strength of the powders and because of the large particle size cold compaction and vacuum sintering techniques are not feasible.

1. Powder production techniques:
Table 2 summarizes the existing techniques. The two methods utilizing a mechanical method to disintegrate the melt (CSC, EBRD) would offer commercial advantages, because they do not require fabrication of a precision machined cylindrical electrode; they do have problems relating to powder chemistry. The optimum (but not the cheapest) process might well be PREP, which has been successfully tested in the USSR |5| and at Creusot-Loire (France) and which is currently being set up by Nuclear Metals (USA).
Concerning the powder quality there is a tendency in the USA to accept defects but define minimum levels which would still give satisfactory mechanical properties. In Europe, particularly in Germany, research |6| is still focussing on completely eliminating any contamination. At present time the PSV-process is favored
compared to other methods, although α-case formation in some particles is cause for concern.

Table 2 Powder production methods and associated defects

<table>
<thead>
<tr>
<th>Method</th>
<th>Heat source/atmosphere</th>
<th>Problems</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rotating Electrode Process (REP)</td>
<td>arc / argon</td>
<td>W inclusions</td>
</tr>
<tr>
<td>Dual Electrode Process (DEP)</td>
<td>arc / argon</td>
<td>black dust (Ti)</td>
</tr>
<tr>
<td>Centrifugal Shot Casting (CSC)</td>
<td>arc / argon</td>
<td>black dust (Ti)</td>
</tr>
<tr>
<td>Plasma Rotating Electrode Process (PREP)</td>
<td>plasma / argon</td>
<td></td>
</tr>
<tr>
<td>Powder Under Vacuum (PSV)</td>
<td>electron beam/vacuum</td>
<td>α-case (Al-evaporation)</td>
</tr>
<tr>
<td>Electron Beam Rotating Disc (EBRD)</td>
<td>electron beam/vacuum</td>
<td>β-flecks (Al-evaporation)</td>
</tr>
</tbody>
</table>

2. Microstructural Control

In general, there is no possibility to introduce cold-working and recrystallization during hot-pressing of prealloyed titanium alloy powders. This presents a limitation as far as microstructural control is concerned and has an effect on mechanical properties. In P/M alloys the as-formed microstructure is martensitic or α-prime and results from the quick chilling during powder production. Annealing of Ti6Al4V powders in the high (α+β)-region produces a plate-like α-structure, annealing at lower temperatures gives an acicular α- or Widmanstätten structure. The limitations due to hot pressing can be overcome by prior cold-rolling of the powders to store sufficient strain energy in the material to recrystallize the microstructure (to equiaxed α+β) during hot pressing.

3. Powder Consolidation Techniques

a. Hot Pressing in vacuum or inert gas atmosphere, using TZM and superalloy tooling similarly as in isothermal forging. Higher pressures than in HIP are possible. The technique could be cheaper
than HIP provided the problems of lubrication and introduction of powder into a heated die can be solved. The process has been applied to a low cost hydride powder [7]. The dissolved hydrogen lowers the hot pressing temperature by ~100°C and the compacting pressure by 35 MPa. Post-processing vacuum annealing is required to reduce the hydrogen concentration to <100 ppm. The technique was also applied to REP Ti6Al4V-powder under argon-atmosphere to establish the densification kinetics up to 95 % T.D. [8]. A variant is the closed-die forging of canned powder. Applying pressures of 500 MPa to an alloy Ti6Al1.5Zr3.2Mo0.2Si densities of 99.6-100 % T.D. were obtained in a 400 mm Ø disc [9]. A tensile strength of 1020 MPa, an elongation of 16 % and a reduction in area of 35 % are an indication of the potential of this technique.

b. **Hot Isostatic Pressing (HIP)**

This is by far the most widely used compaction technique. Powder is filled either into thin metal cans produced by conventional sheet forming technology or by galvanically coating a 1 mm thick layer onto a conducting wax shape; in the Crucible ceramic mold process and a related process developed at Krupp in Germany ceramic molds are prepared by the lost-wax process. A secondary pressing medium contained in a steel can provide sufficient support to prevent distortion of the preform during densification. In the Fluid-Die-process a thick-walled metal container filled with powder is uniaxially pressed in a closed-die under high pressure. The can exerts a nearly isostatic pressure onto the powder. The last 2 processes have net-shape capability (Figure 2 shows two examples of HIP shapes).

c. **HIP + Forge**

This is basically a method to further increase the density and to control the microstructure [10]. It is particularly suited for cost-saving preform designs for complex forgings.

4. **Mechanical Properties**

Mechanical properties of compacted P/M titanium alloys depend on the amount of residual porosity, the type and amount of defects and inclusions and finally on the microstructure which is a function of the deformation history and of the heat treatment. In fatigue also the surface condition has to be included in the characterization process. Until now these parameters have not been
sufficiently discussed.

a. **Effect of residual porosity:** assuming that uncontaminated powder were available a similar trend as in the elemental powder approach is to be expected, where significant strength increments were obtained by reducing residual porosity to the 0.1 % level.

b. **Effect of defects and inclusions:** the best study was conducted by Vaughan et al. [11] on Ti6Al4V powder obtained from 5 different sources. The powders were consolidated by HIP and subsequent hot rolling. All specimens were given the same heat treatment (960°C AC/2hrs 700°C AC). Microstructural analysis and fractography on fatigue specimens showed the type of defects listed in Table 2. In all cases the fatigue strength was below that of conventional bar stock material. In Ti6Al4V 85-99 % of the fatigue life is spent initiating the crack [12] which emphasizes the role of inclusions.

c. **Effect of microstructure:** this is the least investigated effect, primarily because it depends on first eliminating porosity and internal defects. It would be worthwhile to carry out such an exercise on powder produced under optimum conditions (presumably PREP).

The fatigue strength of titanium alloys with as-HIP surfaces can be improved by shot-peening techniques [13] much the same way as this is done on conventional and isothermal forgings [14]. This is an important requirement for many applications made by a net-shape technique to improve the cost-effectiveness. Most of the work done on HIP titanium alloys [13,15] has shown that static mechanical properties, notched fatigue strength (emphasizing the fatigue crack growth life) and fracture toughness compare favorably with the properties determined on wrought material. As mentioned before the most critical and often inferior property is smooth fatigue strength, especially in the higher strength alloys.

**Economical Considerations**

In massive forming of titanium components a series of new net-shape processing techniques have been developed - precision casting, hot die and isothermal forging, and powder metallurgy.
These techniques compete both against each other and against the established shaping techniques like conventional forging and machining. To comment on the economics of titanium powder metallurgy in a realistic way means to look at each component and compare P/M processing with the most economic competing technology. In the past this comparison has only been done against the more expensive conventional methods. As far as shape complexity is concerned P/M and precision casting are comparable, whereas obvious shape restrictions exist for forging techniques. The situation is different for shapes that can be forged: The high cost of titanium powder converted from billet into powder, the significant cost of canning and outgassing plus the HIP costs make it difficult to see where P/M would compete against the newly developed forging techniques. Combinations of powder metallurgy and forging techniques look attractive if P/M is utilized to make complex preforms that need fewer forging operations. It is much easier to see the cost advantages for the press- and sinter-approach, where low cost powders and tooling concepts are employed.

Another area where powder metallurgy might be the cheapest method is in the production of semifinished products where the cost reduction comes from making a less expensive preform. One suggested application would be the production of titanium tubing by shape extrusion of a hollow HIP or sintered preform, analogous to a newly developed process for making stainless steel tubes [16].

Titanium Matrix Composites

The titanium matrix composites field has shown relatively little activity since the subject had been reviewed in 1977 [17,18]. Titanium matrix composite systems may be made from (a) short fibers, (b) continuous fibers, (c) particulates. At present time there are mainly two types of compositions under investigation: silicon carbide coated boron filaments (Borsic/Ti) and Be/Ti, where beryllium metal is used either in powder [19] or continuous filament form. A comparative investigation has shown that BSiC/Ti is superior to B/Al from the foreign object damage point of view [20]. The high cost of such composites has led to investigate the use of Ti-powder preforms to replace the more costly wrought
Ti-foils. Work in the USA is also going on with isothermal forging of Be/Ti composite parts, although the toxicity of beryllium will probably limit the wide-scale use of such composites.

**Summary**

The field of titanium powder metallurgy has experienced an increased interest in the past few years. Significant advances have been achieved in pressing and sintering of blended elemental powders to 99% densities, opening the road to lower cost/high strength components. In the prealloyed powder field a stage of good qualitative technical understanding of powder defects and mechanical properties has been reached. Further improvement in reducing powder defect levels and better quantitative correlations between microstructure and mechanical properties will be required before large-scale use of titanium P/M parts becomes a reality. The cost saving- and market potential have to be further clarified, particularly in light of recent advances in competing techniques like vacuum casting, hot die- and isothermal forging. Relatively few activity has been observed in the field of titanium matrix composites.

**Acknowledgements**

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**References**

3. (a) S. Abkowitz, ibid.;
(b) R.R. Boyer, J.E. Magnuson and J.W. Trip, ibid.
5. V.S. Rakovsky, ibid.
Fig. 1  Connecting link made by pressing and sintering \[2\].

Fig. 2  Complex shapes of Ti6Al4V made by HIP
(a) near-net shape using a metal can (Seilstorfer GmbH)
(b) impeller made by Crucible's ceramic mold process.