A CONTINUOUS PROCESS FOR PRODUCTION OF CP TITANIUM SHEET BY DIRECT POWDER ROLLING

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ABSTRACT

A number of significant research efforts globally are aiming to develop competing processes for the commercial production of low cost titanium powders. If successful this could significantly reduce the current high cost of near-net-shape titanium components produced by conventional powder metallurgical (PM) processes. It would also enable manufacture of semi-finished products including sheet and tube by novel powder consolidation processes. Such a process is described for the continuous production of commercial purity (CP) sheet by direct rolling of powder. It is novel in that it avoids subsequent densification via batch sintering. A binderless green rolled sheet is rapidly heated under a controlled atmosphere before being consolidated to nominally 100% density by hot rolling. Following conventional batch mill annealing, the strip exhibits properties approaching that of conventional wrought sheet of the same grade. In order to achieve this, a number of key variables including powder chemistry, morphology and particle packing, green and hot rolling parameters needed to be understood and closely controlled.

Keywords: Titanium, Sheet, Powder Metallurgy, Powder Rolling, Mechanical Properties.
1.0 INTRODUCTION

Titanium is not easy to extract from its ore, it is difficult to process and expensive to fabricate \[1\]. As a result, the high cost of production, compared to alternative materials has restricted it to critical applications in the aerospace, prosthetics and chemical industries. Consequently, there is a general emphasis on lowering the cost of both primary production and fabrication of titanium through the application of innovative processes \[2\]. A number of efforts\[^{3,4,5}\] are seeking to produce titanium powders either of chemical purity (CP) grade or selected alloys.

As highlighted in Figure 1\[^{6}\], innovative technologies in both the primary and secondary aspects of production will be needed to achieve the greatest cost reductions between powder feedstock and finished product. However, the most critical need is for the reduction of the price and improved availability of the feedstock powder \[^{7,8}\].

![Figure 1](image.png)

**Figure 1.** The current trend in R&D is to replace conventional processes in the production of titanium with new innovative processes in order to reduce production costs (after \[^{6}\]).

New powder metallurgy (PM) techniques that have the potential to further reduce production costs with minimal or no loss of mechanical properties of the resulting product \[^{9,10,11,12}\] are also emerging. These may lead to a significant expansion of Ti PM component production into new applications in the automotive, architectural and sporting goods industries.

Current methods for production of titanium sheet involve a multitude of stages (Figure 2) and therefore the prospect for cost reduction is significant.
There are a number of processing routes that have been developed for the production of strip from powder by direct rolling various metals \([29,14,15,16,17,18]\); and specifically titanium \([26,19,20,21,22,23,24,25]\). These generally involve continuous consolidation using a standard rolling mill to produce a green sheet. This sheet is then sintered and re-rolled (hot and/or cold) to produce a flat product with a tailored degree of porosity \([26]\) or as fully dense strip \([27]\). The general concept of the continuous production of strip from a metal powder originated early in the twentieth century but the process was not fully described until 1950 \([28]\). During the 1950’s and 1960’s there was a great deal of research and development in this field \([29]\). The main advantages of direct powder rolling technology over the conventional ingot/wrought route include:

- Capital equipment savings from a reduction in processing steps.
- Production of higher purity strip, free from segregation and with higher yield.
- Production of fine-grained, high strength strip with no preferred texture.
- Production of sheet from specialty and difficult-to-work materials.
By the 1960s DuPont had developed a process for the direct rolling of Ti sponge fines into strip in commercial quantities. The mechanical properties were reported to be equivalent to the conventional wrought material, however the fines contained small quantities of sodium chlorides (0.01-0.05%), trapped in the microstructure. These originated from the extraction process and reportedly made the material essentially un-weldable. Recently, there have been significant advances in Ti powder production and high purity powders are expected to be available in commercial tonnages. Much interest has also centred on Ti powder generated via the hydride-dehydride (H/DH) process, since it has been established that the addition of hydrogen to Ti powder has added benefits during consolidation.

2.0 THE CSIRO PROCESS

The CSIRO process for the production of strip is shown schematically in Figure 3. It uses a novel rapid in-line thermal heating and hot rolling instead of the sintering used by others as discussed earlier. It also uses binderless powder feedstock.

In the research, the emphasis has been on the use of conventional hydride/de-hydride (H/DH) derived powder (Figure 4(a)) as the feedstock. “Low cost” powder from DuPont (Figure 4(b)) has also been explored.

Figure 3. Schematic of CSIRO process for the continuous production of CP-Ti strip.
Figure 4. Morphologies of a) H/DH and b) DuPont CP-Ti powder.

Powder morphology, size distribution and chemistry have a strong influence on the ease of direct powder rolling and it is therefore critical that these be controlled. An example of as-rolled compacted (green) sheet is shown in Figure 5(a) together with SEM micrographs of the surfaces of the compacted green sheet derived from H/DH and DuPont powders in Figure 5(b) and Figure 5(c) respectively. The compaction behaviour of H/DH (angular particles) and DuPont (sponge like) powders are clearly different and are a function of particle packing characteristics and the level of interstitial impurities. The oxygen content of the H/DH material at ~0.28-0.35% is approximately three times higher than the oxygen content of the DuPont powder. However satisfactory sheet can be made from both powders. This is a good indication that the process can accommodate a range of powder characteristics if adequate controls are in place.

Green strip generally contains from 15% to 25% porosity and has minimal strength and little ductility. Conventionally, in order to improve handling during subsequent processing, the strip is batch sintered. Most, if not all, of the processing routes cited in the literature are based on pressureless solid-state sintering under controlled atmospheres to avoid oxidation [37,38,39,40,41] and it is generally accepted that economic constraints dictate the use of high temperatures (typically in excess of 0.75T_m) to achieve relatively short sintering times.

Sintering times are generally in the range of 2-4 hours, however, in the case of the CSIRO process, time at maximum temperature is in the order of a few minutes. The density increase is relatively low (<10%) but the heating imparts sufficient mechanical strength to enable hot rolling. Control of the atmosphere is also critical to avoid excessive pick up of interstitial elements by the hot, porous sheet. Using a counter flow of inert gas, such pick-up is generally acceptable at <200 ppm oxygen.
For final strip densities of greater than 99.5% to be achieved with CP-Ti, the preheat temperature must be greater than the $\beta$ transus and the percentage thickness reduction must be $>40\%$. During the hot rolling it is important to monitor and control a number of parameters including rolling load and speed, oxygen content, inert gas flow rates and temperature.

Typical optical micrographs of the as-rolled and post annealed microstructures of sheet derived from grade 3 CP-Ti H/DH powder are shown in Figure 6. The DuPont powder, processed under similar conditions, displayed similar equiaxed microstructures after annealing. Tensile specimens were machined from the as-hot-rolled strip with dimensions according to the ASTM E8M standard. After machining, the specimens were annealed under vacuum in a quartz tube at 750°C for 30 minutes. The ultimate tensile strength (UTS) and percentage elongation to failure were determined on an Instron machine with a cross-head speed of 1 mm.min$^{-1}$. 

Figure 5. a) Direct powder rolling of CP-Ti green strip, surface of green strip derived from H/DH powder (b) and DuPont powder (c).
Figure 6. Reflected light microstructure of CP-Ti sheet derived from H/DH (a) as-rolled and (b) annealed under vacuum at 750°C.

The data obtained from the tensile test along with partial interstitial analyses are given in Table 1.

Table 1. Comparison of PM derived sheet properties with wrought material.

<table>
<thead>
<tr>
<th>Material</th>
<th>Ultimate Strength (MPa)</th>
<th>Elongation (%)</th>
<th>Oxygen (%)*</th>
<th>Nitrogen (%)*</th>
</tr>
</thead>
<tbody>
<tr>
<td>H/DH</td>
<td>739-806</td>
<td>15.6-16.1</td>
<td>0.33-0.36</td>
<td>0.02-0.03</td>
</tr>
<tr>
<td>DuPont</td>
<td>484-534</td>
<td>14-19.1</td>
<td>0.15-0.16</td>
<td>&lt;0.01</td>
</tr>
<tr>
<td>Ti Grade 2</td>
<td>343</td>
<td>20</td>
<td>0.25</td>
<td>0.03</td>
</tr>
<tr>
<td>Ti Grade 3</td>
<td>440</td>
<td>18</td>
<td>0.35</td>
<td>0.05</td>
</tr>
<tr>
<td>Ti Grade 4</td>
<td>550</td>
<td>20</td>
<td>0.40</td>
<td>0.05</td>
</tr>
</tbody>
</table>

* After annealing.

The oxygen and nitrogen contents of the annealed sheet made using DuPont powder were much less than that of the H/DH derived sheet and this explains why the UTS values are lower. (Note the hydrogen content of the annealed sheet was also measured and in all cases was less than 0.002%).

A comparison of the properties of sheet derived from H/DH and DuPont powders with data for wrought CP-Ti ASTM Grades 2-4 (also given in Table 1) indicates that the properties of the PM sheet are approaching those of the wrought material. This further emphasises the ability of the process to produce Ti strip with acceptable properties.

The UTS values are higher and the percentage elongation lower than expected based on the interstitial impurity levels. Analysis of the fracture surface of the sheet derived
from DuPont powder has shown the presence of Na-Cl inclusions (Figure 7), which explains the larger variation in the observed elongation. In the case of the H/DH derived sheet, the reasons for this are currently being investigated. While the properties of the current samples differ somewhat from wrought product, they suggest that the sheet could be used in some applications. Further understanding of the reasons for the differences is expected to enable properties equivalent to wrought material.

![Figure 7. Scanning electron micrograph of the fracture surface from hot rolled strip (Na-Cl inclusions arrowed).](image)

3.0. CONCLUSIONS

A novel process, for the production of titanium sheet from powder by direct rolling has been demonstrated to produce sheet with properties approaching those of wrought product. The process combines roll compaction with rapid heating prior to hot roll densification and has the potential to reduce the manufactured cost of titanium sheet.

The process capability has been achieved by implementing an in-line rapid heating step. In addition, a thorough understanding and control of consolidation in rolling has been needed. This is influenced by morphology, particle size distribution, flow properties and packing density and careful control of the heating, atmosphere and rolling conditions. Subsequent annealing using standard mill procedures has been shown to produce a microstructure similar to wrought product. Further work is underway to achieve properties equivalent to wrought material.

This technology is proprietary to CSIRO and patent protection is pending.
REFERENCES