

## Communication

### On a Testing Methodology for the Mechanical Property Assessment of a New Low-Cost Titanium Alloy Derived from Synthetic Rutile

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Mechanical property data of a low-cost titanium alloy derived directly from synthetic rutile is reported. A small-scale testing approach comprising consolidation *via* field-assisted sintering technology, followed by axisymmetric compression testing, has been designed to yield mechanical property data from small quantities of titanium alloy powder. To validate this approach and provide a benchmark, Ti-6Al-4V powder has been processed using the same methodology and compared with material property data generated from thermo-physical simulation software. Compressive yield strength and strain to failure of the synthetic rutile-derived titanium alloy were revealed to be similar to that of Ti-6Al-4V.

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Widespread use of titanium alloys is mainly inhibited by the high cost of the production of titanium alloy components. This costly upstream extraction and multistage processing route have resulted in the restriction of high strength titanium alloys mainly to the aerospace sector.<sup>[1]</sup> Titanium's unique blend of properties such as high strength-to-weight ratio, corrosion resistance, and biocompatibility make it an attractive material for many commercial applications. However, without a step change in the economics of titanium production, the super-metal will be confined to the aerospace industry and niche applications in markets such as the defence and automotive industries.

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One long-term solution is the production of titanium metal components entirely in the solid state *via* the combination of electrochemical extraction (Metalysis FFC process)<sup>[2]</sup> to directly produce a titanium alloy powder and subsequent consolidation *via* near net shaping technologies. Solid-state consolidation techniques such as the use of field assisted sintering technology (FAST) in conjunction with hot forging are capable of producing shaped metal components with full densities and wrought properties from a powder feedstock.<sup>[3]</sup>

Titanium is currently extracted *via* the Kroll process, a discontinuous metallothermic reduction process, which involves the reduction of  $\text{TiCl}_4$  by Mg to produce a titanium metal sponge. Master alloys are added to the Kroll sponge, before compaction and welding into an electrode for melting. Vacuum arc melting requires multiple re-melts to produce homogeneous ingots, particularly in the case of alloying additions such as Fe or Mn, which are prone to segregation.<sup>[4]</sup> After melting, ingots are subject to multistep hot forging and heat treatments to refine the grain structure and homogenize the chemistry in the billet. Finally, significant wastage is endured during expensive machining of titanium alloys, with some critical aerospace titanium alloy parts having a reported buy-to-fly ratio of 40-to-1.<sup>[5]</sup>

Although powder can be produced from Kroll sponge *via* additional procedures such as hydride dehydride processing, plasma rotating electrode process (PREP) or gas atomization (GA), these are expensive powder production routes that reduce the cost effectiveness of using near net shape powder metallurgy (PM).

Hence, producing titanium alloy powder directly *via* the solid-state FFC extraction process, followed by downstream solid-state consolidation using FAST and hot forging ("FAST-forging")<sup>[3]</sup> to near net shape, will significantly reduce the cost of titanium alloy components. Cost reductions are achieved by directly producing an alloy powder, reducing the number of multistep forging and heat treatment steps, and minimizing both wastage and machining. Further, as the entire production route is conducted in the solid state, melting procedures can be eliminated entirely. It is generally the melting stage in which most defects in titanium alloys originate and so its removal has additional benefit.<sup>[6]</sup>

Further cost advantages can be made by utilizing synthetic rutile (SR) as a feedstock to the FFC process. SR is derived from the iron-rich titanium ore, ilmenite ( $\text{FeTiO}_3$ ), and as such contains a range of alloying elements, principally iron. Following reduction of SR an  $\alpha + \beta$  type titanium alloy is produced, without the cost of alloying additions.<sup>[7]</sup> Hence, the use of SR as a feedstock is notably cost-effective, as not only are feedstock costs reduced, but the dependency on master alloy additions further downstream is reduced or eliminated.

As the utilization of a synthetic rutile feedstock within a production route entirely in the solid state is a particularly lucrative operation, this paper assesses the mechanical properties of a synthetic rutile-derived titanium alloy (3.9 wt pct  $\beta$  stabilizers and <1 wt pct Al) consolidated *via* FAST (Figure 1). To hasten the process of mechanical property assessment, a small-scale testing approach has been specifically designed to utilize small volumes of material from laboratory-scale, R&D extractions. Bespoke molds for the FAST machine were designed to produce small pellets utilizing just 4.2 g of titanium alloy powder. The pellets allowed the production of a cylindrical sample for compression testing, a test of resistance to deformation which can give indicative values of strength, and a thin disk suitable for microscopic studies.

In order to benchmark this new SR-derived alloy, Ti-6Al-4V (Ti-64) powder produced by the hydride dehydride (HDH) process from ingot was subjected to the same small-scale processing route, confirming the reliability and accuracy of the small-scale test procedure. Although mechanical properties of commercial alloys consolidated *via* FAST can be found,<sup>[8,9,10]</sup> tensile values are usually reported, or the samples have been produced under different consolidation conditions making direct comparisons invalid. Test results were also validated using mechanical property prediction software JMatPro.<sup>[11]</sup>

Prior to mechanical testing, the synthetic rutile-derived titanium alloy and Ti-64 were consolidated *via* FAST. Consolidation was achieved utilizing an FCT systeme GmbH spark plasma sintering furnace-type HP D 25. A bespoke set of molds (Figure 2(a)) were designed to produce a puck of 11 mm diameter with approximately 10 mm height (Figure 2(b)). The graphite mold set-up consisted of solid standard supports for 20 mm diameter mold assemblies coupled with a diameter reducing adapter, 11 mm punches and a cylindrical die. Due to the small size of the mold set-up, the usual axial pyrometer control could not be used, limiting the temperature to below 1100 °C. Instead, a 1.1-mm hole drilled 13 mm deep allowed for K-type thermocouples to monitor the temperature. Inside of the die was lined with graphite foil, before 4.2 g of powder was added. The samples underwent a predetermined sintering cycle consisting of a 100 °C min<sup>-1</sup> ramp rate to 1363 K (1090 °C) followed by a 30-minute dwell, all under an

applied force of 5 kN (52.6 MPa). Following consolidation, the graphite foil was removed by grit blasting and a thin disk was sectioned (11 mm × 2 mm) before the pellet was machined into a 6 mm diameter × 9 mm height cylindrical compression sample (Figure 2(b)).

As displayed in Figure 3(a), the SR-derived titanium alloy is angular in nature and contains internal porosity. SR-derived alloys have previously been consolidated to 98.6 pct density but no mechanical properties have been reported.<sup>[7]</sup> With respect to morphology, Ti-64 is angular, due to its production *via* HDH processing (Figure 3(c)). Table 1 provides details of the SR-derived titanium alloy and commercial Ti-64. Figure 3 displays the original powder morphology and subsequent microstructure post-FAST consolidation. Ti-64 reached full consolidation, whereas the SR-derived alloy has a slightly lower density, which is due to the presence of internal porosity within the original powder.<sup>[12]</sup> A standardized, non-optimized set of conditions were used for the consolidation of the alloys; it is thought a higher pressure and temperature would facilitate the full consolidation of the SR-derived alloy.<sup>[12]</sup> Further, a much larger particle size range was used for the SR-derived titanium alloy in comparison to the Ti-64 powder, with smaller particle size known to aid the densification process.<sup>[13]</sup>

Microstructures of the as-received powder are compared to their corresponding consolidated preforms achieved in Figure 3. Note, the microstructure achieved following consolidation is not optimum; consolidation *via* FAST is seen as an intermediate processing stage which would be complimented with subsequent forging and/or heat treatments. The consolidated SR-derived titanium alloy exhibits an  $\alpha + \beta$  microstructure, consisting of pro-eutectoid  $\alpha$  laths with  $\alpha + \beta$  lamellar in between. The microstructure observed is similar to previously reported SR-derived titanium alloy microstructures as well as commercially available  $\alpha + \beta$  alloys.<sup>[7]</sup> Similar microstructures are observed for both the consolidated Ti-64 and SR-derived titanium alloy, with Ti-64 displaying a finer lamellar structure with a higher volume fraction of  $\alpha$ .

After FAST consolidation, both alloys underwent room temperature axisymmetric compression testing, performed using a servo-hydraulic thermomechanical compression machine (TMC). Detailed descriptions of good practice for conducting compression tests have

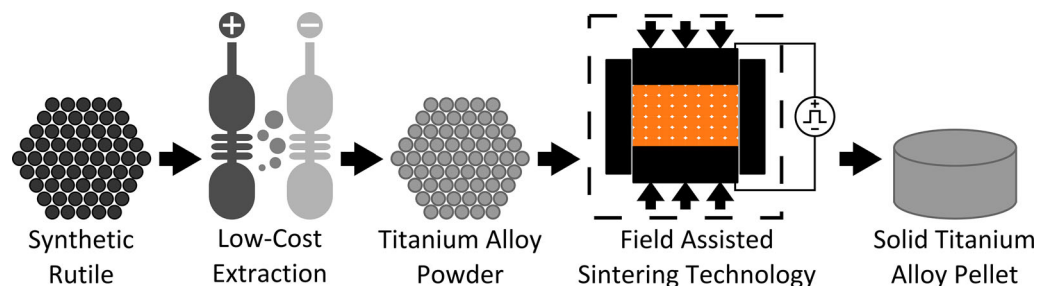


Fig. 1—Schematic illustration of solid-state production of fully dense titanium alloy pellets from a synthetic rutile feedstock.

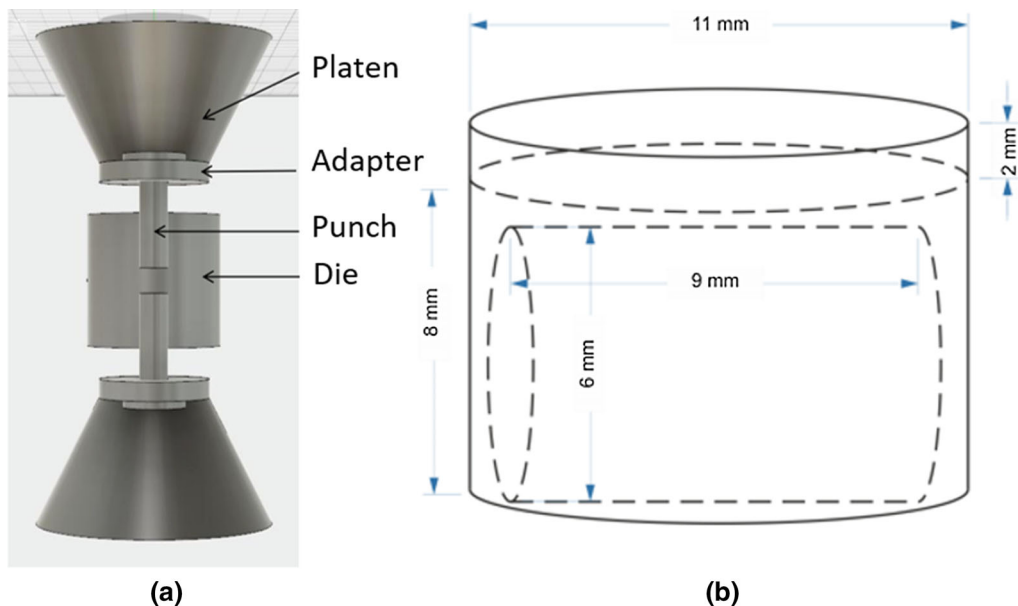


Fig. 2—(a) CAD image of FAST mold set-up manufactured from graphite (b) Schematic of FAST specimen and the orientation of sectioned compression cylinders.

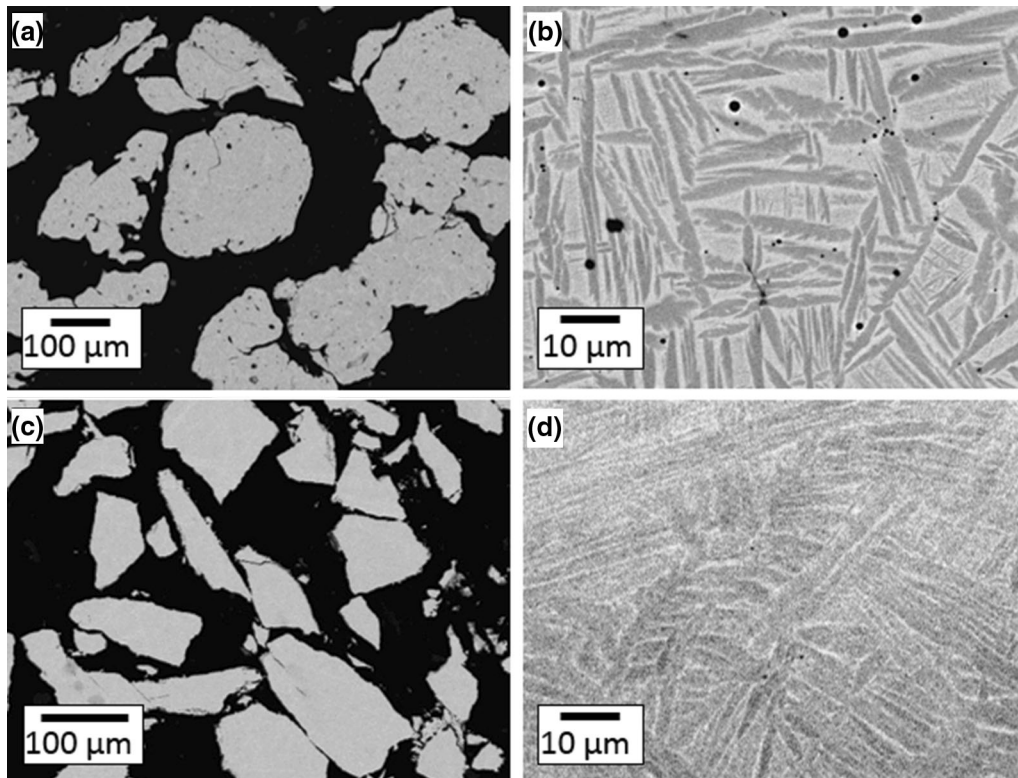


Fig. 3—Backscattered scanning electron micrographs, taken using a JEOL-JSM6490LV using an acceleration voltage of 20 kV (a) SR-derived titanium alloy Metalysis powder (b) FAST consolidated SR-derived titanium alloy (c) Ti-64 hydride-dehydride powder (d) FAST consolidated Ti-64.

been reported by Roebuck *et al.*<sup>[15]</sup> and these were followed as closely as possible. The 6 mm diameter by 9 mm high sample size maintained the recommended aspect ratio of 1.5. Temperature was not recorded as tests were conducted at room temperature; further the

addition of thermocouple holes in the samples would be significantly detrimental to the compressive yield behavior, due to the size of the hole compared with the sample height (>10 pct). A constant strain rate of  $0.1 \text{ s}^{-1}$  was applied up to a final strain of 0.7. Load, time, velocity,



**Table I. Morphology, Oxygen Concentration, Particle Size Range of SR-Derived Titanium Alloy and Ti-64 Powders Alongside Consolidated Post-FAST Density Achieved**

| Alloy | Morphology                 | Oxygen (ppm) | Particle Size Range ( $\mu\text{m}$ ) | Density (Percent) |
|-------|----------------------------|--------------|---------------------------------------|-------------------|
| SR    | angular, irregular, spongy | 3500         | 212 to 500                            | 97.5              |
| Ti-64 | angular, irregular         | 2067         | 45 to 150                             | 99.9              |

Oxygen content was measured using an Eltra ON-900. Post-FAST density values were determined using imaging software Image J.<sup>[14]</sup>

**Table II. Experimentally Determined Compressive Yield Strength and Strain to Failure Values of the SR Derived Alloy and Ti-64 at Room Temperature Using the Small-Scale Rig Alongside Literature Compressive Yield Strength Values of Cast Alloys and Modelled Tensile Strength Values**

| Alloy | Experimental<br>0.2 Pct Compressive YS (MPa) | Experimental Strain<br>to Failure | Compressive YS Literature (MPa)  | Modelled Tensile<br>YS <sup>[11]</sup> (Grain Size) |
|-------|--|-----------------------------------|--|---|
| SR    | 1061, 1099, 1109                             | 0.323, 0.329, 0.370               | —  | 1016 (5 $\mu\text{m}$ )                             |
| Ti-64 | 991, 991, 1009                               | 0.358, 0.368, 0.393               | 855 to 979 annealed, <sup>[16]</sup> 897 cast and HIPed <sup>[17,18]</sup> | 1058 (5 $\mu\text{m}$ )                             |

and displacement were recorded throughout the experiments. Each alloy was tested three times to assess repeatability, with the resulting data shown in Table II and comparative true stress-true strain curves shown in Figure 4.

All three repeats of both compression-tested alloys gave compressive yield strengths with a variance of 4.5 pct or less, which provides good validity and reliability of the small-scale testing approach (Figure 4 and Table II). Differences in strain to failure values between repetitions were no larger than those associated with standard large scale testing. A slightly larger difference in strain to failure values was found in the SR-derived titanium alloy than the Ti-64. This difference has been determined to be due to the higher porosity content resulting in premature failure due to the coalescence of pores.

In terms of validating the accuracy of the small-scale testing approach, the compressive yield strength values attained from FAST-processed material were compared with literature values for Ti-64 in the solution-treated condition, shown in Table II. Note, the solution-treated condition was chosen for comparison as this produces a similar condition to the as FASTed material following the slow cool from above the beta transus. Ti-64 exhibited a yield strength value similar to that reported in the literature, which were produced using standard extraction and casting techniques.

As a further validation test, thermo-physical simulation software JMatPro<sup>[11]</sup> was used to predict the yield strength values of the alloys tested, as shown in Table II. Although the modeling software is predicting tensile yield strength, generally the difference in compressive and tensile yield strength is small (<15 pct). As such, the predictive values were used as a guideline only. Overall, the software predicted similar values to those determined experimentally. The software has been programmed using data acquired from conventionally produced titanium; hence, it underlines the effectiveness of the FAST processing method, which is capable of mimicking values achieved *via* traditional casting methods and hot isostatic pressing (HIP). As the compressive

yield strengths produced *via* the small-scale test were similar to both literature values and values determined by the model, the validity of the small-scale test has shown to be acceptable. Hence, this work has shown how the small-scale testing approach utilizing small quantities of powder can reliably and accurately determine compressive yield strengths of titanium powders. This work also shows that titanium components produced *via* the FAST process are capable of achieving the same compressive yield strength as components processed through traditional melt-forging routes.

Following validation of the small-scale approach, a direct comparison of Ti-64 and the SR derived alloy can now be made. Both Ti-64 and SR-derived titanium alloy are  $\alpha + \beta$  alloys containing approximately >90 pct of the  $\alpha$  phase at room temperature. The lamellar microstructure observed in both alloys leads to their high compression yield strengths. Both alloys behave similarly, with comparable values for compressive yield strength and strain to failure. The high strength observed from the SR-derived titanium alloy is thought to be due to the slightly higher oxygen content and high quantity of remnant elements present, providing the alloy with substantial solid solution strengthening.

Note, despite the encouraging initial results from synthetic rutile derived alloys, it is worth stating that these are preliminary results. Before the full assessment of the viability of the introduction of SR-derived titanium alloys into commercial applications can be made, full, rigorous, up-scaled mechanical testing of this novel alloy must be completed. Hence, future work will consist of experimentation of a range of mechanical properties exhibited by the SR-derived titanium alloy alongside full economic impact studies.

Over the last 60 years, Ti-64 has been the dominant general purpose alloy in both the aerospace and biomedical industries. This paper demonstrates a low-cost alternative derived from SR with mechanical properties comparable to the workhorse Ti-64. For an alloy extracted directly from an ore-like material to produce similar mechanical properties to that of the titanium industry's most widely used alloy is

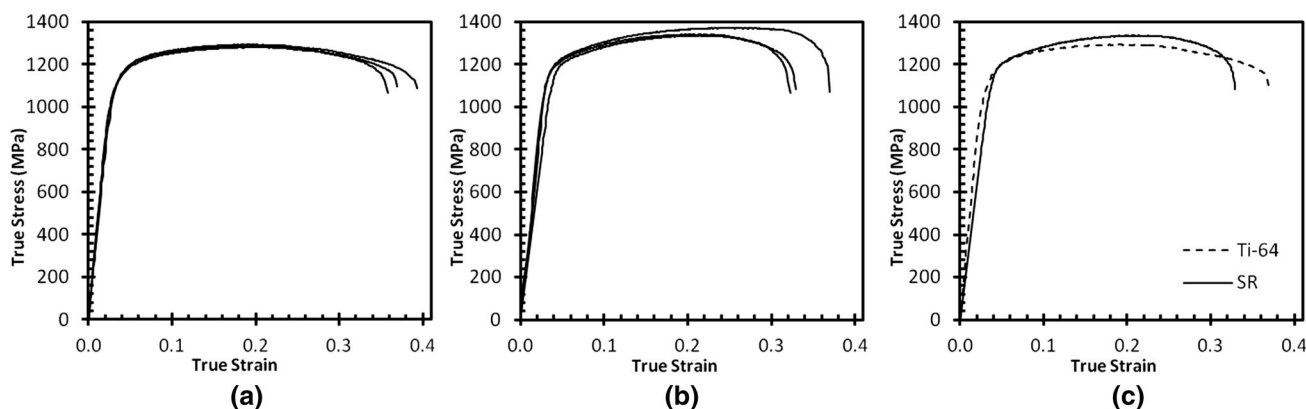


Fig. 4—True stress-true strain plots generated from uniaxial compression tests at RT and a strain rate of  $0.1 \text{ s}^{-1}$ ; (a) Ti-64 (3 repeat tests); (b) SR-derived titanium alloy (3 repeat tests) and (c) comparison of SR-derived titanium alloy and Ti-64.

unprecedented. Further, the synthetic rutile-derived alloy did not achieve full consolidation and it is not unrealistic to suggest that further improvements in the mechanical properties are likely to be observed when this is achieved.

With its promising mechanical properties, SR-derived titanium alloy, in combination with cost-effective near net shape powder metallurgy techniques such as FAST-*forg*e could provide a new production route and the step-change in economics which is necessary to lower the cost of components and infiltrate new markets, particularly the automotive industry. These cost reductions come from removal of the melting procedure, use of low-cost SR feedstock, reduced material wastage and machining *via* producing near net shape as well as the minimization of costly thermomechanical processing steps.

Further, the SR-derived titanium alloy is versatile by nature, the composition of which can be slightly modified depending on the source of ilmenite and extraction procedure. Hence, investigation of the synthetic rutile derived alloys is still in its infancy with many options to be explored for the full exploitation of this resource.

In summary, a small-scale testing approach designed for the mechanical assessment of small volumes of material has been validated. Ti-64 in powder form, consolidated *via* the FAST method, has achieved similar compressive strengths compared to the as cast and solution-treated conditions. A novel SR-derived titanium alloy achieved similar compressive yield strengths and strain to failure values of Ti-64, despite some remaining porosity (2.5 pct). With these initial promising results and the disruptive potential of the technology, further up-scaled investigations of the mechanical properties of the SR-derived titanium alloy consolidated *via* FAST are currently under investigation.

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