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# Article:

Weston, N.S. orcid.org/0000-0002-0515-4573 and Jackson, M. (2017) FAST-forge – a new cost-effective hybrid processing route for consolidating titanium powder into near net shape forged components. Journal of Materials Processing Technology, 243. pp. 335-346. ISSN 0924-0136

https://doi.org/10.1016/j.jmatprotec.2016.12.013

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# FAST-forge – a new cost-effective hybrid processing route for consolidating titanium powder into near net shape forged components

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7 Abstract

8 Reducing the high cost of titanium to a level where it can compete with currently used commodity metals 9 offers opportunities to many industries to exploit its excellent combination of properties to improve 10 performance or reduce weight. The key to decreasing cost is to reduce the number of processing steps to go 11 from ore to component, as well as material wastage from excessive machining. This paper describes a new 12 solid-state hybrid manufacturing route, termed by the authors as FAST-forge, for converting titanium alloy 13 powder into components with wrought properties in two steps; utilising field assisted sintering technology 14 (FAST) to produce a shaped preform billet that is finished to near net shape by a one-step precision hot forge. The route has been demonstrated at the laboratory scale using Ti-6Al-4V hydride-dehydride powder 15 by producing fully consolidated, microstructurally homogeneous, double truncated cone specimens directly 16 17 through FAST, which were then upset forged at a range of temperatures and strain rates. The 18 microstructural evolution and forging behaviour of the Ti-6Al-4V after FAST consolidation is similar to 19 conventional melt, multi-step forged product. Break up of primary  $\alpha$  at high strains was observed at 950°C 20 and 0.01 s<sup>-1</sup>, 0.1 s<sup>-1</sup>, and 1 s<sup>-1</sup>. There is good agreement between finite element modelling of the hot forging 21 and the experimental data, which will enable more complex shaped geometries to be produced via the 22 proposed FAST-forge route in future. Such a route could be used to consolidate powder from a lower-cost 23 alternative extraction method to become a disruptive technology that will enable a step-change in the 24 economics of titanium components.

*Keywords:* Spark Plasma Sintering (SPS); Pulsed Electric Current Sintering (PECS); Field Assisted Sintering
 Technology (FAST); Ti-6Al-4V; thermomechanical processing; hot forging.

#### 27 **1.** Introduction

28 Titanium alloys exhibit excellent properties such as a higher specific strength than steels, exceptional 29 corrosion resistance, high melting point and low thermal expansion. Yet titanium's high affinity for 30 embrittling interstitials, such as oxygen, requires inert atmospheres during extraction and downstream processing. Such requirements are reflected in the high cost of titanium mill product and its limited use in 31 32 non-aerospace sectors. This cost can be approximately broken down into two main areas, which is illustrated 33 by the example of 25 mm thick plate in Fig. 1. The first area is ingot production, accounting for around half of 34 the total cost, which encompasses the ore handling, Kroll process extraction, alloying and melting; most 35 commonly via vacuum arc remelting (VAR), sometimes in combination with electron beam or plasma arc 36 cold hearth remelting. The remaining cost is found in the second area of downstream processing, which in 37 this example is the thermomechanical processing of the VAR ingot; normally multi-stage forging and re-heats 38 to generate the component shape and required properties. As the complexity of the final component 39 increases so does the proportion of the cost from downstream processing due to additional costly steps, 40 such as secondary forging and machining. An approach that targets cost reductions in both areas is required 41 for titanium to compete with commodity metals in non-aerospace sectors. Combining an alternative 42 extraction method with subsequent cost-effective downstream processing offers the potential for significant 43 price decreases.

44 The opportunity presented by a viable lower-cost alternative to the sixty-year-old Kroll process has led to the 45 development of multiple different approaches around the globe; an overview and discussion of a variety of 46 these can be found in (Fray, 2008), with a selection briefly discussed here. In the UK, electro-deoxidation is 47 being developed (Mellor et al., 2015), including the production of novel titanium alloys directly from 48 synthetic rutile feedstock (Benson et al., 2016). Several methods are being investigated in the USA: using 49 hydrogen during the Kroll process' chlorination stage to produce TiH<sub>2</sub> powder, which can then be densified 50 and simultaneously dehydrided by a variety of methods, is reportedly occurring at the pilot-plant scale (Duz 51 et al., 2016); performing nearly continuous sodium reduction of TICl<sub>4</sub> (Armstrong et al., 1999); and

52 electrowinning from carbothermally reduced titanium oxide (Withers, 2015). In South Africa the use of 53 continuous metallothermic reduction of TICl<sub>4</sub> in molten salt is being trialled (Van Vuuren et al., 2011). In Australia the use of continuous magnesium reduction of TICl<sub>4</sub> in a fluidised bed reactor process is being 54 55 explored (Doblin et al., 2012). All these alternative extraction processes produce a powder or particulate 56 titanium product. Importantly, as Fig. 1 illustrates, alternative powder extraction routes alone will be 57 insufficient to achieve the cost reduction necessary for sectors such as the automotive industry. It is the 58 subsequent consolidation of powder into mill product and near net shape components that will have the 59 most dominant effect on cost reduction. Cost savings can be made in downstream processing by removing as 60 many of the traditional multi-stage thermomechanical processing steps as possible.



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62 Fig. 1: Chart demonstrating the two main areas of production costs for 25 mm titanium alloy plate when 63 conventionally processed; with relative cost factors for each sub-area also shown. Produced from data 64

reported in (Kraft, 2004).

65 Traditional powder metallurgy techniques are able to densify powders without the need for melting, due to

66 the mechanism of sintering; where adjacent surfaces bond due to diffusional processes that are enhanced by

67 the application of heat (German, 2014). FAST, also known as spark plasma sintering, allows the solid-state

68 consolidation of powders by combining the effects of high temperature with the application of uniaxial 69 pressure. The heat is generated through Joule heating as DC current is applied through a mould assembly 70 containing the powder, either continuously or pulsed in a chosen pattern, which allows very high heating 71 rates to be attained compared to more traditional sintering methods. Hydraulically actuated rams allow the 72 application of axial mechanical load to produce the required pressure. FAST is considered an effective 73 method for rapid sintering due to the high heating rates and the blend of heat and pressure; with broad 74 agreement that it can produce equivalent or improved properties, compared with conventional techniques 75 like hot isostatic pressing (HIP), whilst operating with reduced processing times and/or lower temperatures 76 (Munir et al., 2011). This has allowed improved sintering of a range of materials, some that were customarily 77 considered more problematic, such as WC (Orrù et al., 2009). The electric current appears to play a role in 78 enhancing the sintering beyond simple Joule heating, and it is routinely proposed that high localised currents 79 create the eponymous spark plasma, which increases sintering via a mechanism of particle surface cleaning 80 or localised melting/evaporation. However, there is currently insufficient experimental evidence of spark 81 plasma, suggesting that the term is misleading at best; Hulbert et al. were unable to detect it in a variety of 82 powders across a wide spectrum of conditions using a range of techniques (Hulbert et al., 2008). In the 83 absence of spark plasma, other authors have suggested the current might increase diffusion through 84 improving mass transport by electromigration; increased neck growth of copper spheres with increasing 85 current under FAST conditions of fixed temperature, pressure, and time has been shown (Frei et al., 2007). It 86 is clear that complex mechanisms are operating to produce the enhanced sintering that is seen and they are 87 not yet fully understood. From a cost saving perspective FAST may also offer benefits: a 90-95% energy 88 saving has been claimed when using FAST to consolidate TiAlO<sub>2</sub> – TiC composites when compared to hot 89 pressing, whilst also reporting a slight improvement in properties (Musa et al., 2009). There is an absence of 90 published work on producing anything other than simple disc shaped specimens via FAST and therefore the 91 limitations of this technology to produce complex geometries is currently unknown.

92 The authors previously indicated the capability of FAST in the sintering of a range of commercial and 93 lower-cost titanium alloy powders (Weston et al., 2015). It was shown that FAST is tolerant of powder 94 morphology and chemistry, and high heating rates could be used to lower processing times with minimal

95 effect on microstructure. Simple disc shapes of constant thickness achieved uniform powder packing and 96 consolidation with no density gradients, which allows microstructural homogeneity throughout specimens, 97 even when scaling up to larger sizes (250 mm diameter and 5 kg); additionally, it was shown that there was 98 limited pick-up (between 100-250 ppm) of carbon, oxygen, and nitrogen from the starting powders. The 99 ability to utilise feedstocks which are larger and angular, including potentially those from alternative 100 extraction methods or even recycled swarf, and still achieve high density and homogeneous microstructures 101 means that FAST has an advantage over traditional sintering operations as these feedstocks are lower-cost. 102 However, the authors believe that the geometries and mechanical properties required by most titanium 103 alloy components will not generally be producible by using FAST as a consolidation process in isolation. The 104 large-grained transformed  $\beta$  microstructure with grain boundary  $\alpha$  is not optimal for components that need 105 a good balance of properties; a bi-modal microstructure, produced by hot-working in the  $\alpha$ - $\beta$  phase region, 106 offers advantages for most applications (Lütjering, 1998). The production of complex near net shape 107 geometries directly via FAST may be possible in the future with further investigation although the 108 microstructure would in all likelihood still need refining. Nonetheless, FAST of titanium powder has the 109 potential to be an effective intermediate consolidation and shaping process prior to further 110 thermomechanical processing in the form of a closed-die hot forging operation. To be the most efficient and 111 cost-effective it is possible, with sufficient process design and control, that this could be a one-step near net 112 shape forging operation. To achieve the desired final post-forge geometry and strain levels, and thus 113 microstructures, it is likely that the preform billet produced via FAST will need shape and definition. Finite 114 element (FE) modelling has become a common tool to provide load and microstructural predictions during 115 complex forging operations, although a comprehensive data set is required to achieve this. Therefore, from a 116 process modelling point of view the effect of thermomechanical processing parameters on microstructural 117 evolution needs to be understood due to their inevitable variation, even when nominally isothermal forging. 118 Levels of strain, strain rate and temperature can significantly affect the microstructure of titanium alloys and 119 a large test matrix would be needed to characterise this if using traditional cylindrical axisymmetric 120 compression specimens. The novel double truncated cone testing approach (Jackson et al., 2000) allows this

microstructural characterisation in far fewer tests due to the predictable and controlled strain distribution in
the forged specimen. A double cone specimen can be tested at a set temperature and strain rate to give
information relating to a larger range of strains, from almost zero at the edge to high strains in the centre.
Small specimen dimensions can limit temperature variations so that a good approximation of isothermal
forging can be realised, as well as allowing metallographic preparation and inspection of the entire

specimen.



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solid-state processing route for producing titanium alloy components from powder.

130 The aim of this paper is to demonstrate, at the laboratory scale, that it is possible to produce components 131 from powder in two steps, as shown schematically in Fig. 2; using Field Assisted Sintering Technology (FAST) 132 to produce a shaped preform billet, which is finished to near net shape with one-step precision forging. 133 Depending upon the application it is envisaged that a subsequent heat treatment would allow tailoring of 134 the microstructure if required and/or a minimal finish machining operation would produce an acceptable surface roughness. This novel solid-state hybrid processing route, termed by the authors as "FAST-forge", 135 will allow manufacturing of components with forged properties for dynamically loaded applications from 136 137 titanium alloy powders. It is hoped that the mechanical properties achieved by the additional forging of FAST

- material will allow FAST-*forge* products to be used in areas and applications not conventionally considered possible for as-sintered PM components. It is envisaged that with further development FAST-*forge* will become disruptive technology for a range of sectors. The combination of this cost-effective consolidation method with powder from a lower-cost extraction method will provide a step-change in the economics of titanium components.
- 143 2. Materials and Methods

#### 144 2.1 Experimental Approach



- Fig. 3: Photograph demonstrating the outcome at each stage of the two-step FAST-forge process; the starting
  Ti-6Al-4V HDH powder (left) to the intermediate shaped preform billet, a double truncated cone FAST
  specimen with a light surface machine (centre), and the final forged specimen (right).
- 149 The experimental approach aimed to demonstrate three key developments. Firstly, the capability of FAST to 150 produce shaped preforms to be used in the FAST-forge process. Secondly that the FAST-forge concept, of 151 producing a component with wrought properties from powder in two steps, was viable through a 152 laboratory-scale demonstration, see Fig. 3. Thirdly, to link microstructural evolution of FAST produced 153 preforms to thermomechanical processing parameters by utilising the double truncated cone specimen 154 geometry as the shaped preform billet; thus gaining valuable information for future process optimisation 155 through FE modelling. Ti-6Al-4V hydride-dehydride (HDH) powder was used for this proof of concept 156 demonstration, to enable comparison with conventional wrought product, as well as setting a benchmark for 157 future work with lower-cost powder from an alternative extraction method.



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Fig. 4: Schematic showing the two methods used to make the double truncated cone specimens. Method 1 produced a 100 mm diameter x 15 mm thick FAST disc, which smaller cylinders were extracted from via electro-discharge machining (EDM), and then machined to the final dimensions shown (known as "bulk" double cone specimens). Method 2 used shaped graphite inserts in a 20 mm diameter FAST mould assembly to produce shaped preforms, which then had a surface machine to give the final dimensions shown (known as "shaped" double cone specimens).

Two methods were used to create the double cone specimens, see Fig. 4. The first method was to electrical discharge machine 20 mm diameter x 15 mm thick cylinders from a 100 mm diameter x 15 mm thick FAST disc, which were then machined to the final dimensions shown in Fig. 4 (known as "bulk" double cone specimens hereafter). The second method was to produce shaped preforms by placing shaped graphite inserts into a 20 mm diameter FAST mould assembly, which were also machined to the same final dimensions (known as "shaped" double cone specimens hereafter). The same sintering cycle and hot compression testing conditions were applied for both methods of double cone specimen production. The aim of creating additional specimens from bulk material was to allow a comparison of behaviour with shaped preform specimens produced directly via FAST; thus demonstrating that the shaped FAST method does not adversely affect either the powder consolidation or subsequent forging response.

### 175 2.2 Materials



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- Fig. 5: Light micrographs of the Ti-6Al-4V HDH powder's particle morphology after etching with Kroll's
   reagent (a) and microstructure under cross-polarised light (b).
- 179 The Ti-6Al-4V HDH powder was purchased from Reading Alloys Inc., (an Ametek Company), Robesonia, PA,
- 180 USA, and certified to contain 6.34% Al, 4.02% V, 0.21% Fe, 0.026% C, 0.016% H, 0.013% N, and 0.16% O;

therefore meeting the ASTM Grade 5 specification, except 0.001% excess hydrogen. The size range was
75-150 μm, with 96.7% of particles within these limits. The powder morphology was angular and irregular in
shape, see Fig. 5a, with a microstructure of equiaxed α grains approximately 5-10 μm in diameter, see Fig.
5b.

185 **2.3 Methods** 

186 2.3.1 Field Assisted Sintering Technology

187 The FAST systems used to consolidate the powder in these experiments were manufactured by FCT Systeme 188 GmbH. The 100 mm disc used for the bulk double cone specimens was made using the Type H-HP D 250 189 system based at Kennametal Manufacturing (UK) Ltd. The shaped double cone specimens were made using 190 The University of Sheffield's Type HP D 25 system, see Fig. 6. The methodology was the same for both 191 machines. The mass of powder required (520 g for bulk and 15 g for shaped) was placed into a graphite ring 192 mould, simply between two graphite pistons for the bulk disc specimen, or with extra shaped graphite 193 inserts for the shaped double cone specimens. Graphite foil was used to line the mould assembly to aid with 194 specimen removal and prolong mould life. The mould assembly was then placed between the two 195 conducting hydraulic rams in the machine's vacuum chamber and held with a put-on load of 5 kN to ensure 196 good electrical contact was made. The sintering cycle used was as follows: the vacuum chamber was 197 evacuated, pulsed DC current was applied in the pattern of 15 ms on and 5 ms off. The values of current and 198 power rose steadily from initial values of 0.45 kA and 2.0 kW to 1.12 - 1.18 kA and 5.9 - 6.3 kW during the 199 dwell period for the shaped double cone specimens. The value of power for the 100 mm disc was a 200 maximum of 163 kW during the heating period and 37 - 42 kW during the dwell period (a sensor fault 201 prevented recording of current data). The heating was uncontrolled up to 450°C due to the operating limits 202 of the pyrometer. Above 450°C a constant heating rate of 100°Cmin<sup>-1</sup> was used up to the dwell temperature 203 of 1200°C, points A-C in Fig. 6. Once 600°C was reached, point B, the pressure began to increase, with a rate 204 so that the maximum of 50 MPa would occur simultaneously with the maximum temperature, point C. A 205 dwell time of 30 minutes at maximum conditions was then used, points C-D. For the 100 mm bulk disc the

current was then turned off and the specimen allowed to "free" cool, points D-E. For the shaped double
cone specimens, the current was used to achieve a "controlled" cooling rate to match the bulk disc cycle.
The "controlled" cool is achieved by the FAST furnace software reducing the applied current to a level where
the heat loss exceeds the Joule heat generated by the correct amount to attain the desired cooling rate. The
pressure was also gradually decreased back to 5 kN during the cool.



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212 Fig. 6: Photograph of the FCT Systeme GmbH Type HP D 25 FAST Furnace at The University of Sheffield (right);

showing detail of the graphite mould assembly held between the conducting hydraulic rams (inset right).

214 Schematic cross-section showing the main components of the FAST system and mould assembly used (bottom

215 *left) and a graph outlining the variation in major processing parameters during a typical FAST cycle (top left).* 

- 216 2.3.2 One-Step Forging
- 217 The replication of the one-step forging stage was undertaken using The University of Sheffield's
- thermomechanical compression testing machine, see Fig. 7. The test furnace contained two M22 steel tool

- 219 posts, where the upper one was servo-hydraulically actuated, allowing a constant strain rate deformation to
- a strain of 1.15. A fast thermal treatment unit (FTTU), located immediately in front of the test furnace,
- allowed induction heating of the double cone specimens at 4°Cs<sup>-1</sup> to the test temperature, with a hold of 30
- seconds to minimise any oscillation.



Fig. 7: Photographs outlining the major components of The University of Sheffield's thermomechanical
 compression machine (a), close-up view of the tool posts and furnace (b) (note the furnace has been moved

226 to the rear to enable viewing of the tool posts), close-up of a double truncated cone specimen held in the 227 robot gripper arms (c).

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Robot gripper arms were used to manipulate the specimen during testing; allowing positioning at the correct height before automatically moving into the FTTU and then into the test furnace for the one-step forge, followed by specimen withdrawal for a water quench. A 1.1 mm hole located centrally in the edge of the double cone specimens allowed an N-type thermocouple to be attached; giving control during induction heating and temperature data during deformation. A boron nitride coating was applied to limit interstitial pick up and reduce friction. A data logger recorded time, temperature, load, velocity, and displacement information throughout the test. Bulk double cone specimens were deformed at 850°C, 950°C, and 1050°C, and at strain rates of 0.01 s<sup>-1</sup>, 0.1 s<sup>-1</sup> and 1 s<sup>-1</sup>. The shaped double cone specimens were deformed at 950°C, at strain rates of 0.01 s<sup>-1</sup>, 0.1 s<sup>-1</sup> and 1 s<sup>-1</sup>.

#### 237 Finite Element Simulation of the One-Step Forge 2.3.3

238 The finite element software DEFORM<sup>™</sup> (Scientific Forming Technologies Corporation, 2016) was used to 239 simulate the compression tests of the double cone specimens to give the strain profiles across the specimens 240 seen in Fig. 11a-11c. These strain profiles allow the linking of microstructural evolution to thermomechanical 241 processing parameters (strain, strain rate, temperature). Due to the axisymmetric nature of the specimens it 242 was possible to use a 2-D model of half the double cone geometry, meshed with 3160 elements, which 243 simplified the simulation and reduced processing time. Rheology data from previous unpublished work, in a 244 tabular form (stress values at a range of strains for each testing condition), was used for the material model, 245 see Table 1 in the appendix. The material response was assumed to be fully plastic. For each test condition 246 the initial temperature was set to that recorded by the thermocouple at the start of the experimental 247 compression, and the measured temperature profile during the tests was used as a boundary condition for 248 the specimen ensuring the simulation matched the experimental conditions as closely as possible. Two 249 non-meshed rigid platens were used to represent the tool posts as their experimental deformation can be 250 treated as negligible. The movement of the upper platen was controlled by setting a condition to produce a

constant global average strain rate to match the experiment; the software determined the magnitude of
displacement necessary to achieve the set strain rate for each time step. Contact boundary conditions
between the specimen and platens were established with a constant shear friction factor (m) of 0.3.

254 2.3.4 Metallography

Typical metallographic preparation for Ti-6Al-4V was used for all specimens: sectioned in half parallel to the compression direction, hot-mounted in Bakelite, followed by grinding using progressively finer SiC papers then 9 μm diamond suspension, and finally chemical/mechanical polishing using colloidal silica of 0.05 μm with 20% hydrogen peroxide. Microstructural observations were performed using a Nikon Eclipse LV150 light microscope under reflected light conditions, either in bright field or polarised light mode. Kroll's reagent was applied as an etchant, if needed, until increased microstructural detail was visible.

#### 261 3. Results and Discussions

#### 262 3.1 Microstructures after FAST

263 Preliminary experiments demonstrated that the initial cooling rate after the current is turned off was 264 significantly higher for the 20 mm diameter mould assembly at ~20 °Cs<sup>-1</sup> than for the 100 mm diameter 265 mould at  $\sim$ 0.33 °Cs<sup>-1</sup>, see Fig. 8a. There is greater thermal mass for the larger mould assembly due to the 266 increased amount of graphite required and it therefore takes longer to cool. This work sought to emulate the 267 bulk material as closely as possible to allow direct comparison and therefore a "controlled" cool to match 268 the "free" cool of the bulk was utilised for the shaped double cone specimen. The difference in 269 microstructure produced by free and controlled cool can be seen in Fig. 8b and 8c; as expected the quicker 270 free cool produced much finer  $\alpha$  laths, where the controlled slower cool coarsened them to a size similar to 271 the bulk specimen (directly compared in Fig. 9.)





Fig. 8: Graph showing the temperature profiles during FAST processing of three types of Ti-6Al-4V specimen
(a). A 100 mm diameter disc used for bulk double cone specimens; allowed to "free" cool after current switchoff (solid line). A 20 mm shaped mould when allowed to "free" cool after current switch-off (dotted line) with
associated microstructure (b). A 20 mm shaped mould with "controlled" cool (dashed line) and associated
microstructure (c).



284 colony structure with some amount of  $\alpha$  phase present on the grain boundaries (Joshi, 2006). The prior  $\beta$ 285 grain size ranges from approximately 200-600  $\mu$ m with an  $\alpha$  lath width in the region of 3-10  $\mu$ m. The high 286 temperature and level of consolidation during the dwell period allowed  $\beta$  grain growth beyond the 287 dimensions of the initial powder particles for both bulk and shaped specimens, which is a significant change 288 in microstructure from the starting powder. This  $\beta$  grain growth demonstrates the high density achieved as 289 at lower levels of consolidation the remaining porosity acts to pin grain boundaries and prevent growth. 290 Image analysis, using the software ImageJ (Rasband, 1997), of multiple bright-field micrographs across each 291 specimen allowed the calculation of density as 99.88% for the shaped double cone specimens and as 99.87% 292 for the bulk double cone specimens. These values are slightly greater than the 99.01% stated by (Xu et al., 293 2014) and slightly less than the 99.9% reported by (Kim et al., 2014) for HIP of Ti-6Al-4V powders, which 294 claimed to have tensile strength and elongation comparable to wrought material. The porosity will also be 295 healed further during the forging process, which will further increase tensile properties and more 296 importantly fatigue strength.

297 It can also be seen in Fig. 9 that microstructural homogeneity was achieved in the shaped double cone 298 specimen, with comparable micrographs from top to bottom and from centre to edge. Graphite has a higher 299 electrical resistivity than Ti-6Al-4V and consequently acts as the main heating element in the mould 300 assembly. Thus, it was hypothesised that a shaped mould, with non-uniform graphite thickness in the axial 301 direction, would produce uneven heating as well as a more complex pressure distribution that would lead to 302 microstructural variations; although this is not observed in the shaped double cones at this scale. If 303 temperature variations were present, they were small enough not to have had a significant effect at the 304 processing conditions used for these experiments. Although this may not be the case if a lower processing 305 temperature is required, especially as the  $\beta$  transus temperature is approached, where there will be a 306 reduction in the diffusional rates with increasing  $\alpha$  content. It should be noted that the shape used here is 307 still a relatively simple axisymmetric profile and that further experimentation will be needed, with the aid of 308 FE modelling, to fully understand the difficulties involved in producing semi-complex shaped preform billets 309 as part of the FAST-forge processing route.



Fig. 9: Micrographs of Ti-6Al-4V double truncated cone specimens produced via FAST at a dwell temperature of 1200°C. Showing microstructures from a shaped specimen (Shaped 1-4) at the locations outlined in the top left diagram; and a characteristic microstructure of the homogeneous bulk specimen (bottom right).

314 **3.2** Experimental Load-Displacement Curves

315 Due to the non-uniform cross-sectional area of the double cone specimens it is not possible to produce 316 meaningful plots of stress versus strain during the thermomechanical compression. Consequently, the data 317 is presented as plots of load versus displacement, which can be seen in Fig. 10 for deformations at 950°C and 318 a range of strain rates. The effect of strain rate is clearly demonstrated; as the rate of deformation increases 319 so does the force required to achieve equivalent displacement. The influence of temperature can be seen in 320 the load-displacement curves at 850°C, 950°C, and 1050°C, see Fig. 12, where there is a marked reduction in 321 the load required for equivalent displacement as temperature increases due to an increase in the more 322 easily deformed  $\beta$  phase and an increase in dynamic recovery and recrystallisation processes. There are 323 some small variations between the load-displacement behaviour of shaped double cone and bulk double 324 cone specimens; at 1 s<sup>-1</sup> the bulk specimen required a slightly higher load, at 0.1 s<sup>-1</sup> the bulk specimen required a slightly lower load, and at 0.01 s<sup>-1</sup> the bulk specimen required a higher load initially before 325 326 finishing requiring a lower load. The level of variation seen is minimal and would be expected even when

327 testing duplicate samples from the same parent material due to attempting to control the large number of 328 variables seen during hot working of metals. Frictional variations were limited by using similar quantities of 329 lubricant for each test and cleaning the tool posts between tests, but small differences would still occur. The 330 strain rate was closely controlled by the testing software and whilst small oscillations around the set value 331 occurred these were the same for every test and it is thought not large enough to cause the variations in 332 load seen. Due to less than perfect control of the heating in the FTTU causing small oscillations around the 333 target test temperature, typically ±5°C, there was some variation in the initial temperature between 334 samples, which would also have a small effect on the loads required.



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Fig. 10: Graphs of load-displacement curves during hot upset forging of Ti-6AI-4V double truncated cone
specimens at 950°C and strain rates of 0.01 s<sup>-1</sup>, 0.1 s<sup>-1</sup>, and 1 s<sup>-1</sup>. Bulk (solid lines) and shaped (dashed lines).

#### 338 3.3 Microstructure Evolution Post One-Step Forging

339 The microstructural evolution for both bulk and shaped double cone specimens under hot uniaxial

- 340 compression at 950°C, for strain rate regimes of 0.01 s<sup>-1</sup>, 0.1 s<sup>-1</sup> and 1 s<sup>-1</sup> is shown in Fig. 11a-11c respectively.
- 341 The location of the light micrograph images, 3 mm apart along the specimen centreline, is also marked on
- 342 the FE simulation generated strain profile.



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At low strains, 12 mm from the centre, there is slight coarsening of the primary  $\alpha$  and the transformed  $\beta$ grains manifest a finer secondary  $\alpha$  lath structure than post-FAST due to the water quench and higher cooling rate. As strain increases, moving towards the specimen centre, it can be seen across both bulk and shaped double cone specimens at all strain rates that primary  $\alpha$  platelets rotate and tend to align perpendicular to the forging axis; all primary  $\alpha$  appears to be fully aligned 6 mm from the centre (a strain of ~1.1). At higher strains break-up of the  $\alpha$  platelets into approximately 1-5 µm spheroidal  $\alpha$  particles is

- 353 observed. As strain rate increases the time for diffusion dominated globurisation of primary α platelets
- decreases and it can be seen the amount of spheroidal  $\alpha$  particles decreases from Fig. 11a-11c.



Fig. 11b: Light micrographs of the microstructural evolution with increasing strain from edge to centre of the double truncated cone specimens after forging at 950°C and 0.1 s<sup>-1</sup>; produced from bulk (top) and via shaped FAST (bottom).

359 The microstructural evolution of shaped double cone specimens compared to double cone specimens

- 360 machined from bulk is similar for all strain rates and strains. There has been a significant coarsening of the
- 361 primary  $\alpha$  in both the bulk double cone specimen at 0.1 s<sup>-1</sup> and the shaped double cone specimen at 1 s<sup>-1</sup>.
- 362 This is due to these specimens failing to remain in the robot gripper arms upon retrieval from the test
- furnace so that the quenching did not occur automatically and a slower initial cool was experienced; the

specimens were manually quenched to room temperature approximately 60-120 s after forging. This slower
 cooling rate somewhat hinders a direct comparison between the two specimen types; however, the same
 microstructural trends are observed. The observed microstructural evolution is comparable to that reported
 during the hot working of conventionally produced Ti-6Al-4V with a colony α microstructure, as reported by
 (Semiatin et al., 1999).



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370 Fig. 11c: Light micrographs of the microstructural evolution with increasing strain from edge to centre of the

- 371 double truncated cone specimens after forging at 950°C and 1 s<sup>-1</sup>; produced from bulk (top) and via shaped
- 372

FAST (bottom).

#### 374 3.4 Finite Element Simulation

375 The load upon the upper tool with respect to its stroke (displacement) was extracted from the data 376 produced by running FE simulations of each experimental point in the test matrix. This data is plotted against 377 the experimentally recorded values of load and displacement for the bulk double cone specimens in Fig. 12. 378 Only data for the bulk double cone specimens is shown to allow clearer comparison, as it has been shown 379 that the shaped double cone specimens produced very similar load data. Overall there is good visual 380 agreement between experimental and predicted values, which gives confidence that the predicted strain 381 profiles are accurate. However, there is slight under prediction at 850°C and 950°C, but slight over prediction 382 at 1050°C. The simulation was set up to mirror the recorded temperature profiles of the experiment, which 383 due to adiabatic heating were not fully isothermal, therefore load changes due to temperature variability 384 were accounted for. However, the material model used was discrete tabulated data (at temperatures of 385 850°C, 950°C, and 1050°C and strain rates of 0.01 s<sup>-1</sup>, 0.1 s<sup>-1</sup>, and 1 s<sup>-1</sup>) with linear interpolation between 386 conditions, which may not be realistic. A constant shear friction factor (m) of 0.3 was used and appears to 387 give good visual agreement with experimental conditions, using boron nitride as a release agent, as the end 388 shape of the simulated curves largely matches the experimental even if the absolute values differ. Friction 389 only has a large effect at higher displacements where the contact area has increased; it can be seen in the 390 850°C at 0.01 s<sup>-1</sup> curve that a good match is achieved early in the test but the curves diverge at the end, 391 which suggests that this test occurred under increased friction conditions. The material used to produce the 392 data for the FE model was Ti-6Al-4V HDH powder processed in an 80 mm mould with a similar FAST cycle to 393 this work, except a lower pressure of 21 MPa and allowed to free cool; the cooling rate was intermediate to 394 those demonstrated in Fig. 8 and produced a transformed  $\beta$  microstructure with  $\alpha$  laths of intermediate 395 thickness to those shown here. This difference in starting microstructure may also explain some of the 396 disparities between simulation and experiment. It should be noted that as tabulated data has been used the 397 FE model can only be employed with confidence within the processing window defined by the extremes of 398 the experimental conditions.



Fig. 12: Graphs comparing the load-displacement curves during the upset forging of double truncated cone
specimens at 850°C (a), 950°C (b) and 1050°C (c) at strain rates of 0.01 s<sup>-1</sup>, 0.1 s<sup>-1</sup> and 1 s<sup>-1</sup> (as labelled); from
bulk FAST material (solid lines) against those obtained from DEFORM<sup>™</sup> FE simulation (dotted lines).

#### 405 **4.** Conclusions

- This paper has demonstrated at the laboratory scale that it is possible to produce a fully dense and
   microstructurally refined forged titanium alloy specimen in only two steps from powder. In the
   long-term the authors believe that this proposed cost-effective hybrid processing route, termed
   FAST-*forge*, combined with a lower-cost powder from an alternative extraction method will be disruptive
   technology that will enable a step-change in the economics of titanium alloys.
- Directly producing shaped FAST double cone specimens did not negatively affect microstructural or
   deformational behaviour when compared to double cone specimens machined from homogeneous bulk
   material. There is very good visual correlation between the two types of specimens. This establishes that
   using FAST to produce shaped preforms has the potential to be an effective intermediate step in the
   FAST-*forge* process. Further work is required to explore the possibilities and limitations of the
   technology prior to scale-up, but in future it should be possible to accurately produce shaped FAST
   preforms for a variety of final components.
- 418 At the current level of FAST technology commercially available the cost-effectiveness achieved by the 419 FAST-forge processing route will vary from component to component; an economic assessment on case 420 by case basis would be required. The initial set-up costs may negate benefits for small batch production 421 and speed of processing limitations may exclude products requiring continuous or very large/quick batch 422 production. However, with expensive feedstocks such as titanium there may still be cost reductions to 423 be found via FAST-forge. The tooling costs for FAST compare favourably against HIP, where the steel can 424 bonds to the titanium and needs to be machined away, as the majority of the graphite mould assembly 425 is reusable. The longevity and cost-effectiveness of the mould assembly in terms of both material and 426 geometry needs to be investigated further to give an understanding of tooling costs as the technology 427 progresses in size and part complexity.

- The response of Ti-6Al-4V FAST material under forging conditions is very similar to that seen when
   thermomechanically working conventional Ti-6Al-4V billet material; post-sintering FAST preforms have
   characteristics similar to conventional melt, multi-step forged product.
- The agreement between experimental load-displacement data and FE simulation data gives confidence
   that the material model utilised can be used to model the forging of more complex geometries as the
   FAST-forge process develops.
- Initial examination of the microstructural evolution indicates the level of strain, temperature and strain
- 435 rate required to break up the post FAST microstructure and achieve a bimodal  $\alpha$ - $\beta$  microstructure, but
- 436 further analysis is needed to tie key microstructural features to thermomechanical processing
- 437 parameters for use in a simple microstructural prediction model.
- It is further anticipated that if the required mechanical properties of a component are identified then a
- 439 microstructure necessary to meet these can be predicted. Using FE simulation, linked to a
- 440 microstructural model, the shape of the preform could be iteratively optimised so that the one-step
- 441 precision forging operation can produce the correct levels of strain at the forging conditions to yield the
- 442 appropriate microstructure to meet the property requirements.

443

#### 444 Acknowledgements

The authors acknowledge the Engineering and Physical Sciences Research Council's Advanced Metallic
Systems Centre for Doctoral Training for funding N. S. Weston (Grant Number EP/G036950/1). Thanks go to
the Defence Science and Technology Laboratory for additional funding, and specifically Dr Matthew Lunt for
his support. The authors also recognise Dr Fatos Derguti for technical discussions and Dr Adam Tudball of
Kennametal Manufacturing (UK) Ltd. for technical knowledge and assistance when using their large-scale
FAST furnace.

# 452 Appendix

TABLE 1: Tabulated flow stress data at the indicated testing conditions and strain levels that was
 used as the material model for FE modelling of the forging of Ti-6Al-4V HDH powder consolidated using
 FAST at a heating rate of 100°Cmin<sup>-1</sup> with dwell conditions of 1200°C and 50 MPa held for 30 minutes.

Strain	Test Conditions								
	850°C 950°C						1050°C		
	0.01 s <sup>-1</sup>	<b>0.1</b> s <sup>-1</sup>	1 s <sup>-1</sup>	0.01 s <sup>-1</sup>	<b>0.1</b> s <sup>-1</sup>	1 s <sup>-1</sup>	0.01 s <sup>-1</sup>	<b>0.1</b> s <sup>-1</sup>	1 s <sup>-1</sup>
0.000	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
0.010	31.3	98.4	94.0	24.7	50.3	39.6	7.2	20.0	43.3
0.015	57.7	146.3	149.1	30.6	58.0	57.6	8.8	28.1	44.0
0.020	90.5	175.6	188.2	32.1	60.5	72.4	10.0	31.5	45.0
0.030	111.5	192.0	213.4	34.0	60.2	75.5	10.6	32.6	46.3
0.050	114.9	192.3	220.4	34.3	59.3	78.5	11.5	33.4	47.4
0.100	114.5	188.0	225.4	34.8	58.4	82.9	12.4	34.1	48.6
0.150	112.5	183.7	227.5	34.6	57.4	87.3	13.2	34.4	49.7
0.200	110.0	178.1	229.6	33.6	56.3	89.5	14.1	34.1	50.8
0.250	106.9	172.8	231.5	33.0	54.8	89.8	14.9	33.7	51.9
0.300	105.1	168.1	228.1	32.4	53.8	89.9	15.8	33.2	53.0
0.400	98.8	160.0	218.5	31.5	51.9	88.7	16.3	32.2	53.3
0.500	95.6	153.7	208.5	30.6	50.7	87.6	16.8	31.2	53.0
0.600	92.6	147.8	202.1	30.1	50.5	87.2	16.2	30.8	53.1
0.700	89.1	143.4	195.4	29.7	50.2	87.2	15.3	30.3	53.1
0.800	84.1	138.4	189.8	29.3	50.2	87.2	14.9	30.2	53.1
0.900	82.1	135.6	187.8	29.0	50.2	87.2	14.2	30.2	53.1
1.000	79.3	135.2	186.6	28.6	50.2	87.2	13.8	30.2	53.1
1.200	73.8	135.2	186.6	28.2	50.2	87.2	12.8	30.2	53.1
5.000	73.8	135.2	186.6	28.2	50.2	87.2	11.5	30.2	53.1

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   Microstructure and Properties of Ti-6Al-4V Alloy from Atomized Powder. J. Mater. Sci. Technol. 30,
   1289–1295. doi:10.1016/j.jmst.2014.04.011
- 518 TABLE 1: Tabulated flow stress data at the indicated testing conditions and strain levels that was
- used as the material model for FE modelling of the forging of Ti-6Al-4V HDH powder consolidated using
- 520 FAST at a heating rate of 100°Cmin<sup>-1</sup> with dwell conditions of 1200°C and 50 MPa held for 30 minutes.
- 521 Fig. 1: Chart demonstrating the two main areas of production costs for 25 mm titanium alloy plate when
- 522 conventionally processed; with relative cost factors for each sub-area also shown. Produced from data
- 523 reported in (Kraft, 2004).

- Fig. 2: Schematic diagram outlining the two-step hybrid "FAST-forge" process a proposed cost-effective
  solid-state processing route for producing titanium alloy components from powder.
- 526 Fig. 3: Photograph demonstrating the outcome at each stage of the two-step FAST-forge process; the starting
- 527 Ti-6Al-4V HDH powder (left) to the intermediate shaped preform billet, a double truncated cone FAST
- 528 specimen with a light surface machine (centre), and the final forged specimen (right).
- 529 Fig. 4: Schematic showing the two methods used to make the double truncated cone specimens. Method 1
- 530 produced a 100 mm diameter x 15 mm thick FAST disc, which smaller cylinders were extracted from via
- 531 electro-discharge machining (EDM), and then machined to the final dimensions shown (known as "bulk"
- 532 double cone specimens). Method 2 used shaped graphite inserts in a 20 mm diameter FAST mould assembly
- to produce shaped preforms, which then had a surface machine to give the final dimensions shown (known as
  "shaped" double cone specimens).
- Fig. 5: Light micrographs of the Ti-6Al-4V HDH powder's particle morphology after etching with Kroll's
  reagent (a) and microstructure under cross-polarised light (b).
- 537 Fig. 6: Photograph of the FCT Systeme GmbH Type HP D 25 FAST Furnace at The University of Sheffield (right);
- 538 showing detail of the graphite mould assembly held between the conducting hydraulic rams (inset right).
- 539 Schematic cross-section showing the main components of the FAST system and mould assembly used (bottom 540 left) and a graph outlining the variation in major processing parameters during a typical FAST cycle (top left).
- 541 Fig. 7: Photographs outlining the major components of The University of Sheffield's thermomechanical
- 542 compression machine (a), close-up view of the tool posts and furnace (b) (note the furnace has been moved
- to the rear to enable viewing of the tool posts), close-up of a double truncated cone specimen held in the
  robot gripper arms (c).
- 545 Fig. 8: Graph showing the temperature profiles during FAST processing of three types of Ti-6Al-4V specimen
- 546 (a). A 100 mm diameter disc used for bulk double cone specimens; allowed to "free" cool after current switch-
- 547 off (solid line). A 20 mm shaped mould when allowed to "free" cool after current switch-off (dotted line) with
- 548 associated microstructure (b). A 20 mm shaped mould with "controlled" cool (dashed line) and associated
- 549 *microstructure (c).*
- 550 Fig. 9: Micrographs of Ti-6Al-4V double truncated cone specimens produced via FAST at a dwell temperature
- 551 of 1200°C. Showing microstructures from a shaped specimen (Shaped 1-4) at the locations outlined in the top
- 552 *left diagram; and a characteristic microstructure of the homogeneous bulk specimen (bottom right).*
- 553 Fig. 10: Graphs of load-displacement curves during hot upset forging of Ti-6Al-4V double truncated cone
- 554 specimens at 950°C and strain rates of 0.01 s<sup>-1</sup>, 0.1 s<sup>-1</sup>, and 1 s<sup>-1</sup>. Bulk (solid lines) and shaped (dashed lines).

- 555 Fig. 11a: Light micrographs of the microstructural evolution with increasing strain from edge to centre of the
- double truncated cone specimens after forging at 950°C and 0.01 s<sup>-1</sup>; produced from bulk (top) and via
- 557 shaped FAST (bottom).
- 558 Fig. 11b: Light micrographs of the microstructural evolution with increasing strain from edge to centre of the
- double truncated cone specimens after forging at 950°C and 0.1 s<sup>-1</sup>; produced from bulk (top) and via shaped
  FAST (bottom).
- 561 Fig. 11c: Light micrographs of the microstructural evolution with increasing strain from edge to centre of the
- double truncated cone specimens after forging at 950°C and 1 s<sup>-1</sup>; produced from bulk (top) and via shaped
  FAST (bottom).
- 564 *Fig. 12: Graphs comparing the load-displacement curves during the upset forging of double truncated cone*
- 565 specimens at 850°C (a), 950°C (b) and 1050°C (c) at strain rates of 0.01 s<sup>-1</sup>, 0.1 s<sup>-1</sup> and 1 s<sup>-1</sup> (as labelled); from
- 566 bulk FAST material (solid lines) against those obtained from  $DEFORM^{TM}$  FE simulation (dotted lines).