Exploitation of field assisted sintering technology (FAST) for titanium alloys

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Abstract

Field assisted sintering technology (FAST), also known as spark plasma sintering (SPS), is increasingly utilised to process powders/particulates of engineering alloys and metal-based composite materials. FAST is currently extensively used at laboratory scale by research institutes and universities as a rapid and cost-effective process to consolidate powders. This includes investigating new alloy compositions and material combinations, improving established materials' properties, and consolidating materials considered challenging/impossible through conventional sintering techniques. FAST is gaining traction for industrial applications with possible benefits as an alternative to hot isostatic pressing or conventional melt-wrought processing. FAST preform complexity is improving and near-net-shape components are becoming a possibility. Demonstrator components for the aerospace and automotive sectors, including aeroengine blades, brake callipers and rocker arms, have been produced from titanium alloy powders. FAST has also been demonstrated as an effective intermediate step for consolidating a range of feedstocks, including recycled materials, into shaped billets that can be further processed to refine shape and/or properties. Hybrid processes such as FAST-*forge* and FAST-DB have been developed that can produce affordable titanium components with forged properties. This paper presents the current status, emerging developments, and challenges of FAST for titanium-based powders and particulates.

Introduction

Background

Titanium alloys have an excellent combination of properties that makes them desirable for applications in many industries. However, a high affinity for embrittling interstitial elements (oxygen and nitrogen) requires inert atmospheres during extraction/fabrication processes. This, combined with downstream processing

complexity and wastage, generates high final part costs. The high cost is a major barrier that limits usage of titanium alloys to applications where performance takes precedence over price. Therefore, there is significant research interest in reducing the cost of titanium alloy components.

The cost of titanium alloy mill products can be approximately divided equally between (1) extraction and primary processing to produce ingots, and (2) subsequent thermomechanical processing of the ingot [1]. Additional downstream processing (e.g. forging/machining/joining) is required to produce finished components, which further increases cost. Current processing routes have largely been optimised, with ever reducing scope for further significant improvements.

A viable lower-cost Kroll extraction process alternative is a sizeable business opportunity, which has directed significant research efforts to this area; Fray [2] has summarised developing approaches. Several processes have been scaled up to pilot-plant level, but commercial quantities of low-cost and/or high quality material are not yet available [3]. Lower-cost extraction in isolation will not produce the desired savings due to the highlighted cost structure. Cost-effective downstream processing that reduces processing steps, energy/time requirements, and material wastage is also needed. One possibility is powder metallurgy (PM), which produces fully dense near-net-shape parts with competitive properties in minimal steps. However, current commercial powders are not low cost, limiting the price benefits of PM for titanium alloys [3]. Several alternative extraction methods produce powders/particulates, offering one source of lower cost feedstock if commercialised. There is also scope for PM to utilise products currently classed as low-value or waste, such as powders that are out of specification for additive manufacturing (AM), or swarf from machining processes. Combining a low-cost feedstock with a novel, cost-effective, and solid-state downstream processing route will provide a step-change in the economics of titanium [4].

Field Assisted Sintering Technology (FAST)

Field assisted sintering technology (FAST), or spark plasma sintering (SPS), is a PM process that simultaneously applies electric current and uniaxial load to a powder specimen and die assembly. The electric current causes Joule heating in the die and/or specimen, allowing rapid heating rates and reduced processing time. SPS emerged in the mid-1960s with the publication of Inoue's patent [5]. The process and terminology have developed over the last fifty years, giving alternative designations such as pulsed electric current sintering (PECS) and FAST [6,7]; the main variations have been summarised by Grasso et al. [8]. This paper uses FAST to describe processes using electric currents and voltages to enable resistive sintering processes; typically lower than a few thousand amps and a few tens of volts.

In general, FAST uses low voltage and high current to produce high temperature whilst applying axial load under vacuum or gas atmosphere. Powder is encapsulated in a conducting die assembly (most commonly graphite) to enable the Joule heating the process is reliant upon. Die complexity varies, but the simplest contains powder in a ring between two punches and then uses spacers to contact with the electrodes, which are actuated to allow load application. A typical processing cycle heats and loads the die and powder sample at a defined rate to a maximum temperature and load, dwelling for a set time, then cooling. Temperature is

normally measured in one of three locations; near the upper surface of the sample by pyrometer, external surface of the ring by pyrometer, or by thermocouple embedded within the ring. A minimum load is required throughout to ensure electrical contact is maintained, and the electric current (DC) can be continuous or pulsed. This gives five primary FAST process variables: dwell time, maximum temperature, maximum load, heating rate, and electric current type.

There is consensus that FAST offers technological and economic advantages compared to conventional sintering techniques [6–13]. It is frequently reported materials can be processed to higher density at lower temperatures and/or reduced times with improved performance/properties. Ultra-fine grain (UFG) or nanoscale microstructures can be produced with suitably prepared powders, which is not possible with conventional routes such as hot pressing or hot isostatic pressing. FAST has consolidated materials considered difficult/impossible to process via conventional sintering. The exact mechanisms allowing these benefits remain unclear, in particular if the electric current has additional intrinsic effects beyond Joule heating. A huge variety of materials have been successfully FAST processed [6], also demonstrating tolerance of feedstock particle morphology, size, and size distribution with appropriate tailoring of process parameters. FAST is established as an attractive tool to improve conventional material processing and as a unique method for investigating new materials. Consequently, there have been exponential increases in scientific publications and patent applications since the 1990s [14]. FAST of ceramic and functional materials is more popular than metals. However, this paper provides an overview of current research on FAST of titanium alloys, highlights some emerging developments, and summarises future challenges.

Current Research on FAST of Titanium Alloys

There are three main areas when discussing FAST processing of titanium alloys. The first is single material systems, where just one type of material is consolidated. The second is deliberately porous materials, typically for biomedical applications. The third is joining two or more materials (both similar and dissimilar), which can produce metal matrix composites (MMCs) or functionally graded materials (FGMs). A recent review offers additional detail to that provided below [15].

Single Material Systems

As discussed, the high cost of titanium alloy powders means FAST needs to produce improved properties and/or reduced cost to be considered effective for processing single alloys. Property improvement investigations tend to focus on achieving high density whilst retaining UFG microstructures, although it is usually necessary to perform intensive milling of powders prior to sintering. Cost reductions may be achieved through lower temperature and/or shorter processing times, utilisation of lower-cost feedstocks, or reduced material wastage through near-net-shape production.

A variety of particle sizes, size distributions, and morphologies of commercially pure titanium (CP-Ti) powders have been successfully consolidated via FAST. Eriksson et al. [16] concluded that consolidation is dominated by plastic deformation of particles and reported observing no evidence of sparks/plasma. Ertorer et

al. [17] demonstrated bimodal microstructures and tensile strength of 840 MPa with 27% ductility by cryomilling powder prior to FAST. Pascu et al. [18] and Sharma et al. [19] produced fully dense compacts from titanium hydride (TiH₂) powder, a potentially lower-cost feedstock, by using FAST to cause desorption of hydrogen above 700°C. Zadra et al. [20] reported that carbon, oxygen, and nitrogen contamination is minimal during FAST. Weston et al. [21] confirmed this, and demonstrated FAST's feedstock morphology tolerance by achieving full density with spherical and angular powders. Shon et al. [22] found particle size had little effect on final microstructure, but additional oxygen content in the finer powder increased hardness.

Ti-6Al-4V (Ti-6-4) has also been successfully consolidated via FAST. Weston et al. [21] demonstrated scalability by processing spherical powder into a 5.5 kg, 250 mm diameter, fully dense billet with homogeneous microstructure. Menapace et al. [23] forged specimens after FAST, which behaved equivalently to conventional cast/wrought product. Weston and Jackson [24] used FAST to produce shaped preforms that were then forged to refine geometries and improve properties; terming this two-step process 'FAST-forge'. Several studies have utilised lower temperature and shorter processing times to produce UFG, nanoscale, or bimodal microstructures [25–28], which typically requires using fine powders or extensive milling; high (compressive) strengths are frequently achieved but with (significantly) reduced ductility.

High-strength beta alloys have also been FAST processed by Calvert et al. [29] (pre-alloyed Ti-5Al-5Mo-5V-3Cr) and Yang et al. [30] (blended elemental Ti-10V-2Fe-3Al). Both reported homogeneous microstructures and good mechanical properties that could be tailored via altering processing conditions.

For biomedical applications Zeming et al. [31] produced Ti-Ag alloys, due to silver's antibacterial properties. Karre et al. [32] studied Ti-Nb alloys as alternatives to alloys with vanadium, which has potential toxicity issues. Mompou et al. [33] reported a bimodal microstructure with high strength and good ductility after FAST of mechanically milled Ti-25Nb-25Zr. Lu et al. [34] prepared fully dense Ti-Mo alloys for dental applications.

FAST of titanium aluminides (TiAl), which are weight saving candidates in aeroengine applications, has been investigated due to difficulties in conventional processing [35–38]. FAST of TiAl has been a research focus of CEMES in Toulouse [39–42], with the Ti-48Al-2W-0.08B IRIS alloy with reportedly excellent mechanical properties developed [43,44], and single-step FAST consolidation to near-net-shape of a γ -TiAl turbine blade with excellent properties [45].

Porous Systems

The excellent biocompatibility of titanium alloys make them ideal for dental, hip and knee implants [46]. Porous materials help address issues with elastic moduli mismatch between bone and implant, infections, and limited component lifespan. Nicula et al. [47] reported a Ti-13V-7Mn-4Cr-Al specimen with high surface roughness and porosity with improved cell adhesion. Yamanoglu et al. [48] studied porous Ti-5Al-2.5Fe as an alternative to possibly toxic vanadium alloys. Zhang et al. [49] produced specimens with 30-70% porosity, and

elastic moduli strength similar to bone, using NaCl powder as a subsequently dissolved space holder. Quan et al. [50] also used NaCl to create porous Ti-6-4 with elastic moduli and strength similar to bone.

Multi Material Systems

Diffusion bonding materials via FAST has been investigated, with potential benefits if techniques such as welding are not possible. Creating FGMs by joining multiple layers, or MMCs by bonding reinforcements into a matrix, is also possible. Daoush et al. [51] oxide dispersion strengthened Ti-12Mo-6Zr by milling elemental powders with alumina, achieving high density, hardness, and yield strength, but near complete ductility loss. Munir et al. [52] investigated adding 0.5 wt.% multi-walled carbon nanotubes to CP-Ti; reporting 1056 MPa compressive strength and 27% failure strain, but no results on tensile strength/ductility. Decker et al. [53] added 10-15 wt.% Ti-42.3Al-3.8Nb-1Mo-0.1B to Ti-6Al-2Sn-4Zr-2Mo to increase high temperature capabilities and reported increased tensile strength with reduced ductility from room temperature to 650°C. Calvert et al. [54] reported a 200 MPa increase in compressive strength with no ductility loss when adding 0-25 wt.% of a 2 GPa yield strength TiFeMo alloy to Ti-5Al-5Mo-5V-3Cr. Ghesmati Tabrizi et al. [55] reported complete formation of TiB whiskers with higher density and reduced clustering after FAST of Ti-6-4 and B₄C powders, compared to vacuum sintering. Grützner et al. [56] increased hardness, stiffness, and compressive strength of Ti-5Al-5Mo-5V-3Cr by formation of TiB/TiC through B₄C powder additions, although ductility was almost zero. After FAST, Ozerov et al. [57] hot multiaxial forged a CP-Ti specimen with 17 wt.% TiB to a total strain of 5.2, which achieved a considerable increase in room temperature ductility with a small decrease in strength. Lagos et al. [58] addressed scalability issues with a 200 mm diameter by ~20 mm thick Ti-6-4 specimen reinforced by 10% TiC, which had homogeneous density and microstructure.

FAST allows diffusion bonding titanium alloys to each other and to other metals. Joining titanium to steel is nearly impossible via welding due to forming brittle intermetallic phases. However, Miriyev et al. [59] joined Ti-6-4 to AISI 4330 steel, reporting reduced intermetallic formation and a 250 MPa bond strength. Kumar et al. [60] joined CP-Ti to AISI 304L steel, achieving a 260 MPa bond strength. He et al. [61] joined solid Ti-6-4 to solid Ti-6-4, with grain growth across the interface and a bond strength 91% of the bulk; a significant gain over hot pressing. Pope et al. [62] FAST diffusion bonded dissimilar titanium alloy powders, showing excellent bond integrity with no voids/cracks for all combinations of CP-Ti, Ti-6-4, and Ti-5Al-5Mo-5V-3Cr; terming this process 'FAST-DB'.

Functional grading can be performed via FAST, and correct design of the gradient allows transition between materials where a distinct bond would fail. Fujii et al. [63] reported functionally grading from titanium alloy to alumina without large flaws or delamination. Martin et al. [64] reported a FGM variation with a spatially controlled 3D microstructure in an 'architected' material; a Ti-6-4 AM scaffold was FAST processed after filling with a dissimilar titanium alloy powder.

Titanium Alloy Components via FAST

The previous section showed FAST is useful for laboratory-scale research into a wide variety of materials. Specimen sizes were usually less than 40 mm diameter by a few millimetres thick, which allowed investigation of process parameters and sintering behaviour with limited quantities of material. However, meeting current engineering challenges and demonstrating industrial applicability is required for FAST to develop beyond research institutes and attract significant commercial investment. This includes improving product size, near-net-shape capability, increasing throughput, and reducing costs. The following section presents case studies that illustrate using FAST to produce titanium alloy components.

An Innovative Way to Produce γ -TiAl Blades

A high impact case study was the use of FAST to produce near-net-shape gamma titanium aluminide (γ -TiAl) turbine blades by Voisin et al. [45], see Figure 1. TiAl alloys have a beneficial combination of properties for low-pressure turbine blades to help meet increasing performance/environmental demands of next generation aeroengines. Yet, TiAl alloys suffer from low room temperature ductility and components have a difficult/expensive manufacturing route consisting of complex heat treatments and machining. Successful near-net shaping of γ -TiAl blades shows FAST is a viable route to assist in overcoming these barriers. Combining this with the alloy developed by Voisin et al. [43], which has excellent mechanical properties at room/high temperatures after a single FAST cycle, may offer significant benefits. This study [45] was the first time in the scientific literature that complex shaped parts, comparable to their conventional counterparts, were produced using FAST.

A full-scale (80 mm by 50 mm) near-net-shape high-pressure turbine blade was produced from Ti-48Al-2Cr-2Nb alloy powder via FAST. The researchers recognise TiAl will not be used for high-pressure turbine blades due to higher property requirements, but the geometric complexities (particularly large changes in thickness from root to sail) allowed FAST process development. Controlling densification/temperature gradients, through careful design of graphite dies by finite element (FE) modelling, was essential to achieve full component density with homogeneous microstructures, see Figure 1b. Shaped punches were used to produce the fully dense blade, with additional inserts to fill gaps between punches and die, see Figure 1d and 1e. Furthermore, the large thickness difference between root and sail necessitated a split punch design, where part of the punch initially pre-densified the root before the whole blade was further compacted [65]. This multi-part die allows movement and redistribution of stresses during initial loading, preventing premature die failure. The Ti-48Al-2W-0.08B IRIS alloy [44] can be FAST processed to give an optimised near-lamellar microstructure, which exceeds the property requirements of a low-pressure turbine blade. It was additionally shown that once shaped FAST dies have been developed it is easy to substitute the materials being processed; a fully dense niobium-silicide blade was produced with the only alteration an increased dwell temperature.

Figure 1 – Near-net-shape high pressure turbine blade obtained via FAST of a pre-alloyed Ti-48-2-2 powder. (a) CAD drawing of blade component. (b) Temperature during FAST calculated by FEM. (c) Photograph of near-net-shape blade after FAST. (d) Schematic of the graphite die showing punches in final position with fully dense specimen. (e) Exploded die view, showing how the inserts give shape to the blade [45]. Reproduced with permission from Advanced Engineering Materials.

Titanium Rocker Arms via FAST-forge

Contrary to the previous case study, it may not always be possible to produce the required geometries and mechanical properties by using FAST in isolation. Weston and Jackson [24] developed FAST-forge, which is a cost-effective solid-state hybrid manufacturing route. Titanium alloy powders/particulates are converted into components with high structural integrity and wrought properties in two steps; using FAST to form shaped preforms that are finished to near-net-shape by precision hot forging, see Figure 2.

Figure 2 – Schematic outlining the FAST-forge process – a cost-effective solid-state hybrid manufacturing route to convert powders/particulates into titanium alloy components. Reproduced with permission from the Journal of Materials Processing Technology.

The automotive industry can benefit from titanium’s properties, although current high costs prevent greater utilisation. FAST-forge technology would initially be well suited to low-volume high-value manufacturers. A proof-of-concept study by University of Sheffield researchers demonstrated FAST-forge producing Ti-6-4 rocker arms for internal combustion engine applications. The rocker arm is currently manufactured from steel by drop forging 32 mm diameter bar stock, with ~47% material wastage as forging flash.

Four 30 mm diameter by 220 mm bars and four shaped preforms were extracted from a 250 mm diameter by 30 mm thick FAST billet produced from Ti-6-4 hydride-dehydride (HDH) powder, see Figure 3a. FE modelling of the forging process allowed optimisation of the preform shape to minimise material wastage whilst ensuring die filling. Shaped FAST preforms were not directly produced due to the available funding, but the previous case study shows the suggested shape is well within FAST capabilities. The bars and preforms were forged using an identical set-up to the current steel rocker arm process, but at reduced temperature. The Ti-6-4 rocker arms forged from FAST bars gave the expected 40% mass reduction and the shaped FAST preforms gave a 55% reduction in material wasted as flash, see Figure 3b.

This case study demonstrates FAST-forge can be applied to current automotive components to enable enhanced performance and/or reduced emissions. Importantly the FAST preforms were directly processed in the current supply chain; no modification of the forging equipment was necessary. Processing a lower-cost

feedstock via *FAST-forge* could provide an attractive step-change in the economics of titanium alloy automotive components.

Figure 3 – Photographs showing (a) shaped FAST preforms prior to forging, and (b) forged rocker arms from shaped preform (left), FAST bar (centre), and current steel component (right).

Optimising Components with Dissimilar Titanium Alloys by FAST-DB

Recently, Pope et al. [62] demonstrated FAST is effective for diffusion bonding dissimilar titanium alloy powders; termed “FAST-DB” by the authors. Titanium alloy components are frequently subjected to complex operating conditions where using single alloy chemistries or microstructures can compromise optimal performance. Introducing different alloys/microstructures into particular sub-component regions would allow the tailoring of properties to meet location specific requirements, allowing designers greater freedom. Such an approach is complex and costly via conventional melt-wrought processing and joining techniques. FAST-DB allows solid-state consolidation and joining of dissimilar alloys with a high level of control and flexibility; Figure 4a shows an AM polymer mould, removed prior to sintering, used to achieve powder partitioning during die preparation. Distinct bond lines are observable after FAST and excellent location control is achieved, see Figure 4b.

Additionally, FAST-DB bonds have excellent structural integrity with no cracks, non-bonded regions, or porosity. Failure occurs in the lower strength/toughness alloy, away from the bond, during tensile/fatigue testing, see Figure 5a. A characteristic feature of FAST-DB bonds that further demonstrates integrity is grains that span the interface, giving grains with both alloy chemistries/microstructures, see Figure 5b. The bond diffusion zone can be predicted using commercial thermodynamic modelling software. FAST-DB billets can also be subsequently forged, analogous to *FAST-forge*, but with multi-material preforms. Demonstrator eye bolt components were closed-die forged from FAST-DB bars; see Figure 5c for a Ti-6-4 – CP-Ti example. Importantly the bond line retains its integrity post-forging, with no cracks/voids visible. Forming flow lines

through the bond during forging further increases strength and is an advantage over conventional joining techniques.

Figure 4 – Photographs of (a) FAST-DB lay-up arrangement using an AM polymer mould in a 250 mm diameter graphite die. (b) Slices of the sintered FAST billet with CP-Ti embedded into Ti-6-4; heat tinting reveals “FAST-DB” running through the billet.

Figure 5 – (a) Photograph of FAST-DB tensile test specimens showing bond structural integrity; higher-strength undeformed Ti-6-4 on the left side and lower-strength plastically deformed CP-Ti on the right side. (b) Backscattered electron micrograph of a FAST-DB bond showing boundary grains having mixed alloy

chemistry/microstructure; Ti-6-4 left and Ti-5Al-5Mo-5V-3Cr right. (c) Photograph of eye bolt forged from a FAST-DB billet with the bond line clearly delineated by the flash; Ti-6-4 left and CP-Ti right.

A Low-Cost Titanium Alloy Feedstock for FAST

As discussed in the introduction, combining lower-cost feedstocks with FAST is a key factor to delivering a step-change in titanium alloy component cost. Commercially available powders are expensive, which minimises the cost-effectiveness of FAST processing. However, work at the University of Sheffield has demonstrated a potential low-cost feedstock. Current titanium component manufacturing tends to include significant material removal via machining operations. There are difficulties returning this machining swarf into the supply chain due to purity/performance issues, which lowers its value.

Cleaned and graded Ti-6-4 swarf was successfully consolidated via FAST at the University of Sheffield. Full density and a homogeneous microstructure were achieved that compare favourably with Ti-6-4 HDH powder processed under the same FAST cycle, see Figure 6. Research is ongoing to assess mechanical properties of FAST processed swarf and performing subsequent forging to produce demonstrator components for testing. Further work needs to address concerns around swarf cleanliness, contamination, and variability. Whilst it remains unlikely the safety conscious aerospace sector will adopt recycled swarf FAST components for safety critical dynamically loaded applications, there are promising signs that titanium alloy swarf could be a low-cost feedstock for the manufacture of non-safety critical components via processes such as FAST, FAST-*forge*, and FAST-DB.

Figure 6 – Cross-polarised light micrographs of Ti-6-4 (a) machining swarf and (b) HDH powder after FAST processing under equivalent conditions.

Concluding Remarks

FAST is an excellent tool for researchers investigating solid-state processing of titanium alloy powders/particulates. The characteristic processing conditions have allowed unique compositions, microstructures, combinations of materials, and levels of porosity to be demonstrated using a wide variety of

established and developing alloys. It is apparent FAST is remarkably tolerant of titanium alloy feedstock characteristics, with the ability to control processing parameters to achieve desired densities and microstructures for particular applications.

Much of the current laboratory-based research reported focusses on small thin disc specimens with little consideration of the feasibility and cost of scaling up to commercial applications. Issues with powder quantity, contamination, and processing times mean caution is needed for materials reliant on extensive milling prior to FAST, although outstanding properties have been demonstrated. Care should also be taken when extrapolating results from laboratory-scale FAST equipment to larger industrial FAST facilities, particularly with heating/cooling rates.

The highlighted case studies begin to tackle areas that need to be addressed when considering FAST for industrial applications; scalability, shape complexity, feedstock cost, and mechanical properties. Although, further work in these areas, and others such as throughput, is required for FAST to become a commercially viable processing route. Robust and adaptable FE modelling capable of predicting powder densification behaviour, temperatures throughout the die assembly, and ideally microstructure will also be an essential tool in moving FAST out of research institutes into industry. However, there are promising signs that using field assisted sintering technology to process titanium alloy components will continue to develop and become a valuable tool for designers and engineers to exploit in the coming years.

Copyright Acknowledgements

Figure 1 – Combination of Figure 1 and Figure 2 reprinted from “Voisin T, Monchoux J-P, Durand L, et al., An Innovative Way to Produce γ -TiAl Blades: Spark Plasma Sintering. *Adv. Eng. Mater.* 2015;17:1408–1413.”, with permission from Wiley.

Figure 2 – Figure 2 reprinted from “Weston N.S. and Jackson M., 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 (2017) 335–346.”, with permission from Elsevier.

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