The Solid-State Spray Forming of Low-Oxide Titanium Components

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INTRODUCTION

This paper presents a novel approach for the production of essentially oxide-free titanium parts in near-net shapes. The hydride-dehydride (HDH) process is used to produce oxide-free titanium (OFTi) powder; the powder is then used as feedstock for the solid-state spray forming (SSF) process. Older spray forming techniques (e.g., thermal, Osprey, and low-pressure plasma spray)[1–4] are not particularly suited to the production of parts of near-net shape and typically introduce supplementary oxidation.

The other process most often used for obtaining near-net-shape titanium parts is powder metallurgy (P/M). The primary material benefits of SSF over P/M is improved strength and increased ductility due to extremely small grain size and low oxide concentration, respectively. SSF and P/M processes use similar feedstock materials, but SSF reduces the manufacturing steps from three for P/M (pressing, sintering, and hot isostatic pressing) to one for SSF (spray forming the OFTi powder). The handling of micrometer-sized powder is currently a deterrent to titanium P/M because of the propensity for metal dust explosions. The SSF process combined with the HDH reactor is expected to mitigate these problems because the feedstock powders are more coarse, and inert gases are used to convey the powder to the nozzle for deposition.

PRODUCING NEAR-NET-SHAPE TITANIUM COMPONENTS THROUGH HDH AND SSF

Hydride/Dehydride Production of Titanium Powders

The continuous HDH production of titanium powders is a new concept for the fabrication of micrometer-sized, essentially OFTi powders. The process can deliver powder directly to downstream processes, such as SSF, for fabrication into finished parts.

SSF is a proprietary process developed by Innovative Technology for the near-net-shape fabrication of metallic components.[5] OFTi powder is produced in micrometer size within a HDH reactor. Figure 1 contains a schematic representation of the system. The reactor uses high-temperature hydrogen gas to remove the oxide layer from coarse titanium and convert the titanium metal to titanium-hydride particles. Titanium-hydride powder is sufficiently friable to spontaneously fracture into micrometer-sized particles.[6] Next, the hydrogen is removed by increasing the temperature, which renders fine oxide-free metal particles. The metal particles must be kept in a gas suspension, because without an oxide or nitride coating, they will readily agglomerate. Oxygen and nitrogen must be excluded from the process also to reduce the risk of particle combustion or explosion. An inert gas (e.g., helium) is used as a purge gas to remove the hydrogen from the dehydride section of the reactor and as a process gas for transporting the OFTi powder to downstream processes such as SSF.

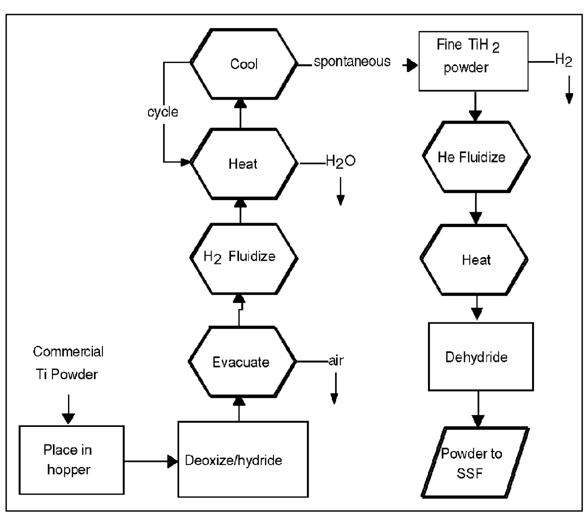

Figure 1. A schematic of the HDH process for the production of micrometer-sized titanium powder.

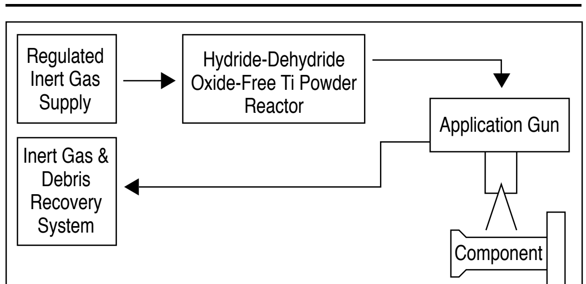

Figure 2. The SSF system for fabricating near-net-shape OFTi components.

Solid-Spray Forming of Near-Net-Shape Components

SSF is a new concept for fabricating near-net-shape components using OFTi powders. This process uses the HDH powder reactor to produce micrometer-sized titanium powders that are rendered oxide-free in passage through the reactor. As a low-temperature deposition process, SSF does not melt the OFTi feedstock and, thus, reduces small grain size. These features permit the near-net-shape fabrication of titanium or titanium-alloy components without metallurgically altering the properties of the feedstock material. Lower oxide concentration will lead to higher ductility while smaller grain size will result in increased strength per the Hall-Petch relationship.[7]

The SSF of OFTi is performed with a patented process developed by Innovative Technology (Figure 2).[8] For this application, the process employs a modified, two-phase, converging-diverging deposition nozzle to accelerate the micrometer-sized OFTi particles entrained in an inert gas. The OFTi particles are deposited onto a substrate or into a mold to enable the near-net-shape fabrication of specific components. The high-speed collision of the OFTi particles causes true metallurgical bonds to form upon contact. Metallurgical bonding is achieved exclusively through solid-state reaction—bulk melting does not occur.[9] Other spray-forming techniques (e.g., thermal, low-pressure

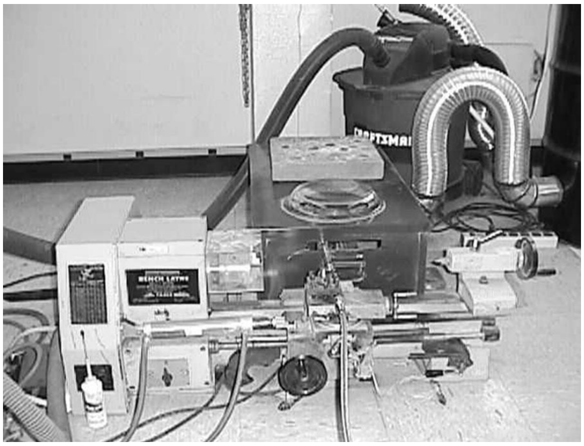

Figure 3. The nozzle translation lathe system.

plasma spray, and Osprey) cannot be used to deposit OFTi powders.

Debris generated during the SSF fabrication process is removed with a coaxial suction nozzle or suction cabinet driven by a debris-recovery system. The debris-recovery system filters contaminants from the recovered effluent and enables the recycling of excess powder and the inert gas.

ECONOMICS AND MARKET POTENTIAL

The cost of operating the HDH reactor to produce 2 kg/h of OFTi is estimated to be approximately \$1.60–2.75/kg, assuming that the inert carrier gas is recycled after the removal of hydrogen and other contaminants. The capital equipment cost for the HDH reactor is approximately \$50,000, exclusive of gas-recovery systems. With a theoretical deposition rate of 2 kg/h, the cost of operating a small SSF nozzle (1.6 mm throat) using helium in a recycle mode is expected to be \$10/kg. The combined cost of producing OFTi powder and then SSF into near-net shape is, thus, approximately \$12–15/kg. The capital equipment cost for a complete SSF unit is approximately \$250,000, including the cost of the HDH reactor.

It may be possible to reduce the cost of the OFTi production by limiting the hydride conversion to a superficial layer on each particle. This cost reduction cannot be accurately estimated at this stage because the threshold temperature for completely removing the oxide layer from the powder is not yet known. This mode of operation would not permit the production of finer titanium powders because the friable characteristics of the titanium-hydride powder are not realized. It is important to note that the above costs are for near-net-shape components and, thus, compare favorably to parts fabricated with conventional techniques.

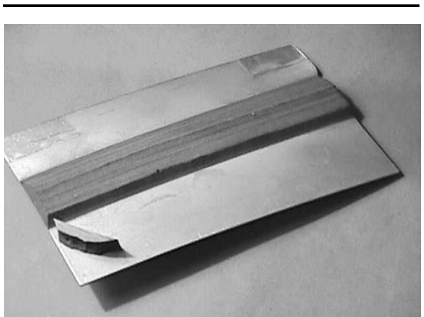

Figure 4. An SSF sample of OFTi on aluminum substrate (a cross-section of sample on left) powder and driving the SSF nozzle. The saddle bed of the lathe, equipped with a pneumatic cylinder, was used to translate the nozzle within the debris recovery cabinet.

Titanium is used extensively by the aerospace, defense, marine, automotive, chemical, pulp and paper, textile, medical, mining, minerals, metal processing, petroleum, and sports industries. Expanded use of titanium in lightweight vehicles is anticipated as a replacement for steel where superior strength-to-weight ratios and corrosion resistance are required. The augmented use of titanium by the automotive industry is expected to reduce machining and manufacturing costs and permit the manufacture of more energy-efficient vehicles.

KEY EXPERIMENTAL RESULTS

Initial tests were conducted on a laboratory scale to demonstrate the feasibility of the HDH-SSF processes. The most critical processing step was to demonstrate that the OFTi powder could indeed be spray formed using the SSF system (Figure 3).

For the HDH laboratory-demonstration tests, TiH_2 (–325 mesh) powder from Micron Metals was used as feedstock. First, the published threshold temperature for the dehydride reaction was confirmed. These tests were conducted by observing the temperature at which a thin coating of TiH_2 powder sprayed onto a brass or aluminum sheet was converted first to titanium metal and then to titanium oxide. The test results yielded a threshold reaction temperature in good agreement with the 750 K temperature cited by Greenspan and Wright.[6]

Next, experiments were conducted to investigate the threshold temperature for the dehydride reaction and generate a sufficient quantity of the OFTi powder (150 g) to assess the feasibility of spray forming the material. A laboratory version of the dehydride reactor was implemented with a tube furnace. Samples (100 cm^3) of TiH_2 powder (–325 mesh) were loaded into the quartz tube and converted to OFTi metal by heating. During the heat treatment, helium gas was used to purge hydrogen from the sample and prevent oxidation. The OFTi sample was cooled to room temperature, then transferred to the sealed cabinet for further processing.

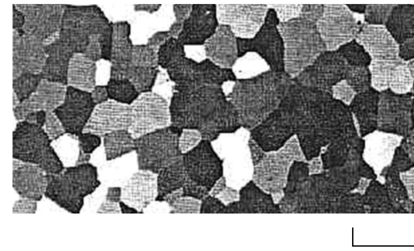

Figure 5. A polarized light micrograph of SSF titanium after HIPing.

Because the laboratory dehydride conversion process was conducted with powder in intimate contact, it was necessary to grind the agglomerated particles with a mortar and pestle and sieve through a 325 mesh. Spray-formed samples 11.5 cm × 1.5 cm × 0.3 cm (Figure 4) were deposited successfully onto an aluminum substrate using the SSF apparatus shown in Figure 3. Helium was used as the inert process gas for both entraining the OFTi powder and operating the SSF nozzle.

Future testing of the OFTi sample is planned to assess the mechanical and metallurgical properties of the SSF depositions. Visual observations of the samples indicate that residual stresses were induced and post-deposition annealing will be required. Hydrogen embrittlement[10] of the SSF material is not expected to be a problem because the hydrogen is driven off at high temperatures. Previous SSF deposition work with commercial (non-OFTi) powders[11,12] produced SSF titanium samples that were subsequently annealed at 780 K, then hot isostatically pressed at 780 K and 10.3 MPa to achieve 100% density. Figure 5 shows a polarized light micrograph of this material. Note that the equiaxed 25–75 micrometer grain size resembles a wrought, normalized structure.

FUTURE DEVELOPMENT

This work successfully demonstrated the concept of using the HDH process for producing micrometer-sized OFTi powder. Furthermore, the research has shown that the OFTi powder can be spray formed using the SSF process for producing near-net-shape components and structures. The prospects of improving the material properties of these SSF depositions are favorable through the use of powder treatments and post-deposition annealing.

The next step in the development of the HDH reactor is to design and fabri-

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cate an engineering prototype unit. A complete market analysis will be required to determine if the HDH reactor, in combination with the SSF technology, can compete with conventional methods of manufacturing near-net-shape components for the automotive and aerospace industries. The post-deposition annealing process will require considerable research also. The development of the HDH reactor in combination with the inert-gas recovery system will require significant engineering to specify a production-prototype system.

It is anticipated that approximately $1 million in funding over a period of one to two years will be required to complete the development engineering and to write product specifications for the SSF system, including the HDH reactor. After the engineering development work is completed, a production prototype system must be constructed.

Finally, to complete the commercialization process, either a production facility or agreements with contract producers will have to be established. Innovative Technology anticipates that the SSF technology will also be licensed for use in various manufacturing plants. While it is too early to estimate the cost of these development tasks, they will be considerable. The feasibility of such expenses will be evaluated on the basis of additional technical results and a more complete definition of market opportunities and barriers to entry revealed by a marketing study now in progress.

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