Proceedings of
ASME TURBOEXPO 2000
May 8-11, 2000, Munich Germany

2000-GT-533

Status of Silicon Nitride Material Properties, Component Fabrication, and Applications for Small Gas Turbines

Danielle D. Newson, John P. Pollinger, and Douglas J. Twait
AlliedSignal Ceramic Components
Torrance, California

ABSTRACT

Extensive progress has been made in the development of high performance silicon nitride structural ceramics and component fabrication. This has in turn led to a number of successful applications in small turbines, including commercial aircraft production components and a number of successful tests and continuing field tests in aircraft auxiliary power units, air turbine starters, and stationary power generation engines. The current status, capabilities, limitations, and material refinement efforts for silicon nitride at AlliedSignal Ceramic Components (ASCC) will be presented, including environmental durability and environmental barrier coating investigations. Two key issues in the implementation of silicon nitride turbine components have been the ability to fabricate engine quality hardware, and fabrication at low-enough costs to allow commercialization. The current status of production forming processes will be presented and the development of new low cost forming and advanced technologies including gelcasting and solid freeform fabrication will be discussed, both in regards to component fabrication capability and production cost potential. Finally, the status of a number of commercial and development applications such as propulsion turbine engine seals, APU hot section wheels, blades, and nozzles, industrial turbine nozzles, and power generation microturbine components will be discussed.

INTRODUCTION

The last five years have seen the maturation of silicon nitride structural ceramic materials and component fabrication processes, leading to the introduction of wear and seal components into production for gas turbine applications, and the extensive evaluation and field testing of high performance components including nozzles, blades, and turbine wheels. The material improvements principally responsible for this progress have been the development of in-situ or self-reinforced (ISR) microstructures leading to high toughness and improved reliability, and the development and refinement of high temperature compositions allowing silicon nitrides to be used at temperatures in excess of 1200°C. At the same time significant improvements in forming, densification, and machining of silicon nitride have led to the ability to fabricate complex shapes such as turbine blades and nozzles, near-net-shape thin sections such as airfoils, and large cross-section parts such as turbine wheels, while maintaining mechanical and high temperature properties and reducing costs towards that required for commercialization. Even with the extensive progress described, significant material and fabrication challenges remain. This paper will discuss the status, needs, and plans for AlliedSignal Ceramic Component's silicon nitride materials and component fabrication processes for gas turbine applications, as well as the status of production and development applications for aerospace and industrial turbines.

STATUS OF SILICON NITRIDE MATERIALS

AS800 silicon nitride was developed in the early 1990's by AlliedSignal for gas turbine applications up to 1350°C. It became commercially available in 1995. AS800 is a gas pressure sintered silicon nitride with a carefully controlled in-situ reinforced microstructure of acicular isotropically oriented beta-silicon nitride grains and a crystallized silicon oxynitride second phase [Pollinger, 1997]. The microstructure results in a fracture toughness of 8-9 $MPa\sqrt{m}$ and typical weibull modulus of 20-30. The unique characteristic of this material is that unlike earlier generation silicon nitrides with finer (~1 μm) and relatively equiaxed grain size, the relatively large grain size and interlocked acicular grains result in the grains themselves being the failure origins. With careful processing, the grains are approximately all the same size, so that very narrow strength distributions, and thus high weibull values, are obtained. This result also makes AS800 powder processing much more robust in that the typical critical flaw size is 75-150 μm, well above typical processing flaw sizes observed in fine grained silicon nitride microstructures of 5-25 μm.

Early generations of AS800 did exhibit some occurrence of a grain clustering/low density region, which although not decreasing the average strength of the material, did result in lower weibull modulus values. Subsequent powder processing refinements eliminated this flaw type and a recent DOE/ORNL sponsored program evaluated the current reliability and reproducibility of AS800. Sixteen lots of AS800 were slipcast and sintered in separate furnace runs. Buttonhead tensile specimens were then machined and tested from each lot at the University of Dayton Research Institute (UDRI). The results are shown in Figure 1. Sixteen different lots and a total of 78 specimens showed a weibull modulus of 32.1 and a characteristic tensile strength of 740 MPa.

Recently, a number of advanced gas turbine applications have identified the need for a 1400°C capable material for application in hot section components. AlliedSignal has further refined the AS800 second phase composition to increase its temperature capability and oxidation resistance to achieve the 1400°C goal. AS950EXP is an experimental material now being refined and characterized. The development of AS950EXP focused on improving creep resistance at 1400°C to achieve creep rates no greater than 10^{-10} sec^{-1} at desired continuous stress loads of 70-100 MPa (typical of hot section 1st and 2nd stage turbine blades operating in advanced propulsion and small power generation gas turbines). The creep rate for AS950EXP at 1400°C has achieved the desired creep/stress target range and is approximately one order of magnitude improved over AS800 at 1400°C (Figure 2). Corresponding tensile stress rupture data (Figure 3) indicates that suitable lifetimes can be achieved for stress levels of < 150 MPa.

Over the past two years, two significant challenges have become apparent for silicon nitride applications in gas turbine hot sections. One is the volatilization of the protective silica oxidation layer on silicon nitride at high pressure, high water vapor content, high velocity, high temperature conditions, and the other is the issue of impact sensitivity due to foreign or domestic object damage (FOD/DOD).

As progress has been made in the development and maturation of monolithic and composite non-oxide ceramic materials and

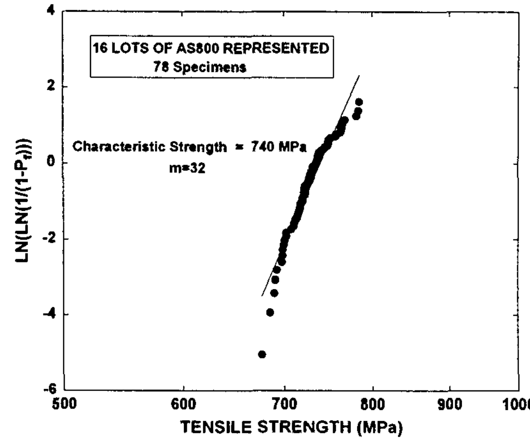

Figure 1. Reproducibility of AS800 Silicon Nitride Tensile Strength.

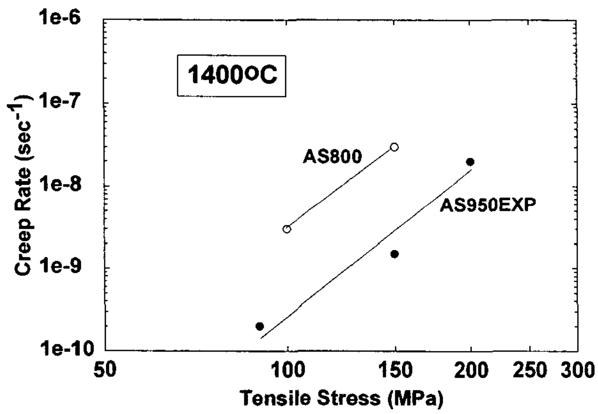

Figure 2. Comparison of AS800 and AS950EXP Tensile Creep Rate at 1400°C.

component fabrication processes, a series of investigations [Opila and Hann, 1997; Ferber et. al. 1999] have explored the durability of these materials in test environments simulating the combustion environment of medium and larger gas turbines. Previously, most environmental testing of these materials has been in either static air or simulated combustion environments at atmospheric pressure and at relatively low velocities. Under these conditions, silicon nitride materials generate a self-protecting passivating layer of silica, with higher temperatures accelerating the oxidation process and oxidized layer thickness. For a number of lower temperature (< 1150°C) applications in relatively low pressure and low velocity environments and cyclic applications such as aircraft APU's and small automotive propulsion and turbogenerator

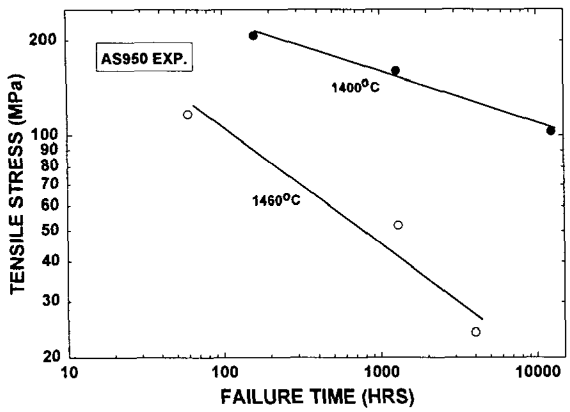

Figure 3. Tensile Stress-Rupture Properties of AS950EXP Silicon Nitride.

engines, these tests adequately represented the material performance. Medium (> 1 MW) and larger gas turbines continuously operate at much higher pressures and gas flow velocities, with significant amounts of water vapor, and at significantly higher temperatures (1200°C and higher in many cases). Evaluations of silicon based non-oxide materials in test rigs and in actual engine tests under high pressure, high velocity, high water vapor, and high temperature conditions have demonstrated that the protective silica layer is volatilized, resulting in recession of material from the surface. This mechanism can result in aggressive surface recession and is a significant issue limiting application of silicon based non-oxide structural ceramics in gas turbines operating under these conditions. As an example, Rolls Royce Allison has just completed an 800 hour field test of their 501 industrial gas turbine engine, retrofit with AS800 silicon nitride 1st stage nozzles. The 501 engine nozzles are shown in Figure 4. Although the nozzles performed well with no performance

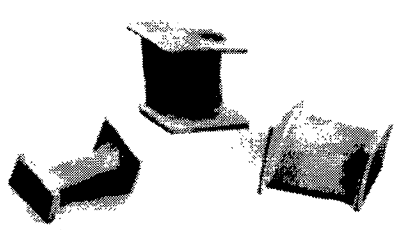

Figure 4. AS800 Silicon Nitride 1st Stage Nozzles for Rolls Royce Allison 501 Industrial Gas Turbine Engine.

issues during the test, inspections during and at the end of the 800 hours of testing showed that the nozzles were experiencing significant surface recession, up to 30-40% of trailing edge thickness loss after 800 hours. Preliminary analyses of exposed nozzles underway at ORNL show that the recession is very much a surface effect, with mechanical properties and microstructure of the remaining bulk material in the airfoil sections equivalent to starting properties [Lin and Ferber, 1999]. The amount of surface recession is much greater than allowable for desired component lifetimes (>10,000 hours) and thus a solution to the volatilization process is required.

A number of organizations are investigating possible solutions to silica volatilization of monolithic silicon nitride and silicon carbide based continuous fiber composites, including AlliedSignal. The primary path being pursued is to place a protective environmental barrier coating (EBC) on the ceramic to prevent oxygen and water vapor reaction with the silicon in the ceramic. To be effective and durable, the coatings must: a) be an oxygen/water vapor barrier, b) closely match the thermal expansion of the ceramic, c) have relatively low modulus to prevent spalling during cyclic loading, d) be oxidation resistant, e) non-silicon containing or self-healing to minimize volatilization, and f) be non-reactive with the ceramic base material at desired operating temperatures. The coatings must be able to be applied effectively to bond to the ceramic and are obviously desired to be low cost. Mullite (Lee, 1999) and a proprietary coating developed under the NASA Enabling Propulsion Materials (EPM) program are currently being evaluated on both composite and silicon nitride monolithic materials and show some promise. AlliedSignal is developing an oxide coating system that does not contain silicon, and which is currently being evaluated at ORNL in their high-pressure high-water-vapor test rig on AS800 silicon nitride test specimens.

A number of applications of silicon nitride components have seen minimal or no FOD/DOD issues due to specific component robustness, design for impact resistance, cleanliness of engine, small size, and low rotational speeds. Larger gas turbines have less flexibility and catastrophic damage to ceramic parts has occurred, an example being the CSGT program Solar Centaur 50 engine with ceramic blades, where DOD events have resulted in catastrophic failure of the entire set of silicon nitride 1st stage blades [Price et. al., 1999]. Continuous fiber composite ceramics (e.g. SiC fiber reinforced SiC) have shown significant damage tolerance capability in preliminary impact testing [Parthasarathy, 1999] but monolithic silicon nitride is required for the blades due to the high stresses and temperatures of this application. Besides design for impact resistance and the need for engines with very minimal FOD/DOD potential, the damage tolerance of monolithic silicon nitride must be improved. ASCC is exploring a number refinements to ISR silicon nitride to improve impact resistance and has provided a number of specimens to Solar Turbines to support their impact testing investigations under the CSGT program. Impact resistance improvement from the material standpoint can be addressed by strength improvements and fracture toughness improvements. ASCC has determined that there is no obvious way to significantly improve material strength without compromising ISR properties in silicon nitride materials, but is exploring/considering a number of routes to improve toughness including hierarchical reinforcement structures such as fibrous monolith structures using ISR materials, continuous fiber composite/monolithic hybrids, and compressive coatings.

STATUS OF COMPONENT FABRICATION PROCESSES AND APPLICATIONS

ASCC fabricates structural silicon nitride materials such as AS800 and GS-44 by liquid phase sintering of powder compacts. The powder compacts consist of ~90-95% silicon nitride powder with the remainder being the oxide sintering additive powders that allow formation of the second phase during densification. These powders are required to be submicron in size to provide reactivity and subsequent ISR microstructure development. Submicron powders are difficult to process and require very precise and robust processes to generate powder compacts for structural ceramic applications without processing defects such as voids, inclusions, and agglomerates. The additional constraint in making structural ceramic turbomachinery components is the very tight dimensional tolerances typically required for complex shape components such as turbine nozzles, blades, and combustors. ASCC has developed and implemented bisque and final machining of simple shaped stock to address the fabrication of simple geometry and low volume/prototype complex geometry parts. Gelcasting and Solid Freeform techniques (SFF) are being developed and demonstrated as near-net-shape forming approaches for complex shaped components, directed at cost reduction and production scale-up to meet aerospace and industrial turbomachinery needs.

Bisque and final machining of simple shaped stock is the process where a ceramic powder compact of simple geometry is partially heat treated to provide enough strength for machining by typical turning, milling, and rapid grinding techniques, but not densified enough to require diamond grinding, as is needed for fully densified material. The bisque firing process results in an increase of only ~0.5-1.0% density, allowing carbide and other relatively low cost cutting tools to be used. ASCC has used this process to fabricate volumes of simple shaped silicon nitride production wear components by isopressing simple shaped stock for turbomachinery applications - such as the GS-44 silicon nitride seal runners for the AlliedSignal Engines family of 731 engines (Figure 5).

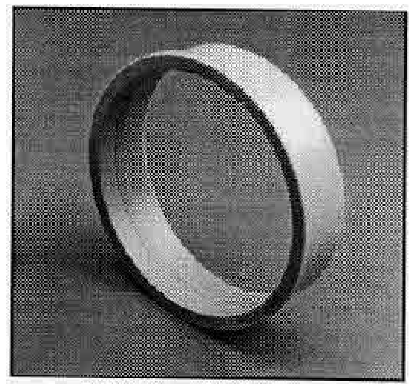

Figure 5. Seal Runner for AlliedSignal Engines 731 Turbofan Propulsion Engine (GS-44 Silicon Nitride).

The bisque machining process is most appropriate for these parts since the relatively low volume (< 1000 month) does not justify investment in near-net-shape forming tooling, and because all part surfaces must be machined to meet tight tolerances (many at +/- 0.005 mm). Final machining is relatively simple because of the seal's symmetrical shape. ASCC has also used bisque machining of simple shaped slipcast stock to fabricate development quantities of complex shaped components such as turbine blades and nozzles, where the immaturity of net-shape forming processes to form these shapes to tolerance and quality, and the cost of near-net shape molding tooling, has prevented their use. The bisque machining process has proven very effective for fabrication of engine quality blades and nozzles, where the airfoils are bisque machined to net-shape and only tight tolerance attachment features such as dovetails, platform edges, and features such as blade tips are final machined. Examples include the Solar Turbines Centaur 50 1st stage turbine blade of AS800 silicon nitride (Figure 6) fabricated for Solar under the DOE CSGT program.

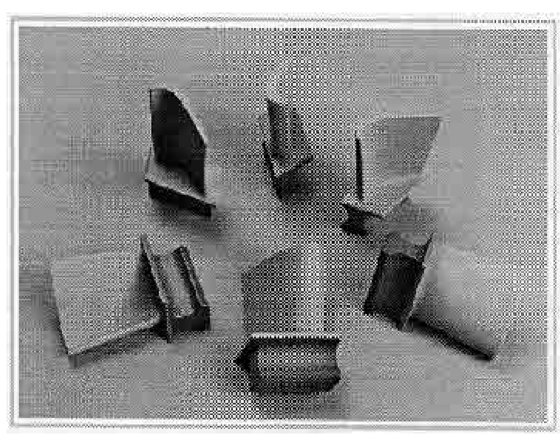

Figure 6. 1st Stage Turbine Blades for Solar Centaur 50 CSGT Industrial Turbine Engine (AS800 Silicon Nitride).

The AS800 blades ran more than 950 hours successfully in field test. An example of a very thin airfoil formed by bisque machining is the Rolls Royce Allison 501 industrial turbine engine 1st stage nozzle of AS800 (Figure 4), fabricated for Rolls Royce under the DOE ATS program. The nozzles performed successfully for 800 hours in field test in 1999, but significant surface recession was observed, as described previously.

ASCC also fabricated 331-200 aircraft auxiliary power unit (APU) blades and nozzles (Figure 7) for AlliedSignal Engines (ASE) by bisque machining simple shaped AS800 silicon nitride stock. To-date, the nozzles have performed very effectively in 331-200 test engines with over 2500 hours on a single set and over 5100 hours on multiple sets. The blades were successfully tested to over 1080 hours with the development of effective dovetail and compliant layer technology by ASE. Because of the cost (ceramic and metallic hardware) and complexity of mounting 23 individual ceramic 331-200 blades (Figure 8), AlliedSignal Engines designed a monolithic ceramic turbine wheel for evaluation. ASCC has fabricated a number of these wheels by bisque machining of simple shaped disks of AS800 silicon nitride (Figure 9). The blisks were tested in a 331-200 test engine and performed effectively again - over 750 hours as the only ceramic component, and over 300 hours with the AS800 nozzles in combination.

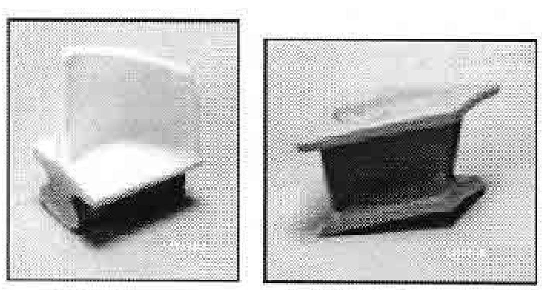

Figure 7. AlliedSignal Engines 331-200 APU Turbine Blade and Nozzle (AS800 Silicon Nitride).

To further evaluate the performance of the AS800 331-200 wheel, ASE is now planning a 10,000 hour field test with the industrial version of the 331-200 APU, in an application where volatile organic compounds are burned off [Schenk, 1999].

Although the bisque machined complex shaped components have performed well as engine quality hardware, this fabrication process for complex shaped components is capable of relatively low volumes and is high cost due to extensive bisque machining time using CNC machining. Thus, there still exists a strong need to have viable near-net-shape forming processes to address production volume and cost target

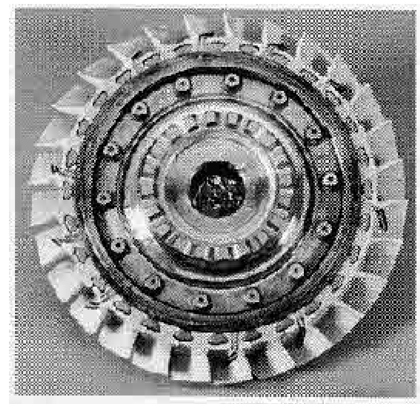

Figure 8. AlliedSignal Engines 331-200 APU 1st Stage Turbine Wheel with Inserted AS800 Silicon Nitride Blades.

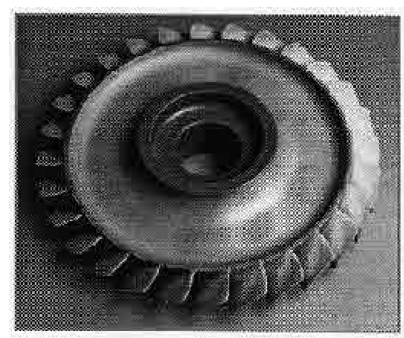

Figure 9. AlliedSignal Engines 331-200 APU 1st Stage Turbine Blisk of AS800 Silicon Nitride.

capabilities. Additionally, there is a desire for further component complexities (e.g. cooling passages, multiple materials/hybrid structures), and the fact that the performance of larger volume ISR components is leading to consideration of even larger components - where the benchmark isopressing and slipcast forming processes are not viable for these size parts.

ASCC has been developing the gelcasting technology [Omatete et. al., 1991] invented by ORNL scientists, in conjunction with ORNL, DOE, the NIST Advanced Technology Program, and DARPA/ONR, to near-net-shape form silicon nitride structural components and scale the process up to manufacturing viability. Gelcast AS800 nozzles (Figure 10) have been certified for the 85 series APU after successfully passing the 150 hour FAA certification test at ASE. ASCC has also developed near-net-shape prototype cooled vanes of AS800 for United Technology Research Center's (UTRC) FT-8 industrial gas turbine engine under DOE funding support. The parts, shown in Figure 11 (as-cast and after densification), do not include the full design small trailing edge cooling passages, which require further

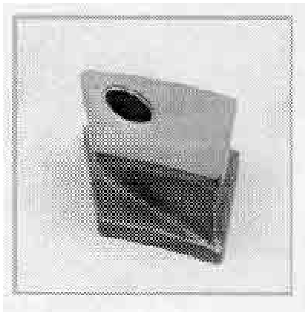

Figure 10. AlliedSignal Engines Series 85 APU Turbine Nozzle (Gelcast AS800 Silicon Nitride).

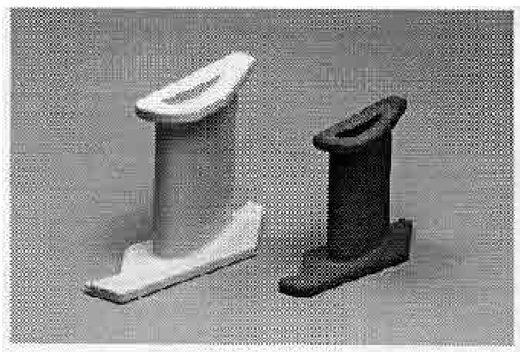

Figure 11. UTRC FT-8 Industrial Turbine Engine Nozzle Prototypes (Gelcast AS800 Silicon Nitride) As-Cast and Fully Dense.

gelcasting and tooling development, planned for future investigation under UTRC/DOE/DARPA support. ASCC has also developed and demonstrated gelcasting of near-net-shape GS-44 silicon nitride impulse turbine wheels for commercial and military aircraft propulsion air turbine starters for AlliedSignal Engines and Systems under NASA and DARPA funding support. The silicon nitride starter wheels (including active metal braze metal shaft attachments), shown in Figure 12, allow reduced weight containment structures, and improved erosion and corrosion durability for cartridge starter applications. The gelcast wheels are currently in testing in starter units and have successfully completed over 1700 of 3000 planned air start/stop cycles to-date. Cartridge starts were also successfully performed at –65°F, 72°F, and 120°F to simulate emergency start conditions in the field with no damage to the silicon nitride wheels or braze joint attachment.

DARPA and DOE have supported ASCC in developing automation of the gelcasting process to fabricate complex shaped components such as turbine wheels to meet expected cost targets and required production volumes for potential applications such as missile turbojet engines and microturbines for power generation. The result is the

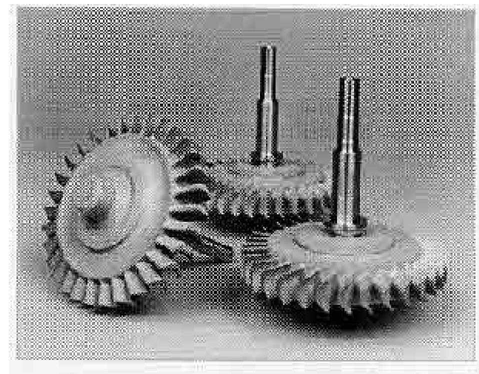

Figure 12. AlliedSignal Turbine Starter Wheels (Gelcast GS-44 Silicon Nitride).

development of a first generation automated gelcasting system (shown in Figure 13) designed to fabricate 10,000 silicon nitride near-net-shape turbine wheels per year. The Teledyne

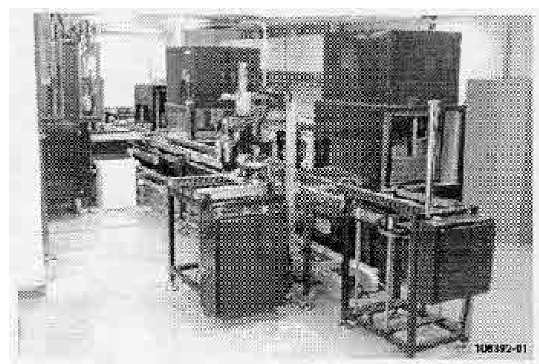

Figure 13. Automated Gelcasting System for Silicon Nitride Turbine Wheels.

M304 turbojet wheel (shown in Figure 14) is the demonstration component. To-date, the automatic gelcasting system has shown capability of fabricating 4 parts per hour, meeting the 10,000/yr production rate. Wheels are now being fabricated for performance evaluation.

The gelcasting process has demonstrated the ability to effectively form large silicon nitride powder compacts in addition to near-net-shapes, leading to a recent evaluation of the ability to fabricate larger components, such as the ~190 mm dia. 331-200 turbine wheel previously formed from slipcast blanks, by gelcasting in conjunction with ASE and DOE. Gelcast AS800 331-200 spin disks were fabricated (Figure 15) and spun-to-burst, demonstrating equivalent performance to slipcast bisque machined spin disks and wheels.

These results have led to investigations to evaluate the ability to fabricate even larger components, principally axial turbine

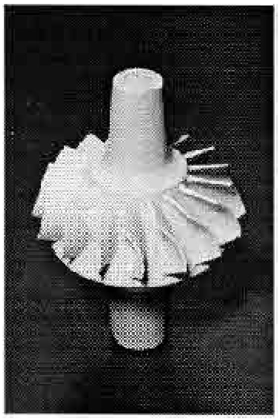

Figure 14. Teledyne M304 Turbine Wheel (Gelcast AS800 Silicon Nitride).

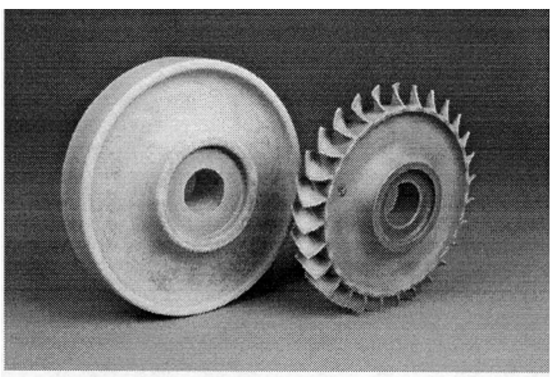

Figure 15. Comparison of Net-Shape Gelcast 331-200 Spin Disk and Bisque Machined Slipcast Blisk of AS800 Silicon Nitride.

wheels and large static structures of ISR silicon nitride. An example is shown in Figure 16, where a 420 mm dia. spin disk of AS800 silicon nitride was successfully gelcast and is shown in comparison to 331-200 wheels and individual blades. The spin disk is planned for future performance evaluation to determine the scalability of the gelcasting process and to verify/refine probabilistic lifing models.

ASCC is also evaluating solid freeform fabrication (SFF) techniques to address the needs to: 1) rapidly make functional near-net-shape prototype parts without expensive tooling or machining, 2) fabricate even more complex structures such as complex cooling passages in small turbine blades, and 3) build in multiple materials and hybrid microstructures in a single component. CC is working with SRI and Rolls-Royce Allison to evaluate SRI's Direct Photo Shaping (DPS) SFF technology for the 501 engine 1st stage nozzle, funded by DARPA. The DPS process combines photopolymerizable preceramic polymer with ceramic powder into a slurry, which is tapecast

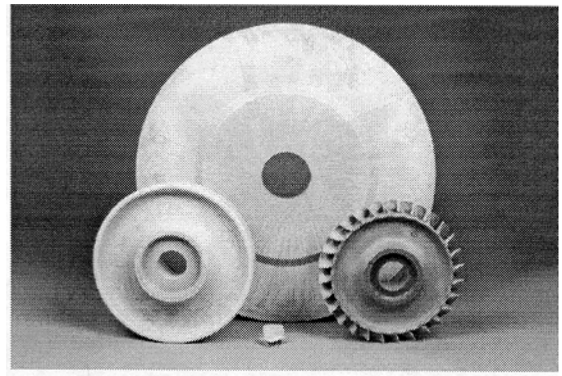

Figure 16. Comparison of 331-200 APU Turbine Blade, Blisk, Gelcast Spin Disk, and Large (420 mm Dia.) Prototype Gelcast Spin Disk (AS800 Silicon Nitride).

into layers and polymerized by projecting light in desired patterns onto each layer in sequence. An example AS800 nozzle made by DPS is shown in Figure 17. The AS800 made by DPS has been demonstrated to have equivalent mechanical

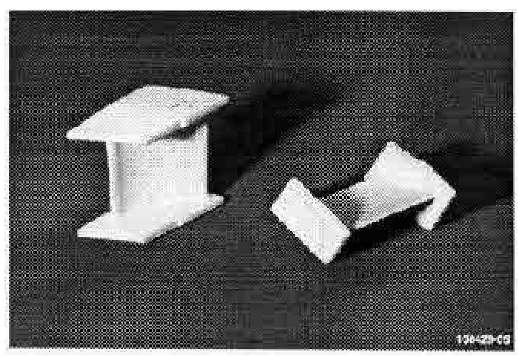

Figure 17. Rolls Royce Allison 501 Engine Nozzles Formed by SRI Using DPS Solid Freeform Fabrication (AS800 Silicon Nitride).

mechanical properties to benchmark slipcast AS800. The DPS nozzles exhibit very good dimensional control, and process refinements are now addressing the different scaling factors unique to DPS to generate parts meeting nozzle dimensional requirements. ASCC is also working with AlliedSignal Corporate Research and Rutgers University under DARPA funding to develop Fused Deposition of Ceramics (FDC) to fabricate silicon nitride turbine blades for ASE advanced propulsion engines that contain complex cooling passages needed for ultra high operating temperatures (> 1400°C). Additionally the FDC process is being developed to generate hybrid microstructures/materials in single components. Examples include placing different modulus materials (e.g. silicon nitride and silicon carbide) strategically within the component to reduce stresses due to thermal gradients or mechanical loads. Also, fibrous monolith structures are being developed for fabrication by FDC to potentially generate hybrid properties between monolithic and continuous fiber composite ceramics such as improved damage tolerance with high strength retention.

SUMMARY

A number of advances have been made in silicon nitride materials and fabrication processes for turbomachinery components, leading to production of wear components and successful field testing of hot section components. The silicon nitride ISR microstructure benefit of increased flaw tolerance is leading to the ability to achieve large components with acceptable reliability and silicon nitride material temperature capabilities have been increased to 1400°C through second phase refinements. But impact resistance and environmental durability are key technical hurdles that must be overcome to allow for use in many applications. Bisque machining processes have matured and demonstrate the ability to make engine quality components, but at low quantities and at relatively high price for complex shaped components. Gelcasting has been demonstrated to result in engine quality complex shaped near-net-shaped components, and has shown initial capability as a high volume fabrication process but further maturation is needed. Finally, SFF fabrication techniques show promise for fabrication of small volumes of near-net and complex-shaped components, and are also currently being developed to achieve very complex features and hybrid material improvements.

ACKNOWLEDGMENTS

Much of the work presented in this paper was performed under a number of programs supported in part by the Department of Energy, Department of Commerce, Defense Advanced Research Projects Agency, NASA, and under subcontract to AlliedSignal Engines and Systems, Rolls Royce Allison, Solar Turbines, UTRC, and SRI.

REFERENCES

Ferber, M.K., Lin, H.T., Parthasarathy, V., and Brentnall, W., "Degradation of Silicon Nitrides in High Pressure, Moisture Rich Environments," Am. Soc. Mech. Eng., Turbo Expo 99, Paper 99-GT-265, 1999.

Lin, H.T., and Ferber, M.K., Personal Communication.

Lee, K.N., "Key Durability Issues with Mullite-Based Environmental Barrier Coatings for Si-Based Ceramics," Am. Soc. Mech. Eng., Turbo Expo 99, Paper 99-GT-443, 1999.

Omatete, O.O., Janney, M.A., and Strehlow, R.A., "Gelcasting – A New Ceramic Forming process," Am. Ceram. Soc. Bull., **70** [10] 1641-1649 (1991).

Opila, E.J., and Hann Jr., H.E., "Paralinear Oxidation of SiC in Water Vapor," J. Am. Ceram. Soc., **80** 197-205 (1997).

Parthasarathy, V., Personal Communication.

Pollinger, J.P., "Status of Silicon Nitride Component Fabrication Processes, Material Properties, and Applications," Am. Soc. Mech. Eng., Turbo Expo 97, Paper 97-GT-321, 1997.

Price, J.R., Jimenez, O., Parthasarathy, V., and Miriyala, N., " Ceramic Stationary Gas Turbine Development Program – Sixth Annual Summary," Am. Soc. Mech. Eng., Turbo Expo 99, Paper 99-GT-351, 1999.

Schenk, B., "Ceramic Gas Turbine Technology Development," Am. Soc. Mech. Eng., Turbo Expo 99, Paper 99-GT-315, 1999.