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NT164 SILICON NITRIDE GAS-TURBINE ENGINE TURBINE BLADE MANUFACTURING DEVELOPMENT

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ABSTRACT

Norton Advanced Ceramics (NAC) has performed ceramic turbine blade fabrication development as part of several DOD and DOE-sponsored programs including: (1) The Experimental Turbine Engine Concept (ETEC); (2) The Advanced Turbine Technology Applications Project (ATTAP); (3) The Ceramic Turbine Engine Development Project (CTEDP); and (4) The Ceramic Stationary Gas Turbine (CSGT). NAC has developed two HIPed silicon nitide materials for fabricating turbine blades within these programs -One is designated NT154; and the second is designated NT164. Under the ETEC program with AlliedSignal Engines, NT154 blades were fabricated and delivered for proof and engine testing. Blade fabrication development efforts were augmented by NAC's work under the ATTAP, which was directed at developing manufacturing technologies for rotors, stators, scrolls, vanes, and other components. Under the ATTAP, complex-shape forming was emphasized utilizing pressure slip-casting. NAC has employed pressure slip casting developed under the ATTAP to fabricate ceramic turbine blades and other gas-turbine components for various advanced heat-engine efforts. NT154 nozzles have been delivered to AlliedSignal Engines under internally sponsored and DOD-sponsored programs. NT154 diffusers, nozzles, and monorotors have been delivered to Sundstrand Power Systems. Under the CTEDP and CSGT programs, continued efforts on turbine blade fabrication

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development are anticipated for 1995 and beyond. Work under the CTEDP program with AlliedSignal Engines is focused on cost reduction through process simplification and scale-up. Under the CSGT program, NAC is participating with Solar Turbines Incorporated to deliver prototype quantities of NT164 silicon nitride blades using a controlled fabrication process. NAC is utilizing its prior experience in fabricating similar blade geometries under the ETEC, ATTAP, and CTEDP programs in the CSGT effort.

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NOMENCLATURE

ATTAP - Advanced Turbine Technology Applications Project.

APU - Auxiliary Power Unit

CMM - Coordinate Measuring Machine.

CTEDP - Ceramic Turbine Engine Development Project.

CSGT - Ceramic Stationary Gas Turbine.

ETEC - Experimental Turbine Engine Concept.

HIP - Hot-Isostatic Pressing.

NT154 and NT164 Si₃N₄ - HIPed Silicon Nitride.

NAC - Norton Advanced Ceramics.

ORNL - Oak Ridge National Laboratory.

PSC - Pressure Slip-Casting.

SPC - Statistical Process Control.

UDRI - University of Dayton Research Institute.

INTRODUCTION

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Efficient gas-turbine engines require the incorporation of high-reliability advanced ceramic components. To achieve this goal, the Department of Energy (DOE) has initiated several major ceramic development and characterization programs. Norton Advanced Ceramics (NAC) is a participant in two of these programs that include work on turbine blades: (1) The Ceramic Turbine Engine Development Project (CTEDP); and (2) The Ceramic Stationary Gas Turbine (CSGT). The CTEDP is administered by NASA-Lewis Research Center with AlliedSignal Engines as the prime contractor. The CTEDP is a demonstration program which utilizes AlliedSignal's 331-200 APU gas-turbine engine as a functional test-bed. The goals of this program include: (1) Demonstration of the reliability and durability of ceramic components in modified, available gas-turbine engines; and (2) Scaleup and improvement of manufacturing processes for ceramic turbine engine components. NAC is a subcontractor to AlliedSignal Engines for developing and demonstrating a cost-effective, controlled production process for NT164 331-200 blades. In addition, under parts-buy efforts separate from the CTEDP, NAC has fabricated and delivered prototype quantities of NT154 silicon nitride 331-200 blades. The CSGT is administered by DOE with Solar Turbines Incorporated as the prime contractor. NAC is a subcontractor to Solar Turbines for fabrication and delivery of prototype quantities of CSGT first generation design NT164 blades.

SILICON NITRIDE MATERIALS AND PROCESS DEVELOPMENT

The two silicon nitride materials utilized in turbine blade fabrication development efforts, NT154 and NT164, are both 4 percent Y_2O_3 -doped compositions densified by hot isostatic pressing (HIP). HIPing is accomplished using the glass encapsulation process. Characterization of these materials has been conducted by NAC, AlliedSignal Engines, Allison Engine Company, Solar Turbines Incorporated, and by a number of additional laboratories [1-9]. Updated typical physical, thermal and mechanical properties are given in Table 1.

NT154 possesses excellent flexural fast-fracture behavior up to 1370°C, accompanied by an acceptable Weibull Modulus. Failure origins have been dominantly associated with surface related machining flaws or impurities. Reported tensile strengths under fast-loading conditions parallel flexural tests.[4] Principal failure origins in tension were volume inclusions generally identified as iron impurities. Under slow-loading conditions at elevated temperatures, creep and slow crack growth behavior have been characterized.[1,5-6,9] NT154 exhibits creep through a cavity nucleation and growth mechanism. Failure occurs via cavity link-up. In comparison with other advanced materials, NT154 exhibits excellent elevated temperature durability.

NAC utilized the experience in processing NT154 to make several alternations within the material itself in an effort to improve elevated temperature properties. Recent research focused on slight changes of the glass phase composition, along with adjustments to selected heat-treatment schedules. These modifications resulted in an improved version of NT154, which NAC has designated NT164. For bulkground, fully machined surfaces, NT164 has room temperature properties equivalent to NT154. However, significant improvements are noted in hightemperature strength and creep resistance.[6,9] Typical NT164 properties are presented in Table 1 as well.

In addition to an improved creep rupture life, NT164 exhibits higher as-processed surface strength at room and elevated temperatures. Figure 1 shows a plot of as-processed surface flexural strength for both materials as a function of temperature. NT164 has $\approx 10\%$ higher strength than NT154 at room temperature, and $\approx 30\%$ higher strength at 1316°C.[7-8] The underlying mechanism for the asprocessed surface strength improvement is not well understood at this time. However, one postulate is that the slight changes in the NT164 glass phase composition reduces the severity of reaction with the HIP encapsulant glass. SEM photographs of KOHetched fracture origins of typical NT154 and NT164 as-processed surface specimens, shown in Figures 2 and 3 respectively, bear this out. The NT154 fracture origin is a "worm-hole" type flaw occurring at the site of a pre-existing surface defect. Preexisting surface flaws, thought to originate in the pressure slip casting operation at or just below the mold-part interface, are exploited through reaction with the HIP encapsulant glass at high temperatures and pressures. NT164 is believed to have the same population of surface casting defects as NT154, however, upon HIPing, these flaws do not extend as deeply into the bulk material. A typical as-processed surface fracture origin for NT164 is shown in Figure 3.

NT154 COMPONENT DEVELOPMENT

An overall process flow-chart for NT164 component fabrication is shown in Figure 4. This process is the same as for NT154, except for the additional powder heat treatment step. The processes for NT154 and NT164 were developed and optimized within prior years under the ATTAP. NAC employed Taguchi and other fractional factorial design experiments to optimize these processes.[10-11] During 1990-91, all process optimization work was completed for Emphasis was then placed on NT154 NT154. component forming technology development and optimization of heat-treatment cycles for NT164. NAC selected pressure slip-casting (PSC) as its preferred forming technique for NT154 and NT164. To produce components in a crack-free state, NAC has developed several casting modifications. These techniques were developed in prior years and described in previous reports.[10-11] Using these processes, NAC has utilized PSC developed under the ATTAP to fabricate ceramic turbine blades, nozzles, and other gas-turbine components for various advanced heat-engine efforts including:

- NT154 turbine blades for AlliedSignal Engines under the ETEC program.
- AGT101 rotors and stators for AlliedSignal under the ATTAP.
- NT154 and NT164 ATTAP AGT-5 rotors for Allison.
- NT154 nozzles delivered to AlliedSignal Engines under internally sponsored and DOD-sponsored programs.
- NT154 diffusers, nozzles, and monorotors delivered to Sundstrand Power Systems.
- NT164 attachment and other specimens under the CSGT.
- NT164 combustor liner tile and panels.

NAC is continuing component development under the CTEDP with AlliedSignal. NAC's work under the CTEDP is focused on developing a production process for NT164 silicon nitride 331-200 turbine blades. The objective of this effort is to scale-up the current blade prototype process to achieve quality levels,

delivery times, and part costs required for aerospace volume production. NAC's overall approach to improving and scaling-up the existing blade fabrication process is based on the following key elements:

Technology - NAC believes achievement of a cost effective process is paced by the implementation of available technology. Within this program, NAC is utilizing conventional processing techniques including pressure slip casting, hot isostatic pressing, and diamond grinding. Work is focusing on simplification, optimization, and automation of each unit operation to improve production rates and process yields.

Learning - NAC believes that learning will play a significant role in defining the cost and quality of ceramic engine components over time. Learning translates into cost reduction through process simplification, enhanced throughput, and improved yields.

NAC's CTEDP effort includes work on the following tasks: (1) Statistical Process Control; (2) Powder Processing; (3) Slip Preparation; (4) Mold Fabrication and Casting; and (5) Machining and Inspection.

Statistical Process Control - Based on a data base of all NT164 process inputs and outputs, NAC has proposed to adopt a set of key process and product characteristics for blade production. A list of these key characteristics for SPC is shown in Table 2.

Powder Processing - NAC has completed an experiment to optimize the parameters for the continuous heat treatment of raw materials. The experiment encompassed two factors (heat treatment temperature and hot zone soak time) at three levels each. Optimal conditions were selected based on powder chemical analysis. In a follow-up demonstration, a larger quantity of powder raw material was processed through the continuous heat treatment operation at the selected conditions. This raw material was utilized in a powder batch scale-up demonstration. The NT164 powder batch size was scaled-up by a factor of 2.2X. The optimum milling time to reach the nominal surface area and particle size distribution was determined by measuring powder characteristics in real-time. The specifications for all powder characteristics of the scaled-up powder processing operation were kept at the same values as for the smaller prototype process

batch size.

Slip Preparation - NAC has completed a scale-up of the NT164 slip batch size by 3X. The optimum mixing time to reach a stable slip viscosity was determined by measuring slip viscosity in real-time. Slip from both of the 3X slip batch scale-up demonstration runs is being used to cast 331-200 blades. Processing of these demonstration components is in progress.

Mold Fabrication and Casting - NAC is transforming its current process to a production level through the implementation of durable porous and non-porous plastic mold sections, and the utilization of high pressure, semi-automated pressure casting. NAC's approach for development of a production process for blades is based on NAC's experience in pressure slip casting NT154 blades for previous parts-buy contracts using all plaster molds. NT154 blades pressure slip cast using all plaster molds exhibited: (1) A high occurrence of surface pits, (2) Relatively low as-processed surface strength; and (3) Dimensional distortion. This distortion closes the airfoil toward the pressure side as shown in Figure 5. A mechanism for the dimensional deviations is differential shrinkage during HIP densification due to a low green density layer. One can predict the existence of this layer by first principles of pressure filtration through a porous medium, however, its magnitude in actual components is not well known. An example of a green density gradient in an NT154 AGT101 rotor section is shown in Figure 6. The data reveals a low green density layer that extends approximately 6 mm into the casting from the plasterpart interface.

An initial screening experiment was conducted to demonstrate a single directional casting approach for 331-200 blades. This approach incorporates a mold material on the blade flowpath features that does not draw moisture. A layout of the mold design for single directional casting 331-200 blades is shown in Figure 7. The single directional casting screening experiment encompassed the following variables: (1) Powder formulation at two levels (NT154 vs. NT164), (2) Slip solids content at two levels; and (3) Mold design (plaster clamshell configuration vs. single directional). A total of ten 331-200 blades were cast at each condition in a full factorial array. All blades were processed through HIP densification and inspected in accordance with AlliedSignal Engines surface quality requirements. Key results are summarized as follows:

- Single directional blade castings exhibited small areas of slip starvation at the middle of the platform edges (pressure and suction sides).
- The size and frequency of pits was not affected by slip solids content.
- The frequency of pits was not affected by material type or mold material adjacent to the flowpath surface. The pit size distribution was skewed toward smaller diameter pits in comparing the single directional cast NT164 blades castings to NT154 blades cast in plaster molds. As shown in Figure 8, the percentage of pits ≤ 0.005" was ≈73% for single directional cast NT164 blades and ≈23% for NT154 blades cast in plaster molds.

A subsequent experiment to optimize single directional casting of NT164 blades is in progress. Specific details of this optimization experiment are as follows:

- The dovetail feature stock geometry was changed to eliminate the areas of slip starvation at the middle of the platform edges.
- Purpose To increase casting rate and yield, dense casting yield, and improve net-shape dimensional control.
- Key Process Inputs A total of five input variables are being evaluated at two levels each. The input variables are slip solids content, mold material adjacent to blade flowpath features, drawing surface mold material, casting pressure, and HIP encapsulant treatment.
- Key Process Outputs Slip viscosity, green density, casting time, type, location, and size of surface defects on as-HIP'ed surfaces, net-shape dimensional control.
- Expected Results Identification of treatments that concurrently improve casting rate and yield, reduce relevant surface defects, and improve net-shape dimensional control.

Machining and Inspection - Currently, NAC works with outside machining vendors to grind the blade dovetail in prototype quantities. Quality, process cycle time, and cost have been difficult to control. Under the CTEDP, NAC is developing a production machining process for 331-200 blades. NAC has determined how best to hold the blade during each step of the grinding process, including the initial set up of the blade casting. The process will allow for a best fit of the actual casting geometry in the initial grinding fixture. This will replace procedures used in the past which located the blade casting against hard datum target points. This locating method did not take into consideration deviation of the actual casting to the nominal airfoil design geometry. A shop print of the 331-200 blade dense casting is being generated that will identify alternate cast datum targets and method of adjustment. Based on the selected method of holding the blade for each unit operation, NAC will design and fabricate the appropriate grinding fixtures. Initially, only a single element of an envisioned gang fixture will be fabricated.

Component processing technology developed under the CTEDP is being utilized in the fabrication of CSGT program first generation blades. Under the CSGT program, NAC is participating with Solar Turbines Incorporated to deliver prototype quantities of NT164 silicon nitride first generation design blades using a NAC is utilizing controlled fabrication process. NT164 for this effort for its improved as-processed surface strength and superior elevated temperature durability. These first generation NT164 blades have been pressure slip cast using the single directional casting mold configuration to minimize the occurrence of pits on and other surface defects on the blade flowpath features. To minimize dimensional deviations of blade flowpath features with respect to machined datums, the dovetail grinding process allows for a best fit of the actual casting geometry in the initial grinding fixture. A CMM dimensional inspection plot of the tip section of a first generation blade, shown in Figure 9, shows an oversize condition but no evidence of closed airfoil distortion.

HISTORICAL PERSPECTIVE - CERAMIC BLADE MANUFACTURING

NAC has participated with various engine builders in developing materials and fabrication processes for silicon nitide turbine blades. A summary of the history of silicon nitride turbine blade development is provided in Table 3. In the time period 1976-1981, Norton Company participated with the Garrett Turbine Engine Company (Now AlliedSignal Engines) to produce NC132 silicon nitride turbine blades.[12] These components were fully machined from hotpressed billets by profile grinding. Norton/TRW Ceramics (now part of NAC) produced and delivered to AlliedSignal Engines net-shape ETEC program blades from Revision 0 of the NT154 process. These blades were fabricated using all plaster molds, and exhibited surface pitting and relatively low asprocessed surface strength. For the 331-200 blade parts-buy programs during 1993 and 1994, NAC's NT154 Revision 1 process was used. Revision 1 of the NT154 process reflects the change from alcohol milling to water milling and includes implementation of optimized processing conditions from NAC's In 1994, the 331-200 blade ATTAP work. manufacturing development effort under the CTEDP program evolved to utilization of NT164 and single directional pressure slip casting. This same process is being employed for NT164 CSGT first generation blades. Mechanical properties have improved markedly over the 1976-1994 time period. Asprocessed surface flexural strength for the current process now exceeds the bulk-ground strength for the NC132 material used in Norton Company's first blade development effort.

SUMMARY AND CONCLUSIONS

NAC has successfully completed its ATTAP effort in which processes for pressure slip casting of NT154 and NT164 were optimized. Blade process development activity has continued under the CTEDP, CSGT and other advanced gas-turbine programs. Accomplishments during 1992-1994 are summarized below:

- Characterization of NT154 and NT164 has continued. A significant data-base of critical mechanical properties for this material now exists both at NAC, Allison, AlliedSignal, Solar Turbines, other engine builders, and a number of independent laboratories. Flexural strength, fracture toughness, static and dynamic fatigue, creep, and thermal property information are available for engine design and analyses. NT164 has been measured to have approximately four times the creep life of NT154 at 1370°C. The as-processed surface strength of NT164 is ≈ 10% than NT154 at room temperature and ≈ 30% higher at 1316°C.
- NAC is utilizing the expertise and experience of the

ATTAP to supply hardware into other privately sponsored or DOD sponsored ceramic demonstration programs. One such effort was AlliedSignals's GTCP85 Nozzle Demonstration program. NT154 ceramic nozzles have performed very successfully within actual engine tests -enduring more than 13,000 total hours of operation, including more than 3000 starts and 10 hours of sand ingestion -- all without failures.

- NAC has developed a single directional pressure slip casting approach for blades under the CTEDP program. This process minimizes: (1) The occurrence of pits on and other surface defects on the blade flowpath features; and (2) dimensional deviations in the form of closed airfoil distortion.
- Component processing technology developed under the CTEDP is being utilized in the fabrication of CSGT program first generation blades. These first generation NT164 blades have been pressure slip cast using the single directional casting mold configuration.
- Blade process development efforts under the CTEDP and CSGT programs will continue in 1995.
 Work under the CTEDP will focus on development of a production process for NT164 331-200 blades. The CSGT effort will focus on fabrication of first generation engine NT164 blades.

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Properties	Typical Values		
	NT154	NT164	
Density (g/cc)	3.232 ± 0.004	3.190 ± 0.004	
Elastic Modulus (GPa)	310-320	306 (1)	
Shear Modulus (GPa)	126	N/A	
Poisson's Ratio	0.273	N/A	
Hardness (Kg/mm²)	1620	1891 (1)	
Thermal Expansion Coefficient	3.93 × 10 ⁻⁶ /°C	4.30 x 10 ⁻⁶ /°C	
Thermal Conductivity (W/m°K): @25°C @1400°C	37.6 15.8	N/A N/A	
22°C Bulk-Ground Surface Mechanical Properties: Average Flexural Strength (MPa) Characteristic Strength (MPa) Weibull Modulus Fracture Toughness (MPam ^x) Tensile Strength (MPa)	896 ± 55 (2) 917 8 - 12 6.0 762 ± 112 (2)	887 ± 100 917 9 - 12 5.6 546 - 700 (4)	
22°C As-Processed Surface Mechanical Properties: Average Flexural Strength (MPa) Characteristic Strength (MPa) Weibull Modulus	490 ± 90 524 6	637 ± 62 669 8	
1370°C Mechanical Properties: Average Flexural Strength (MPa) Characteristic Strength (MPa) Weibull Modulus Fracture Toughness (1200-1400°C) MPam [*] Tensile Strength	641 ± 40 669 13.0 4.1 (5) 240 - 520 (4)	630 ± 49 664 13.0 N/A 388 - 567 (4)	
1260 °C Tensile Creep Rate (300 MPa) /s	1.9 x 10*	2.2 x 10 ^{.4}	
1370°C Tensile Creep Rate (200 MPa) /s	8.7 x 10 ^{.7}	1.0 x 10*	
(1) UDRI Data.			

Table 1 - Properties of NT154 and NT164 Silicon Nitride

(2) Controlled Flaw Method; 10 Kg Indent.

(3) Includes CIP and Cast Samples, (UDRI and ORNL Data).

(4) Loading Rate Dependent, (UDRI and ORNL Data).

(5) Indentation Method.

Table 2 - Blade Process Proposed Key Characteristics for SPC.

Process Step	Key Characteristic				
Powder Processing	(1) Wt. % Oxygen.				
Forming	(2) Viscosity.(3) Green Density.				
Dense Casting Inspection	 (4) Density. (5) Yield of Castings Meeting Surface Quality Requirements. (6) Room Temperature Weibull Modulus (Bulk-Ground). (7) Room Temperature Average and Low Flexural Strength (As-Processed). (8) Airfoil Chord Length at Base, Mid, and Tip. (9) Maximum Deviation Between Nominal Flowpath and Best Fit (5 points on airfoil, 5 points on platform). 				
Machining	(10) Dovetail Profile to Itself. (11) Platform Edge Location With Respect to the Dovetail.				
Final Inspection	(12) Airfoil Location With Respect to Machined Datums. (13) Overspeed Spin Proof Test Yield.				







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Figure 2 SEM Photograph of Fracture Surface of NT154 Silicon Nitride As-Processed Surface Flexural Specimen

Figure 3 SEM Photograph of Fracture Surface of NT164 Silicon Nitride As-Processed Surface Flexural Specimen





Figure 5 - CMM Inspection Data of Tip Section of Typical 331-200 NT154 Blade



Figure 6 Grey-Level Line Scan of a Microfocus x-ray Film of a Green AGT101 Rotor Cross Section



Figure 8 - 331-200 Blade Pit Size Distribution



Figure 7 - Cross Section of 331-200 Production Process Blade Casting Mold

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Figure 10 Photograph of NT154 and NT164 Silicon Nitride Ceramic Turbine Blades



 Table 3

 Summary of Silicon Nitride Turbine Blade Development Programs

Program	DARPA	ETEC	CTEDP Parts- Buy	CTEDP Development	CSGT
Customer	Garrett Turbine Engine Company	Garrett Engine Division, Allied Signal Aerospace.	AlliedSignal Engines	AlliedSignal Engines	Solar Turbines Incorporated
Time Period	1976-1981	1988-1990	1993-1994	1994-1996	1994-1995
Silicon Nitride Material	NC132	NT154	NT154	NT164	NT164
Process	Profile Grinding from Hot-Pressed Billets	NT154 Process Revision 0, Pressure Slip Casting Using All Plaster Molds	NT154 Process Revision 1, Pressure Slip Casting Using All Plaster Molds.	NT164 Process Revision 1, Pressure Slip Casting Using Single Directional Casting.	NT164 Process Revision 1, Pressure Slip Casting Using Single Directional Casting.
Room Temperature Mechanical Properties (Bulk-Ground Surfaces): - Average ± 1 Standard Deviation (MPa) - Characteristic Strength (MPa) - Weibull Modulus	634 ± 62 655 12.6	896 ± 138 N/A N/A	896 ± 55 917 8 - 12	887 ± 100 917 9-12	952 ± 117 1002 13.4
Room Temperature Mechanical Properties (As-Processed Surfaces): - Average ± 1 Standard Deviation. - Characteristic Strength - Weibull Modulus	634 ± 62 655 12.6	614 ± 48 N/A N/A	658 ± 51 524 6	712 ± 114 N/A N/A	558 ± 49 580 13.3
1370°C Mechanical Properties (Bulk-Ground Surfaces): - Average ± 1 Standard Deviation. - Characteristic Strength - Weibull Modulus Note: NC132 tested at 2200°F.[12]	407 ± 28 421 13.4	462 ± 55 N/A N/A	641 ± 40 669 13.0	630 ± 49 664 13.0	714 ± 64 742 13.42
1370°C Mechanical Properties (As-Processed Surfaces): - Average ± 1 Standard Deviation. - Characteristic Strength - Weibull Modulus Note: NC132 tested at 2200°F.[12] (1) Test Temperature = 1316°C.	407 ± 28 421 13.4	441 ± 55 N/A N/A	495 ± 102 (1) N/A N/A	667 ± 95 (1) N/A N/A	N/A N/A N/A