RECEIVED
JUN 30 2000
OSTI

ORNL/TM-1999/240

**OAK RIDGE
NATIONAL
LABORATORY**

**THE STATUS OF CERAMIC TURBINE
COMPONENT FABRICATION AND
QUALITY ASSURANCE RELEVANT TO
AUTOMOTIVE TURBINE NEEDS**

FINAL REPORT

**David W. Richerson
Richerson and Associates**

**MANAGED AND OPERATED BY
LOCKHEED MARTIN ENERGY RESEARCH CORPORATION
FOR THE UNITED STATES
DEPARTMENT OF ENERGY**

ORNL-27 (3-96)

This report has been reproduced from the best available copy.

Reports are available to the public from the following source.

National Technical Information Service
5285 Port Royal Road
Springfield, VA 22161
Telephone 703-605-6000 (1-800-553-6847)
TDD 703-487-4639
Fax 703-605-6900
E-mail orders@ntis.fedworld.gov
Web site www.ntis.gov/ordering.htm

Reports are available to U.S. Department of Energy (DOE) employees, DOE contractors, Energy Technology Data Exchange (ETDE) representatives, and International Nuclear Information System (INIS) representatives from the following source.

Office of Scientific and Technical Information
P.O. Box 62
Oak Ridge, TN 37831
Telephone 865-576-8401
Fax 865-576-5728
E-mail reports@adonis.osti.gov
Web site www.osti.gov/products/sources.html

Reports produced after January 1, 1996, are generally available via the DOE Information Bridge.

Web site www.doe.gov/bridge

This report was prepared as an account of work sponsored by an agency of the United States government. Neither the United States government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

**THE STATUS OF CERAMIC TURBINE COMPONENT FABRICATION AND
QUALITY ASSURANCE RELEVANT TO AUTOMOTIVE TURBINE NEEDS**

FINAL REPORT

**David W. Richerson
Richerson and Associates**

**Prepared by
David W. Richerson
Richerson and Associates
2093 E. Delmont Dr.
Salt Lake City, UT 84117**

Date Published: February 2000

**Funded by
U.S. Department of Energy
Assistant Secretary for
Energy Efficiency and Renewable Energy
Advanced Automotive Materials Program
Office of Transportation Technologies
EE 07 01 00 0 and
Microturbine Technology Program
Office of Industrial Technology
ED 19 04 03 1**

**for
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831-6585
Managed by
Lockheed Martin Energy Research Corporation
under Contract DE-AC05-96OR22464**

TABLE OF CONTENTS

ABSTRACT	1
EXECUTIVE SUMMARY	1
INTRODUCTION.....	3
OBJECTIVES AND APPROACH.....	3
NDE SYMPOSIUM AT THE AMERICAN CERAMIC SOCIETY MEETING	4
MEETING WITH AUTOMOTIVE MANUFACTURING REPRESENTATIVES	5
SUMMARY OF MEETING WITH AUTOMOTIVE COMPANIES	11
VISIT TO DELPHI ENERGY AND ENGINE MANAGEMENT SYSTEMS	11
VISITS TO THE CERAMIC MANUFACTURING COMPANIES ALLIED SIGNAL CERAMIC COMPONENTS AND KYOCERA INDUSTRIAL CERAMICS CORP.....	13
NDE METHODS.....	18
SUMMARY AND DISCUSSION	26
ASSESSMENT OF CURRENT TECHNOLOGY STATUS	26
ASSESSMENT OF NDE AND COST ISSUES.....	28
REVISED STATUS OF CERAMIC TURBINE COMPONENT DEVELOPMENT.....	29
RECOMMENDATIONS.....	30
ACKNOWLEDGEMENTS.....	31
APPENDIX A – CERAMICS FOR TURBINE ENGINES.....	33
APPENDIX B – QUESTIONS POSED TO AUTOMOTIVE REPRESENTATIVES.....	37
APPENDIX C – QUESTIONS POSED TO CERAMIC COMPANIES.....	39
APPENDIX D – NONDESTRUCTIVE EVALUATION TECHNOLOGIES FOR AUTOMOTIVE CERAMIC APPLICATIONS.....	41

Research sponsored by the U. S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Transportation Technologies as part of the Advanced Automotive Materials Program, and the Office of Industrial Technology as part of the Microturbine Technology Program, under contract DE-AC05-96OR22646 with Lockheed Martin Energy Research Corporation.

ABSTRACT

This report documents a study funded by the U.S. Department of Energy (DOE) Office of Transportation Technologies (OTT) with guidance from the Ceramics Division of the United States Automotive Materials Partnership (USAMP). DOE and the automotive companies have funded extensive development of ceramic materials for automotive gas turbine components, the most recent effort being under the Partnership for a New Generation of Vehicles (PNGV) program. As the PNGV program approached a decision point regarding which propulsion engine concepts to focus on in the near term, the concern was raised whether ceramic components could meet automotive cost and reliability standards. A significant part of this concern was directed towards the current high cost of non-destructive evaluation (NDE) and whether NDE or other technology could assure reliability.

EXECUTIVE SUMMARY

The specific tasks of this study were the following:

- * Review the quality standards required by the automotive industry and the current level of NDE commonly used to meet these standards.
- * Review the current status of ceramic component fabrication in comparison with automotive quality standards.
- * Assess whether it is possible for ceramic turbine component manufacturing to meet both reliability and cost requirements.
- * Review NDE technologies and recommend a development path with potential to meet automotive needs.
- * As a secondary issue, consider NDE needs for ceramics in automotive electronics systems.

Automotive companies have well-defined quality standards (QS-9000) that commonly require 100% NDE and/or proof testing to assure that components such as axles, electronics systems, spark plugs, and even complete engines meet specifications. Some of these inspections can contribute to around 20% of the cost of the component, although the percent of total cost is usually much lower. Presently, the cost of NDE (surface and internal defect examinations plus dimensional measurements) for an advanced gas turbine component such as a silicon nitride automotive-size rotor exceeds 20%.

To assess the potential for reducing the cost of NDE, the quality systems and silicon nitride commercialization experience at several advanced ceramics companies were reviewed. Ceramic companies have made major progress in recent years in establishing quality systems that meet automotive QS-9000 standards, and in developing advanced silicon nitride ceramics that are capable of performing in an automotive turbine duty cycle. However, most ceramic turbine components have only been fabricated in prototype quantities, so production level of

manufacturing is at an early stage of maturity. Some silicon nitride components, though, have reached various levels of production and can provide an insight into the future commercialization of turbine ceramics. For example, Kyocera silicon nitride automotive turbocharger rotors reached production volume of 30,000 units per month in the late 1980s and early 1990s in Japan. They were fabricated in a semi-automated production line that included several NDE steps and 100% proof testing. Over a five-year period, the price was reduced by nearly a factor of 10 to reach a level, which approaches the automotive target for a ceramic turbine rotor. The silicon nitride turbocharger rotors have achieved a record of reliability that surpassed metallic turbocharger rotors.

Another example of where large volume production of silicon nitride has been achieved is high-performance bearings. Production began in about 1990 and has now reached a volume exceeding 25 million balls per year. The price of a half-inch ball bearing has dropped from about \$50 to about \$7. Reliability has exceeded that of metals.

Silicon nitride turbine nozzle guide vanes are presently at the field test stage. AlliedSignal Ceramic Components (ASCC) is producing about 100 per month. In transitioning from the initial prototypes to 100 per month, ASCC achieved greater than 75% cost reduction. Inadequate data are available to assess long-term reliability, although over 50,000 hours of field-testing have been accumulated.

These examples suggest that the ceramic suppliers are on track to have the fabrication technology to produce reliable turbine components at acceptable cost. However, inspection costs are still too high, and procedures are not well defined to sort good parts from bad parts and assure reliability to automotive standards. Presently fluorescent-penetrant inspection, visual inspection, and conventional film X-ray radiography are used, but do not have verified accept-reject criteria and are not applied in a cost-effective, production-viable mode. Efforts need to be initiated to establish a database of correlation of component inspection versus reliability for these and other NDE techniques. Fast X-ray computed tomography (CT) with high-resolution amorphous silicon detectors, resonant ultrasonic inspection (RI), and laser scattering are other techniques recommended for evaluation. This report contains recommendations for programs, including a cooperative effort between the engine companies and ceramic companies.

The study further concludes that design is an integral factor in both reliability and cost of ceramic components. Virtually all of the ceramic turbine component failures in recent years can be traced to design problems such as foreign object damage and abnormal contact stress. Designs need to be reviewed to minimize the possibility of failure due to abnormal extrinsic factors, or to make the ceramic parts robust enough to survive these factors. This may require compromises in aerodynamic performance and in fabrication and inspection. Concurrent engineering involving active participation of the engine companies and ceramic suppliers is necessary.

The remainder of this report covers discussions with automotive manufacturing representatives and ceramic turbine component developers, reviews the viability of various NDE techniques, and provides conclusions and recommendations. Appendices provide supplemental information.

INTRODUCTION

Extensive programs have been conducted in the US since the early 1970s to develop ceramic materials for gas turbine engines. The primary driver for these programs has been the automotive application and the desire to achieve an alternate powertrain with > 40% efficiency, reduced emissions, and multifuel capability. Ceramic materials are required to allow the turbine to operate at a high enough temperature to have an acceptable level of fuel efficiency.

Major progress has been achieved in the properties and fabrication of advanced ceramic materials, in design and life prediction, and in engine testing. A brief review of the challenges that were encountered and of the progress that has been accomplished is included in Appendix A. In spite of the progress and some impressive engine demonstrations, there is still concern regarding reliability and cost. Presently, extensive in-process and post-process characterization and non-destructive evaluation (NDE) plus proof testing are deemed necessary to qualify a ceramic turbine part for assembly into an engine. These add substantially to cost, so much so that there is concern if the cost can be reduced to a level compatible with the allowables for the automotive industry. Furthermore, there is an inadequate database to validate whether all the inspections correlate with a guaranteed reliability. The purpose of this document is to assess the current status of ceramic turbine component fabrication/qualification and to recommend an appropriate course of action.

OBJECTIVES AND APPROACH

The study has two primary goals:

1. Obtain an understanding of the current and emerging NDE processes and their potential for inspecting/qualifying ceramic components compatible with automotive industry reliability requirements and cost requirements.
2. In collaboration with NDE specialists and ceramic component manufacturers, identify specific directions for future developments needed to demonstrate an inspection/qualification procedure that meets automotive requirements.

The study has been funded jointly by DOE-Office of Transportation Technologies (OTT) and DOE-Office of Industrial Technologies (OIT) through Oak Ridge National Laboratory (ORNL) in cooperation with the Ceramics Division of USAMP. Table 1 identifies the key steps in the study.

Table 1. Schedule and key activities of the study

Schedule	Activity
April 12, 1997	Draft plan submitted to DOE and USAMP
May 4, 1997	Meeting with DOE and USAMP to finalize plan
May 5-7, 1997	NDE Symposium at American Ceramic Society meeting and individual discussions
June 10-11, 1997	Meetings with USAMP and automotive manufacturing representatives to discuss current procurement standards and cost and inspection scenarios
June, July 1997	Meetings with AlliedSignal Ceramic Components and Kyocera Industrial Ceramics
July, August, 1997	Review of NDE approaches
August, September, 1997	Analysis of information gathered
October, 1997	First draft of final report; meeting with USAMP
November 1997-January 1998	Supplemental NDE information from William A. Ellingson, Argonne National Laboratory
June-October 1998	Update of report and reviews
March 1999	Final revisions and updates

The scope of the study includes the role of NDE in in-process quality assurance (QA), post-process QA, process development/optimization, and in-service life prediction.

The following sections summarize the information gathered under the activities of Table 1 and provide recommendations.

NDE SYMPOSIUM AT THE AMERICAN CERAMIC SOCIETY MEETING

This was a special symposium at the 99th Annual Meeting of the American Ceramic Society (ACerS) held in Cincinnati, Ohio, in May 1997. Papers from the symposium have been published as *Nondestructive Evaluation of Ceramics, Ceramic Transactions Vol. 89*, Christopher H. Schilling and Joseph N. Gray, eds., ACerS, Westerville, Ohio, 1998. A portion of the symposium organized by Dave Stinton of ORNL and Bob Powell of General Motors was directed specifically towards automotive ceramic gas turbine issues. The following are some of the key points that were presented or were introduced during a subsequent discussion session. Note that some address NDE directly, but others address steps in the fabrication process that might decrease the need for NDE.

1. Take advantage of modern instrumentation and conduct a focused effort to demonstrate an intelligent, automated system for a specific component or application.

2. Presently there is inadequate correlation between NDE and part performance, i.e., the point of failure is typically not at a specific defect previously identified by NDE. However, very little testing to failure has been conducted for parts containing known flaws. Greater testing to failure, especially of component shapes, with careful accumulation of a database of flaw size and type associated with the failures is needed to determine whether NDE can be effective for identifying critical flaws. A useful database could be obtained by proof testing to failure parts that have been extensively inspected by NDE.
3. Test bars of turbine ceramics often fracture at surface damage resulting from machining damage. A NDE technique that quickly detects these types of flaws is needed.
4. Close collaboration between manufacturers and users on inspection techniques and specifications is needed.
5. National laboratories and universities feel that they can contribute to solving some of the NDE/inspection issues, but need real samples from industry.
6. NDE/ inspection costs for ceramic components currently often exceed 20% of total costs. The general consensus at the meeting was that this is too high to be viable for automotive application.
7. The developers of ceramic turbine components need to learn from the semiconductor industry. They use highly refined powders and other raw materials to avoid contamination with defects. Many defects in structural ceramics are picked up during the fabrication process either as debris in the starting materials or as in-process contamination. Much progress has been made by the raw material suppliers and by the ceramic fabricators, but is there room for even more progress? Can materials and processes reach a point where the reliability and robustness are built in and NDE is either not required or minimized?
8. The general attitude of attendees of the symposium appeared to be that 100% NDE is not consistent with automotive cost structures.

The above perceptions of the attendees of the symposium may or may not be valid. Part of the objective of the study was to identify some of the key issues and perceptions and to dig deeper in an effort to understand in what ways current perceptions are valid or invalid.

MEETING WITH AUTOMOTIVE MANUFACTURING REPRESENTATIVES

A meeting was held at the offices of USCAR in Detroit on June 10, 1997. It was attended by USAMP members, manufacturing/procurement representatives from the automotive companies, and representatives from DOE and ORNL. The purpose of the meeting was to review the current procurement guidelines, standards, and NDE practices for a variety of automotive components to provide a baseline from which to assess the future needs of ceramic turbine components. Keith Carson of Ford Motor Company was the key individual selected by USCAR to provide the primary input. A list of questions sent to Keith and to USAMP members is included in

Appendix B. Following a brief introduction by Keith, these questions were used to guide discussion during the meeting.

Keith started the meeting by providing four booklets on quality inspection practices prepared jointly by Chrysler, Ford, and General Motors between 1992 and 1995. These are briefly described in Table 2.

Table 2. Documents provided by the automotive companies to guide suppliers

Document Title	Description of Document
Quality System Requirements - QS-9000	Defines the quality system expectations of the automotive companies for internal and external suppliers of production and service parts and materials. The goal is to provide the suppliers with an ISO-9000 based quality system that provides for continuous improvement by emphasizing defect prevention and the reduction of variation and waste in the supply chain.
Quality System Assessment - QSA	Formal procedure for evaluation of a supplier's Quality System. Includes (1) Quality System Documentation Review to determine if the quality manual meets QS-9000 requirements, (2) On-Site Audit to assess the effectiveness of implementation of the quality system, and (3) Analysis and Report.
Statistical Process Control - SPC	Standardized reference manual for statistical process control and continuous improvement.
Production Part Approval Process PPAP	Defines the procedure for determining if the supplier has established a process that delivers parts that meet all of the customer's requirements and specifications. Requires evaluation of a negotiated number of parts manufactured at the production site using the production tooling, gaging, process, materials, operators, environment, and process settings.

All production suppliers to the automotive industry must be QS-9000 certified. Ceramic companies hoping to become a supplier to the automotive companies should obtain copies of the four booklets. They can be obtained by calling Automotive Industry Action Group (AIAG) at 810-358-3003. Figure 1 (from the QS-9000 booklet) summarizes the Quality System that each supplier is required to have for each part.

QUALITY SYSTEM DOCUMENTATION PROGRESSION

Fig. 1. Customer quality expectations defined in the QS-9000 booklet.

A formal sequence of events is followed before a new part, concept, or design is introduced into an automobile. The sequence is illustrated in Fig. 2. First, an idea is presented and discussed. If the idea is judged to have merit, a cost assessment is conducted. If the economic assessment is favorable, a product engineering team and plan are established. This involves system and component design, working with suppliers, preparation of prototypes, and testing. If viability is demonstrated, advanced quality planning (AQP) is conducted to define all of the controls that will be imposed on the manufacturing to assure that each part will meet the specifications of the application. The final step before the part or system can enter production is the Production Part Approval Process (PPAP). This involves evaluation of roughly one hour to one shift of production and usually a minimum of 300 parts to demonstrate that all specifications of properties and dimensions are met. PPAP is required for all new parts and also whenever there has been a change in material, design, tooling, or supplier or when a line has produced discrepant parts.

Fig. 2. Sequence used by the automotive companies to transition an idea to a product.

The question was asked in the meeting where ceramic components for a turbine engine presently fit into the sequence shown in Fig. 2. The response from the representatives of the automotive companies was the Idea stage. Their response seems reasonable if we look at the production requirements defined by Mr. Carson (see Table 3). It is apparent from Table 3 that extensive engine and vehicle testing are required before durability and warranty life can be demonstrated to the satisfaction of the automotive companies.

The remainder of the meeting on June 10 focused on discussion of the questions listed in Appendix B. The following are some highlights.

Question 2a. What parts currently require functional measurements or demonstrations as part of the quality assurance before an auto company will accept the part?

Answer: All parts.

Questions 2b-d. These questions pertain to spark plugs and oxygen sensors and are addressed in the next section reviewing the visit to Delphi.

Question 2e. Are there functionality checks specified for drivetrain components?

Answer: The checks vary depending on the part. The following are some examples.

(1) Every axle is tested for stress, torque, and gear noise. This is done in-process using equipment that makes go-no go decisions based on measurements such as amplitude and decibels. Inspection for the axle can be greater than 25% of the cost of the part.

(2) Every component in the engine is evaluated dimensionally with check fixtures at some stage or multiple stages in process and/or post-process. Every assembled engine is tested on a dynamometer. This is automated and takes about 60 seconds.

Table 3. Quality issues to be assessed before ceramics can enter production (prepared by Keith Carson)

FUNCTIONALITY	Functional Fit Bench Test Road Test In-process Inspection Non-destructive Testing
DURABILITY	Test Track Fleet Feedback Information Lab Test/Fatigue Test
WARRANTY LIFE	Product Design/Proveout Customer Feedback Government/Independent Testing Dealer Reports and Back Charges
DIMENSIONAL	Part Dimensional Layout - CMM Part/Check Fixture Variable/Attribute - SPC GDT PPAP AQP
BASIC REQUIREMENTS	Proof Testing - 100%? Material Certification Destructive Testing \$\$ Tight Tolerances XXX Check Fixture Scrap/Reclaim? Controlling the Unknown Controlling the Process Improving Product CPK/Distribution Continuous Improvement/Price Improvement Material Handling
PROCESS CONTROLS	Non-destructive Testing Special Testing - Non-traditional Requires Special Training/Certification

- (3) Saturn does X-ray inspection of engine blocks to look for porosity and retained sand.
- (4) Cast connecting rods receive 100% eddy current and fluorescent dye penetrant inspections.
- (5) A sampling of casehardened parts such as gears are cut and polished and hardness measured.
- (6) All glass parts are inspected for optical quality.

Question 2f. What functionality-based inspections might be required for ceramics?

Answer: Essentially the same types required for metals selected specifically to verify the key functions that the ceramic component must perform. The actual tests will be determined during the Product Development and AQP stages.

Question 2g. Are any components proof tested?

Answer: Yes. Every oxygen sensor is proof tested for strength with internal pressurization. The engine dynamometer test is essentially a proof test for the complete engine.

Question 3a. Are there any specifications for inspection that relate to durability/reliability/warranty life?

Answer: These factors are addressed during product development by extensive long-term testing before a commitment to production is made. Even with this developmental testing, certain components receive durability-type tests during production. For example, axles are tested in a horizontal fatigue test for 100,00 cycles. Each vehicle is operated on a test roller for one hour at engine red line conditions.

Question 3b. Will standards and quality assurance testing be required to verify durability/reliability/warranty life for ceramic turbine components?

Answer: Verification of these for each component and the complete engine will be the focus of extensive testing during product development. The determination of need for specifications and quality assurance procedures will be defined during this testing and during the Advanced Quality Planning (AQP) stage prior to production. The consensus seemed to be that ceramics would probably require a much larger amount of product-development testing than metals to provide assurance of durability and reliability.

Question 3c. Are there any existing cases that might be similar to that expected for ceramic turbine components?

Answer: No. The key concern with ceramics will be premature catastrophic failure. Perhaps we should review the history of use of ceramic turbocharger rotors, pre-chambers, glow plugs, oxygen sensors, and cam follower rollers, as well as developmental testing of valves, to better identify issues that we should address. Included in these reviews should be a comparison of the stress state versus the anticipated stress in future automotive turbine parts.

Question 3d. Do any components currently have testing to verify resistance to shock loading, such as electronic control systems?

Answer: These issues are addressed during product development such that highly robust systems are verified before release into production. Systems and vehicles are exposed to shaker tests and to on-road tests such as the Belgian block track. Ceramic turbine components survived these tests during the DOE/Allison CATE program, so we know that ceramics can survive the shock loading if they are properly designed into the engine.

Question 4a. What current dimensional inspection criteria and technology are applicable to future needs with ceramic turbine components?

Answer: All metallic parts have dimensional specifications and inspections, including some that are quite tight. For example, the fuel injector nozzle is measured to less than one-ten thousandth of an inch. Piston pins also have tight tolerances. These tolerances are met by matching.

SUMMARY OF MEETING WITH AUTOMOTIVE COMPANIES

In some ways these discussions were encouraging and, in others, frustrating. The encouraging news is that many parts currently used in automobiles receive 100% inspection, and some receive 100% proof testing; fatigue testing, or dynamometer testing. In some cases, the inspection/QA accounts for greater than 25% of the cost of the part. So, high cost of inspection alone has not prevented production use of key automotive components. The frustrating news is that the automotive manufacturing personnel seem to believe that ceramics are at a very early stage of consideration regarding turbines, essentially still at the "Idea" stage. They seem to have a lack of confidence that ceramic turbines can meet the cost and reliability needs of the automotive industry.

One key piece of information that could potentially benefit the ceramic companies came out of the meeting. The automotive companies are so concerned about quality and ability to maintain a competitive warranty that they are willing to help their suppliers establish effective Quality Systems. In the past, this has included buying and installing expensive manufacturing and inspection equipment at the suppliers. In exchange, the supplier is expected to maintain the equipment, provide favorable pricing, and be responsible for any of their parts that fail under warranty.

VISIT TO DELPHI ENERGY AND ENGINE MANAGEMENT SYSTEMS

Delphi in Flint, Michigan, was visited June 11, 1997, with Dr. Fred Kennard as the host. Three topics were discussed: (1) spark plugs, (2) oxygen sensors, and (3) silicon nitride.

Spark Plugs

Delphi was previously AC Spark Plug and has been in the business of manufacturing spark plugs since the early 1900s. Worldwide production of spark plugs is more than one billion per year, so a plant the size of the Delphi plant is likely to produce at least half a million per day (Dr. Kennard would not divulge their production output). Each spark plug has a high-alumina ceramic insulator. The ceramic insulator is exposed to high levels of thermal shock, mechanical pressure pulses, and high voltage. It, therefore, must perform both as an electrical ceramic and as a structural ceramic, and requires in-process and post-process inspections to assure function and reliability.

Most spark plug insulators in the world are fabricated by automated dry-bag isostatic pressing of a spray dried powder, followed by green machining (machining the as-pressed part before high temperature firing). Tight specifications are required on raw materials. Even with these tight specifications, slight modifications must be made with each batch of powder during the pressing operation (usually pressure setting). A very small percentage of parts are inspected after pressing

to assure that tooling is in adjustment and to determine the correct pressure setting. Other in-process inspections are also conducted on a small level of sampling basis at key points in the process. The actual inspection details are proprietary, but involve things such as assurance that the density is acceptable after firing, that the glaze is complete, and that the electrical resistance is acceptable. Individuals at Delphi did not provide information on total process yield or percentage of the total cost attributed to inspections. Based on personal experience, my guess is that the yield is well over 90% and that the inspection cost is less than 5%. The inspection steps appear to be automated or semi-automated.

Spark plug manufacturing and inspection are probably not good direct comparisons for turbine ceramics. Turbine ceramics will require a higher level of inspection, probably including a proof test. However, some aspects of the spark plug manufacturing process are encouraging and applicable to turbine ceramics. Specifically, a number of automated or semi-automated in-process inspections have been built into the process without resulting in a cost-prohibitive product.

Oxygen Sensors

Oxygen sensor manufacturing is one step closer to the envisioned needs for turbine ceramics. Oxygen sensors require a much more expensive raw material powder (zirconium oxide) than spark plugs (aluminum oxide). Every oxygen sensor also receives an internal pressurization overstress proof test. The proof test replaces dye penetrant inspection.

Fewer oxygen sensors are produced daily than spark plugs, but still a large number (probably at least 200,000 per day in individual plants). Automobiles initially required one oxygen sensor, but others are being added to help meet increasingly stringent emissions regulations. Some vehicles now have as many as four sensors.

Oxygen sensor ceramics are fabricated in a batch process rather than a continuous process. The tooling in the molding and green machining steps often must be tweaked for each batch, so the first parts from the batch must be audited for dimensions. This is done with a series of gages and by shadow profile. Typically less than 0.1% of the parts are measured. If the setup is right, then production proceeds with the batch. Additional random sampling of a small percentage of parts is conducted after the firing operation. The parts are then 100% proof tested and 100% visually inspected for chips on the seal surfaces and for contaminants.

Silicon Nitride

Delphi (when they were AC Spark Plug) conducted development of silicon nitride for a number of years around 1980. A couple of the individuals now at AlliedSignal Ceramic Components were at AC at the time. Delphi is no longer actively pursuing silicon nitride. However, I asked their opinion on the challenges of producing silicon nitride turbine components for automobiles. They mentioned several factors to consider:

1. A large percentage of the powder removed in the green machining process of spark plugs to achieve final dimensions is recycled. This is an important factor in being cost competitive, even with a very inexpensive powder such as alumina. Silicon nitride powder is much more expensive (probably by a factor of 20 at this time). Furthermore, processes such as gel casting make recycling of powder difficult. This means that silicon nitride fabricators will be challenged either to learn to recycle powder, or to make parts close to net shape to minimize waste that is not recycled.
2. The critical flaw size (the surface or internal material defect where a fracture starts) for highly stressed turbine components is on the margin of detection limits for standard NDE techniques such as dye penetrant and X-ray radiography. Very refined processing will be required to avoid flaws of the critical size for failure under the application stresses. Proof testing offers a better way of assuring that no defective parts are released to production.
3. If an NDE technique is developed with acceptable resolution, it will have to be real-time (as opposed to film, for example) to be viable from a cost perspective.

VISITS TO THE CERAMIC MANUFACTURING COMPANIES ALLIED SIGNAL CERAMIC COMPONENTS AND KYOCERA INDUSTRIAL CERAMICS CORP.

The following agenda was recommended to the ceramic manufacturers:

- * Review of the objectives of the study
- * Review of the meeting with the auto company representatives
- * Discussion of the manufacturer's philosophy of NDE/proof testing/SPC evolution to meet automotive requirements
- * Discussion of the issues of cost, verification of function, guarantee of reliability/durability/warranty life
- * Brainstorming program needs to achieve reliable ceramic turbine component fabrication and inspection

Prior to each meeting, the ceramic manufacturing company was sent a copy of the letter to Keith Carson (Appendix B) and a list of specific information I believed was important to include in the assessment for USAMP. This list is included as Appendix C.

AlliedSignal Ceramic Components

AlliedSignal Ceramic Components in Torrance, California, was visited on June 17, 1997.

AlliedSignal Ceramic Components (ASCC) has been working for the past few years to establish a quality system that meets U.S. and International standards. They received ISO 9001

certification in March 1995 and were aware of the automotive companies' quality system documents prior to my visit.

ASCC has process-control documents to define each step of the process and to assure that the proper measurements are made relevant to customer specifications. ASCC monitors their process continuously using SPC techniques. They are establishing a significant database, including strength measurements for each batch. Most customer specifications for turbine components require NDE. All seem to require 100% visual and dye penetrant and most require conventional film X-ray radiography. The inspection philosophy is presently conservative, i.e., if there is a question or uncertainty, the part is usually rejected. ASCC does not believe there has been much correlation between observed defects and component fractures.

AlliedSignal conducted extensive process development during the late 1980s and early 1990s. They used NDE and destructive testing at various stages in the process and on final parts to guide process improvements. They found that some critical flaws such as density gradients and high-density inclusions could be detected at the powder compact stage by X-ray radiography. CT and microfocus X-ray provided greater resolution than conventional X-ray, but were too expensive for other than process development. If a low-cost, rapid-flow-through CT unit were available, it would be a valuable in-process inspection tool. Locating parts with defects at an early stage in the fabrication process, especially before expensive sintering (firing) and machining, is an important part of the strategy to minimize costs.

Based on the discussions at ASCC, their AS-800 silicon nitride material appears to be a viable candidate for automotive applications, but is only at an intermediate stage of maturity. There are occasional variations between batches, but not as frequently as during earlier development. The Weibull modulus is typically around 20, which is double that of silicon nitride ceramics available 10-15 years ago. The fracture toughness is about $8 \text{ MPa}\cdot\text{m}^{1/2}$, which is about double that of conventional ceramics and also is important to reliability. [Note to reviewer: This is presently outdated information, but it is consistent with what was presented at the time of the meeting (June 17, 1997), which is what is being described here].

AS-800 is being evaluated as nozzle guide vanes for two auxiliary power engines at AlliedSignal Engines. One is the 85 Model APU. Over 50,000 hours field-testing have been accomplished under a DARPA insertion program. This has allowed sustained fabrication of a single configuration at a level of about 100 per month. Cost per part has been reduced about 75% and yield has increased to over 75%. ASCC has gained valuable experience during this sustained prototype "production" regarding process control, dimensional inspection, and post-fabrication NDE/proof testing.

Based on their experiences, ASCC identified the following recommendations/needs:

1. Inspection considerations should be an important part of the design process. The ceramics company should be involved with the engine company in the design effort to achieve a design that is (1) amenable to the chosen fabrication process, (2) has potential for low-cost fabrication, (3) can be readily and quickly inspected for key dimensions, and (4) is robust in the application.

The last item includes issues such as aerodynamics versus resistance to impact damage; a trade-off involving slight reduction in performance might result in a large improvement in reliability or improved inspectability.

2. Fracture can be caused by both intrinsic and extrinsic factors. The intrinsic factors are the flaws that can result in the materials during the fabrication process. These have been dramatically reduced over the years as raw materials have been refined and as each process step has been optimized. But can process control be good enough to assure reliability in the application without 100% NDE or proof testing? We need to focus some effort on answering this question, since it can have a large impact on cost. Extrinsic factors include foreign object damage and contact stress. Most ceramic components in recent years have failed due to these causes rather than due to intrinsic flaws. This implies that more effort is required to optimize the component design and the interface with adjacent components. [Note to reviewer: These are issues for net-shape processing, as well as other processing, but are especially important for net-shape processed parts. I chose not to differentiate at this point].

3. Dimensional inspection has been a major challenge. Customers have required tight tolerances and have often specified reference (datum) points that are either difficult to measure from or require expensive tooling. Inspection costs could be reduced if the customer and manufacturer work together during the design phase to define the most cost-effective referencing and establish a single design of inspection tooling.

4. Tight profile tolerances are perceived by designers to be necessary to achieve aerodynamic performance. Is this perception correct? Do the profiles really need to be as tight as currently specified? Focused development on dimensional measurement methods might provide a substantial benefit in speed of measurement and, thus, cost reduction. One option might be to integrate profile measurement into numerically controlled (NC) machining equipment. Non-contact techniques should also be explored.

5. Not enough component testing has been conducted to provide an adequate correlation between flaw size, component survival, NDE, and proof testing. As a result, accept/reject criteria are presently somewhat arbitrary.

6. Recycling powders will be difficult with silicon nitride. To avoid toxic and polluting chemicals, aqueous processes have been developed. Silicon nitride powders and the sintering aids hydrate in the presence of water. This has successfully been accommodated for single use, but would be a significant composition challenge in trying to recycle powder. Emphasis presently is on developing processes that produce the component to near-net-shape.

7. Sustained production of a ceramic component linked to evaluation of the component in the application is necessary to learn the key lessons regarding design, processing refinement, tooling, and inspection. This type of combined effort is required to establish a fixed process, to define what inspections and techniques are necessary, to identify accept/reject criteria, to determine if reliability objectives can be met, and to bring cost down to levels acceptable to the automotive companies.

Kyocera Industrial Ceramics Corporation

A meeting was held at Kyocera Industrial Ceramics Corporation (KICC) in Vancouver, Washington, on July 15, 1997.

The present Kyocera candidate ceramics for turbines are SN 282 (for stationary components) and SN 281 (for rotating components). These are relatively new compositions at the Vancouver facility, but have several years' experience at the Japanese fabrication facility. The US KICC facility has processed multiple batches of test bars, but has not yet achieved the reproducibility demonstrated in Japan. I would classify the SN 281 and 282 materials processed in the US as still in the development stage of maturity. KICC has gone through a similar sequence of process optimization for other silicon nitride materials and is confident that SN 281 and 282 will reach production quality status. KICC is presently fabricating prototype turbine components for Solar, Allison, and AlliedSignal.

KICC received ISO 9002 certification in 1995 and QS-9000 certification a year later. They are in production with silicon nitride materials for three applications. One of these applications is cam rollers for diesel engines. Dedicated equipment has been installed and optimized for fabrication of the cam rollers. Since beginning production, substantial improvements in the processing have resulted in improved quality, reduced cost, and about 35 percentage points increase in yield. As experience was gained, KICC learned that no X-ray radiography was needed and that dimensional inspection was required only on a portion of the rollers. The only NDE required is fluorescent penetrant, which is used for 100% of the rollers.

A substantial portion of the meeting was spent discussing what a production line might look like for fabrication of turbine components for automobiles. We focused on lessons learned from Kyocera experience in Japan with production of 30,000 turbocharger rotors per month. The turbocharger production line was built about 10 years ago. It included proprietary in-process measurements for powder characteristics, slurry properties, and other factors. Specialized equipment and fixtures were developed for surface grinding, with instrumentation integral with the equipment to measure the key dimension after each grinding step. Following fabrication, each rotor was examined by X-ray radiography for gross defects using real-time fluorescent screen imaging and taping for later evaluation. Each rotor was rotated in the X-ray fixture. The inspection took about one minute per rotor. Once the production was established, there was reasonable evidence that the X-ray inspection could have been eliminated.

The Kyocera turbocharger rotor was attached to a metal shaft. The joint was 100% inspected by an ultrasonic technique. All rotors were proof tested by spin testing. The rotor was manually loaded into a fixture. A button was pushed to activate an automated test sequence. Each test required about two minutes. The silicon nitride rotors installed in automobiles had a lower failure rate than metal rotors, indicating that the quality of processing and the inspections were effective in assuring reliability. I have heard that the price of a turbocharger rotor ready to install was less than \$50, but have not received confirmation or denial from Kyocera. If this is correct, it is within reasonable range of the target for an automotive turbine rotor.

Based on their turbocharger experience and more recent experience with cam rollers, Kyocera believes that cost-effective fabrication of rotors and other ceramic components for an automotive turbine is feasible. The key is to establish a fixed process with specialized tooling and substantial automated or semi-automated steps.

The following summarize additional comments from KICC personnel:

1. Achieving low-cost, reliable production is a learning process that involves a sequence of iterations in process parameters, equipment, and tooling. This is expensive and can only be justified if a clear path to commercialization (production) is defined and visible. KICC has concerns with doing tooling and fabrication iterations for a component design that is not a strong candidate for production. They feel that such an approach is backwards. Instead, a detailed design with major input from the ceramic component manufacturer should be conducted to identify a component that is viable for fabrication and inspection and has estimated application stresses within a safe range for the material.
2. KICC suggests implementing ceramics in stages, starting first with lower stress and smaller quantity applications, and then working towards the more difficult applications (such as an automotive turbine) as experience is gained. The key is to locate early stage applications that provide a payoff to the customer and have potential for near-term production. An example might be microturbines (turbogenerators for small-scale power generation such as businesses or small buildings).
3. Accept/reject criteria for current turbine components are arbitrary and not based on a database. We need to find a way to develop a significant database to establish meaningful accept/reject criteria. For example, one customer presently specifies radiography inspection to a 2-.5T level. This requires film X-ray radiography and about 8 hours per part. It is not clear that a 2-.5T level is required. If 2-2T level of inspection were acceptable, real-time X-ray radiography could be used at a rate of 100 parts per day. If improved-resolution, real-time radiography were available, this might be another alternative to reduce inspection time and cost.
4. Fluorescent penetrant inspection (FPI) has proven effective for surface defect detection. A technique also is needed for near-surface detection, such as to detect abnormally large subsurface cracks occasionally resulting from surface grinding. Eddy current and magnetic particle inspections are effective for metals, but do not work for ceramics. We need a comparable technique for ceramics.
5. New inspection techniques are needed that can cover a complete part and follow complex geometries.
6. Can dimensional inspection and defect inspection be integrated into a single method? For example, can a real-time CT scan be devised that can detect material flaws and also inspect for stacking (such as the profile of a rotor blade relevant to the rest of the rotor)?

7. Can non-contact inspection techniques be developed that are faster than present contact methods for dimensional measurement?

NDE METHODS

The objective of this section is to identify various NDE techniques that have been tried with ceramics and to briefly review their viability for inspection of ceramic components. Table 4 lists the general NDE approaches and some modes in which they have been used. Subsequent paragraphs briefly discuss each technique. Appendix D, prepared by Bill Ellingson of Argonne National Laboratory, discusses in greater detail NDE approaches (especially emerging technologies) that have the best chance of meeting automotive requirements for cost-effective qualification of ceramic turbine components.

Visual

All ceramic turbine components are presently inspected 100% visually. Regions of high stress are inspected with an optical microscope typically up to 40X magnification.

Dye Penetrant

Dye-penetrant inspection involves applying a liquid dye to a sample either by painting onto the surface or immersion. The dye penetrates into any open space that intersects with the surface. This can include cracks, isolated pores, networks of pores, and pits. When the dye is washed off with a controlled procedure, residual dye is only present in the defects. Some dyes are visible due to their color, but the ones of proven value to ceramics fluoresce (glow) when stimulated by ultraviolet light. Fluorescent penetrant inspection (FPI) has been used for virtually all prototype ceramic turbine parts. An effective procedure that increases detection limits through the use of magnification is described in Appendix D.

X-ray Radiography

Conventional through-transmission X-ray radiography is illustrated in Fig. 3 using photographic film as an example of the detector. X-ray wavelength photons generated by any one of a number of sources pass through the part being examined and are detected by film, an image intensifier, or other detector. The material absorbs a portion of the X-rays. If the material is of constant thickness and contains no flaws, the detector adjacent to the part will be uniformly exposed and show no variations. Thicker sections of the part and high-density inclusions will absorb more of the X-rays, resulting in lower intensity reaching the detector. Pores, low-density regions, and cracks will absorb less of the X-rays and allow higher intensity to reach the detector. The result is an image with varying gray levels that can be interpreted by the operator.

The size of defect that can be detected by X-ray radiography depends on (1) the thickness of the part and its X-ray absorption, (2) the size of the flaw compared to the thickness of the part, (3) the difference in X-ray absorption between the flaw and the part, and (4) the orientation of the flaw. X-ray radiography is relatively sensitive at detecting metallic inclusions in ceramics and

cracks that are parallel to the direction of the X-ray beam. Tight cracks that are perpendicular to the X-ray beam are difficult to detect. Therefore, X-ray images must be taken from various directions relative to the sample.

Table 4. NDE methods

General NDE Approach	Specific Variant
Visual	
Dye Penetrant	<ul style="list-style-type: none"> * Visible dye * Fluorescent dye * Fluorescent dye with magnification
X-ray Radiography	<ul style="list-style-type: none"> * Conventional film radiography * Microfocus radiography * Synchrotron radiation source * Digitizing and enhancement from film * Scintillation detectors * Amorphous silicon detectors * Computed Tomography (CT)
Ultrasonic	<ul style="list-style-type: none"> * Standard low frequency * High frequency * C-scan * Fluid coupled versus air coupled * Pulse-echo versus through transmission * Surface wave * Acoustic emissions / internal friction * Resonant inspection (RI) * Acoustic microscopy * Scanning laser acoustic microscopy
Thermal Imaging	<ul style="list-style-type: none"> * Thermal image * Diffusivity image * Photoacoustic microscopy (PAM)
Laser Scattering	<ul style="list-style-type: none"> * Surface scattering * Subsurface scattering
Others	<ul style="list-style-type: none"> * Microwave * MRI * Neutron Radiography * Eddy Current

Fig. 3. Schematic illustrating conventional film X-ray radiography (from D.W. Richerson, *Modern Ceramic Engineering*, Marcel Dekker, Inc., 1992, page 627).

Conventional film X-ray radiography is commonly used for structural ceramics. It provides a compromise of resolution capability and cost. Conventional X-ray radiography has a focal spot several millimeters in diameter, which limits the resolution. Microfocus X-ray radiography provides a focal spot ranging from about 15-50 μm in diameter (depending on the equipment), which improves resolution. Furthermore, because of the small focal spot, the film can be placed at a distance from the X-ray source to obtain image magnification up to about 20x. For large areas or volumes of material inspection, microfocus radiography is more expensive than conventional radiography. But if only a small high stress region of a ceramic component needs to be inspected, microfocus may be cost-effective.

Image enhancement can provide further improvement in resolution, but adds more cost. Image enhancement involves scanning the film with a TV camera, digitizing the image, and manipulating the image by computer to increase contrast.

One problem with any film method is artifacts, i.e., false images that are caused by the film, fixturing, or developing. To be sure an image is not an artifact, the part must be X-rayed a second time with different position relevant to the film and fixtures. Another way to avoid artifacts is to use a "real-time" approach such as a fluoroscope or a scintillation detector. Unfortunately, these "real-time" methods have not provided comparable resolution to film methods. However, recently a new detection system, which uses amorphous silicon to convert directly from X-ray intensity to electrical impulse, has potential to provide high resolution. This amorphous silicon detector is being evaluated at Argonne National Laboratory for industrial applications and at GE Medical for medical applications. Used in conjunction with X-ray CT, this technology might allow rapid, automated, real-time inspection of ceramic parts with digital analysis and decision making.

X-ray CT has provided some of the best NDE images of ceramic components, but has been limited to developmental efforts (in-process and post-process) because of its high cost. CT involves sequentially scanning a part with X-rays from all directions, gathering the information in digital form. The information is then analyzed by computer. By scanning from multiple directions, orientation and position of defects are much better defined than for X-ray

examinations from only one or two directions. The major reasons for the limited use of CT have been (1) cost associated with the long time required to complete the scans, and (2) inferior resolution associated with scintillation detectors. The new amorphous silicon detectors have the potential to increase resolution and substantially reduce scan time. Howmet is already using CT with amorphous silicon detectors for examination of complex metallic turbine components. An automated or semi-automated system is feasible as a low-cost NDE procedure for ceramics for automotive turbines. This is one of the most promising NDE technologies and is discussed in more detail in Appendix D.

Ultrasonic NDE

A variety of techniques can be classified as "Ultrasonic." All of these evaluate the interaction of acoustic (sound) waves with the material. Most of the techniques use piezoelectric ceramic transducers to convert an electrical input into pulses of acoustic waves. As the waves travel through a ceramic sample, they interact with any discontinuity by either being scattered or reflected. The reflected and transmitted waves are detected by piezoelectric transducers, converted to electrical signals, and exhibited on an oscilloscope or other visual format. The most common technique is ultrasonic C-scan, which is illustrated in Fig. 4. By scanning the transducer back and forth, the total area of the sample can be scanned and the peak intensities printed out as various gray levels on chart paper. An example is illustrated in Fig. 5. The configuration illustrated in Fig. 4 is for through-transmission where one ultrasonic transducer emits acoustic waves and another transducer receives the waves that transmit through the ceramic part. Another option is to have the pulser and receiver on the same side of the part and is referred to as the pulse-echo technique.

Fig. 4. Schematic illustrating the basic principles of conventional ultrasonic NDE (from D.W. Richerson, *Modern Ceramic Engineering*, Marcel Dekker, Inc., 1992, page 639).

Fig. 5. Ultrasonic C-scan with a 25-MHz transducer of a 0.64-cm-thick hot pressed silicon nitride plate containing seeded defects (from D.W. Richerson, *Modern Ceramic Engineering*, Marcel Dekker, Inc., 1992, page 640).

As shown in Fig. 5, ultrasonic C-scan is effective for detection of a variety of defects in a flat plate of a ceramic. However, it is not easily applied to complex shapes. Furthermore, it does not effectively detect near-surface flaws. Another limit of ultrasonic inspection is the loss in intensity of the acoustic waves by scattering, which is referred to as attenuation. Attenuation can result from rough surfaces and from scattering by microstructural features, porosity, and inclusions. The amount of attenuation limits the thickness of part that can be inspected. It also affects the frequency that can be used. Higher frequencies can provide improved resolution, but are also more easily attenuated. Most inspection of ceramics has been in the 5-25 MHz range. Some studies showed good detection resolution (pores and inclusions in the range 10-130 μm range) with 25-45 MHz. A little research has been conducted up to 450 MHz.

Most ultrasonic inspection is conducted with the part and transducers immersed in water. The water provides a suitable coupling agent to transmit the acoustic waves to the ceramic. Some materials cannot be immersed in a liquid; so air-coupled ultrasound has been developed. This has only been achieved with low frequency (~ 400 KHz) and has only been used in a limited way for composites.

Conventional ultrasonic inspection is not effective for near-surface flaws. Some success has been achieved with "surface wave" inspection, but I am not aware of it being used for ceramic turbine component inspection. A special transducer emits longitudinal waves at a low angle to the surface of the sample. These waves stay near the surface, rather than traveling into the interior.

Another technique that can detect surface and near-surface defects is acoustic microscopy. There are several variants of this approach. One scans the surface with a focused high-frequency transducer. This is scanning acoustic microscopy (SAM). It operates at 100-400 MHz and only penetrates 1-10 μm . A more widely applicable technique is scanning laser acoustic microscopy (SLAM). A transducer, typically 10-100 MHz, is placed on one side of the sample. A plastic coverslide coated with a thin metallic mirror surface is placed on the other side. The acoustic waves that exit the sample cause a microscopic ripple pattern in the mirror. This is scanned from above 30 times per second with a helium-neon laser that accurately detects variations in the ripple pattern. This information is converted to an electrical signal and displayed on a video screen as a real-time image. Magnifications in the 10X-500X range are possible. This technique has been very successful for inspection of constant cross section parts with a very smooth or polished surface. An example is hybrid microelectronics substrates for mounting of silicon chips, which is an important technology to advanced automotive electronics systems.

The degree of attenuation of ultrasonic waves can provide substantial information about the microstructure of a ceramic material. An as fabricated, pore-free ceramic typically exhibits very little attenuation. A ceramic with porosity or microcracks exhibits higher attenuation due to scattering. This information can be obtained very quickly by pulsing the sample with a transducer and measuring the response. This technique has been successfully used to monitor onset of thermal shock damage in ceramic test bars following various degrees of severity of thermal quench. A slight variant called "acoustic emissions" has been used for real-time monitoring of damage initiation. In this case, a transducer is contacted with the ceramic through a wave guide. The ceramic is then stressed either mechanically or thermally. Any crack that forms emits an acoustic wave that is detected by the transducer.

Another bulk ultrasonic inspection technique is resonant inspection (RI), sometimes also referred to as ultrasonic spectroscopy or resonant ultrasonic spectroscopy (RUS). RI can be conducted in several different ways. One involves "pinging" the sample with a light mechanical impact and measuring the natural resonant frequencies induced in the sample. This is the approach used with Grindo-Sonic equipment. The other involves "driving" the sample sequentially with a range of frequencies and measuring the vibrational response of the sample. When the excitation frequency matches a natural frequency of the sample, the sample vibrates in that specific mode. This is the approach used with Quatrosonics and Magnaflux equipment. Its elastic properties, shape, size, density, microstructure, flaw distribution, and possibly other factors determine the resonance spectrum of a solid sample.

RI is used for inspection of production parts as a rapid discriminator of variation from a pre-determined standard. Each part is touched with a transducer and evaluated over a selected range

of frequencies. The spectrum is compared by computer with a standard and the part accepted or rejected. It is presently not known if this technique is applicable to ceramic turbine components. It has been used very effectively with ball bearings, where one peak of the spectrum discriminates between acceptable and unacceptable hardness. The inspection of each ball takes a fraction of a second and is completely automated. Kyocera tried RI on cam rollers, but was not able to define a suitable discriminator.

RI also has potential for in-service inspections of ceramic components. Cai and co-workers at ORNL demonstrated that RI could detect accumulation of creep damage in silicon nitride tensile test bars and thus could potentially be used for predicting remaining life of a component. Ferber and co-workers at ORNL demonstrated that RI could discriminate clamping loads in the attachment of ceramic rotor blades into a metal hub. This might allow monitoring service-induced increases in contact stress, which has been identified as a potential cause of blade failure.

Thermal Imaging

Several thermal-imaging approaches have been devised for NDE. One applies a heat source to one side of a part and photographs the other side with an infrared camera. Any inhomogeneity in the material that changes the conduction of heat through the part will alter the infrared image. Previously the resolution was not very high because of limitations in the infrared camera. Resolution was only about 1 mm. However, this was satisfactory to inspect ceramic-matrix composite combustor liners 8 inches long and 30 inches in diameter to look for delaminations. This work was done at Argonne National Laboratory and took about one hour for the scan. A new 12-bit camera is available that theoretically can image down to 50 μm . This system has not been evaluated for monolithic ceramic turbine components, but is not likely to be a viable candidate for high-speed inspection of complex shaped automotive turbine components.

Another thermal-imaging technique is photoacoustic microscopy (PAM). A laser beam is scanned across the sample. This produces localized heating. The heat waves travel a short distance from the point of excitation. They interact with inhomogeneities they encounter and produce a different temperature distribution than homogeneous material. By enclosing the sample in a closed gas-filled cell, a microphone can detect acoustic waves generated by the temperature differences. The technique has detected surface and subsurface features 100-150 μm long. PAM is slow and is restricted by the gas-cell-detection geometry. It has been used to aid process development of microelectronics for applications such as automotive electronics systems, but is not a likely candidate for production turbine component inspections.

Laser Scanning

Laser scattering involves scanning a laser beam (typically from a helium-neon laser) across a surface and examining the reflected light for signs of light diffusion or scattering. It is used to inspect very smooth surfaces where pores, pinholes, waviness, or protrusions would be detrimental. A good example is for examining silicon wafers and ceramic substrates for fabrication of hybrid microelectronics systems. A laser scanning system developed by ADTEC Engineering Company of Tokyo, Japan has been reported to detect cracks as small as 0.5 micron

wide and 0.7 mm long for alumina substrates at a rate of three to six seconds per piece. Alumina substrates as large as 120 mm by 120 mm by 10 mm thick have been successfully examined.

Laser scattering has also been found to work for silicon nitride, as is described in Appendix D. The technique appears able to discriminate microstructure, porosity, some individual defects, and the degree of subsurface machining damage.

Magnetic Resonance Imaging (MRI)

Nuclear Magnetic Resonance is very sensitive to the detection of hydrogen. Most binders used in the fabrication of ceramics contain hydrogen. Also many ceramics are fabricated from water-based slurries. MRI has been used to explore the uniformity of binder distribution in as-fabricated ceramic parts (prior to binder removal and sintering) and to thus provide guidance in process optimization. It has also been used to examine porosity distribution of porous samples saturated with water. MRI is not a candidate for production NDE of ceramic turbine components.

Neutron Radiography

Neutron radiography is also sensitive to the presence of hydrogen. It also has been used to explore binder distribution, but is not a candidate for final part inspection.

Microwave NDE

Some ceramics are adequately transparent to microwaves to be scanned similarly to acoustic waves. However, the resolution has not been comparable to ultrasonic scanning.

Eddy Currents

Eddy current NDE can only be applied to an electrically conductive material. Ceramics being considered for turbines are electrical insulators and thus not suitable for eddy current NDE.

Recent Technological Advances that Can Impact Ceramics NDE

Bill Ellingson of Argonne National Laboratory has pointed out in Appendix D and personal communications that recent key advances in different fields can be brought together to increase the effectiveness of NDE of ceramics and to dramatically reduce cost. These advances include "new high-power microfocus x-ray imaging systems; new high-speed digital x-ray detectors (such as the new EG&G/GE amorphous silicon detector) with high spatial resolution; high speed, high capacity, low cost computers; high definition digital video image display systems; high capacity digital image storage systems; digital image processing software for automated pattern recognition; coupling of CAD software packages with NDE data; and coupling of finite element analysis software packages with NDE data". The timing is excellent to combine these technology advances to develop an automated, fast system for NDE of ceramic turbine

components. The most promising opportunity appears to be for a CT system that can scan for defects and dimensions simultaneously.

SUMMARY AND DISCUSSION

This section first summarizes the assessment of current materials and engine technology status, NDE status, and cost issues based on the study and then discusses the relevance to ongoing and future ceramic component development.

Assessment of Current Technology Status

1. NDE and proof testing are routinely used in the automotive industry and sometimes account for over 20% of the cost of a part, although the average is much lower. The primary concern of the industry is assurance of reliability of each part to meet warranty requirements. Ceramic materials are currently used in large quantity in the automotive industry and are essentially treated in the same way as other materials, i.e., they are given the degree of inspection and testing necessary to assure reliability. For example, every ceramic oxygen sensor element is proof tested to an overstress condition; every electronic control system ceramic substrate is inspected for flatness and later for function once the electrical devices and circuits are in place; and samplings of ceramic spark plug insulators are inspected at several steps in the fabrication process.
2. The closest experience to turbine components has been the production in Japan of silicon nitride turbocharger rotors. Since 1988, well over one million of these have been placed in service and have accumulated a performance and reliability record that has fully met automotive standards. These rotors received 100% NDE and an overspeed proof test. The turbocharger rotor experience provides encouragement, but does not represent as severe a service condition as a gas turbine engine rotor. Also, further cost reduction appears necessary to meet automotive targets.
3. The ceramics companies have made major improvements in structural ceramic materials during the past 25 years. They have established quality systems that meet automotive standards. Silicon nitride materials are now available that have room temperature strength above 700 MPa, 1300° C strength above 550 MPa, Weibull modulus above 20, toughness above 6 MPa·m^{1/2}, and stress rupture life far exceeding that of metals. These silicon nitride materials have been evaluated successfully in component proof tests, rig tests, and engine tests at companies such as Allison, AlliedSignal Engines, and Solar Turbines. Current grades of silicon nitride have demonstrated large margin when tested to failure in proof test rigs. For example, rotor blades at Solar Turbines all survived room temperature spin testing to over 180% of design rotational stress conditions. Components also have survived normal engine operation conditions.
4. In spite of the successes, there also have been failures. These failures have usually been the result of an abnormal stress that substantially exceeds the normal engine operation stress, specifically foreign object damage (FOD) and biaxial interface contact stress. Calculations indicate that these abnormal stresses are so high that NDE and proof testing cannot assure reliability. The only solution is to achieve an engine and component design that either eliminates

or tolerates these abnormal stresses. For example, Rolls-Royce Allison had a problem of FOD on rotor blades caused by carbon lumps that built up in the combustion system. They redesigned the rotor to increase the mass of the blades. These thicker, "ruggedized" blades were able to tolerate the impact of the carbon lumps. In contrast, Solar Turbines had a failure in their much larger Centaur engine, which they believe was caused by a metal locating pin impacting the first stage ceramic blades. Solar concluded that "ruggedized" ceramic blades would not have survived that type of FOD incident.

5. The primary message of items 1-4 is that ceramic materials processing and NDE/proof test procedures are capable of producing ceramic components that can perform reliably under normal gas turbine engine duty (including start-up, steady-state operation, and even emergency shutdowns), but cannot guarantee survival under abnormal conditions such as FOD. Several solutions have been suggested: (1) redesign the ceramic components, especially the rotor, to be more rugged, (2) redesign the engines to minimize carbon buildup in the engine and to screen the inlet to not allow foreign objects to enter the turbine section, (3) for multi-stage turbines, use ceramics only in later stages where they will be less susceptible to impact, and (4) increase the fracture toughness of the ceramics. These potential solutions are addressed in subsequent paragraphs.

6. Ruggedizing ceramic components requires a complete aerothermal redesign to minimize reduction in engine performance. For some applications, the resulting performance may not be high enough to justify the use of ceramics. However, in the studies conducted at Rolls-Royce Allison on an axial rotor under a DOE program, the increased temperature capability and dramatic improvement in FOD resistance clearly outweighed the slight reduction in aerodynamic performance resulting from the ruggedized airfoil design. Similar results have also been reported from AlliedSignal Engines and from companies in Japan. Ruggedizing appears to be a viable option.

7. Most engines already have some type of inlet screens to prevent the engine from ingesting debris. The degree to which the inlet can be screened varies for different engines depending on factors such as the amount of flow restriction that can be allowed, the availability of space in the engine compartment, allowable power to weight ratio, and cost. Furthermore, external screens do not protect against parts within the engine coming loose and going through the turbine, or against other modes of failure such as biaxial contact stress. Other design modifications must address these.

8. The first stage rotor seems to absorb most of the energy of FOD. Metal rotors have demonstrated ability to survive FOD and continue operating with minimal performance degradation. Perhaps in some applications, benefits could be derived from a design with a cooled metal first stage rotor and ceramics in later stages.

9. The fracture toughness of silicon nitride has been roughly doubled during the past 15 years. Further increases in toughness are possible, but we may have reached the point of diminishing returns. The increases in toughness have been achieved by control of the microstructure, specifically optimizing the formation of an intertwined elongated grain structure during the

densification process. This has involved extensive R&D and tradeoffs in composition, nucleation and growth of grains, and other factors. Higher toughness has been demonstrated, but at the sacrifice of other key properties such as creep resistance or stress rupture life. Further toughness increases without other detrimental property decreases are possible, but will require long-term, focused R&D. Ceramic matrix composites and graded structures represent other approaches that might provide increases in the overall fracture toughness of a turbine component.

10. The conclusion from the above discussions is that ceramic material process control and NDE/proof testing alone will not assure reliability in an automotive or other turbine application. Design must also contribute to the reliability. In my opinion, a reliable design for an automotive turbine has not yet been achieved and demonstrated in the U.S. Future efforts should be conducted on a concurrent engineering basis with the engine companies and the ceramics companies jointly exploring tradeoffs of design, performance, fabrication, NDE/proof testing, and cost.

Assessment of NDE and Cost Issues

1. Cost is also closely linked to reliability and design. Presently, a significant portion of cost of experimental ceramic turbine components is inspection to assure that no defective parts are delivered. This includes extensive in-process measurements and 100% post-process NDE and proof testing. Furthermore, rejects may be synthetically high due to an inadequate database to clearly define accept-reject criteria. Another significant portion of cost is the small quantity of component fabrication, resulting in being at a low level on the manufacturing learning curve. Every manufacturing study that has been conducted on any material has shown that costs decrease dramatically as the quantity of components manufactured is doubled, tripled, quadrupled, etc. Many follow the 80% rule that states that each doubling in production results in an 80% reduction in cost. This is a result of improved understanding of the process, refinements in tooling, experienced workforce, quantity purchasing, incorporation of automation and semi-automation into the process, and sometimes fabricating closer to net shape to minimize expensive machining. Also, as the process becomes more mature, less key measurements are required to assure quality. This might include sampling rather than 100% inspections, or elimination of NDE because a simple proof test satisfactorily assures reliability.

2. Each case where silicon nitride has entered production, costs have dramatically decreased as volume of production has increased. A good example is bearings. Silicon nitride bearings were introduced by Norton Company (Cerbec) in about 1990. A half-inch ball sold for about \$50. This was reduced to about \$16 by 1995 and to about \$7 by 1997. Norton now is producing about 25 million balls per year in all size ranges at an average cost of less than \$2 per ball. Similar cost reductions are starting to show up for turbine components. AlliedSignal Ceramic Components scaled up to only about 100 nozzles per month for the Model 85 APU field-testing and was able to decrease costs by over 75%. Kyocera has demonstrated similar decreases in cost for their turbocharger rotors and cam follower rollers as the quantity of production increased.

3. Many different NDE techniques have been tried with ceramic materials, but there is still no significant database to clearly relate the results to failure causing flaw populations in the

ceramics or to component survivability. As a result, present accept-reject criteria are poorly defined and validated.

4. FPI, visual, and conventional X-ray radiography are used currently for NDE of ceramic turbine components, usually on a 100% basis. Ultrasonic scans are used in some cases to inspect ceramic-metal joints. Ultrasonics is also used for ceramic matrix composites, as is thermal imaging. CT has been used effectively for process development, and new detector technology should allow CT to be a candidate for production inspection of complex-shaped turbine components. There is still a need for an inspection technique for detecting near-surface defects resulting from machining. Laser scattering has potential. RI has potential as a rapid production procedure for quickly screening components and may be a way of sorting so that some components go directly to a proof test and others receive a selected intermediate NDE inspection. RI also has potential for in-service inspections to help assess the remaining life of a part.

5. Production-viable dimensional inspection is at an early stage of development for ceramic turbine components and has room for substantial innovation, improvement, and cost reduction. The requirement of increased quantity of ceramic parts for recent field test efforts has provided good experience and database for physical and optical dimensional inspection procedures. The resulting information should be useful in predicting and planning needs for automotive or other turbine applications. The new CT technology described in Appendix D sounds very promising as a low-cost alternative to the current labor-intensive dimensional inspection techniques. This new technology could allow simultaneous automated defect detection and dimensional inspection.

6. NDE is presently important in the development of ceramics for automotive electronics systems and for quality assurance. Commercial NDE systems exist for laser scanning to examine electrical substrates for cracks; Moiré fringe patterns to test substrates for flatness; acoustic microscopy to find debonds in surface mount devices; three-dimensional measurements of laser trimmed resistors on ceramic substrates using a phase-shift-technology-based surface profiler; and an automated, real-time, X-ray radiography system to find shorts, opens, voids, misalignment, and missing balls in ball grid arrays.

Revised Status of Ceramic Turbine Component Development

The automotive companies and DOE have made important decisions during the past year. The automotive companies under the PNGV program have chosen not to pursue the gas turbine for the next generation of automotive propulsion engines. This decision was based on several factors: (1) Other concepts closer to current automotive engine design practice and production meet near-term performance goals and represent less risk and cost in getting to the marketplace, (2) To have acceptable performance, an automotive turbine will require ceramics, and neither the turbine engine designs or ceramics materials are perceived as far enough along, and (3) Cost projections based on current technology are too high for the turbine. Even though the turbine is not the optimum approach for near-term automotive application, it still is a viable candidate for longer-term U.S. performance goals.

Based on the above decision, DOE has shifted their near-term efforts on demonstration of ceramics for small turbine engine technology to the Office of Industrial Technologies (OIT). Efforts will be focused initially on microturbines (turbogenerators) for small industrial distributed power. The technology developed will be applicable later to a high-efficiency, multi-fuel-capable automotive turbine.

RECOMMENDATIONS

The following recommendations suggest work that needs to be conducted to address the issues of design, ceramic component inspection, and demonstration of reliability.

1. Use the shift of focus from automotive turbines to microturbines as an opportunity to prepare a master plan that is based on concurrent engineering. Specifically include fabricability, inspectability, cost, and reliability issues as integral parts of the design assessment to come up with a program plan that addresses these issues. For example, include within the program fabrication of ceramic parts designated for use in NDE development. Furthermore, plan for a portion of these parts to be tested to failure to build a database to correlate flaws/inspection/performance to begin establishing accept-reject criteria.
2. Focused effort is needed to demonstrate production-viable NDE and proof test systems for ceramic turbine components, especially for near-net-shape fabricated parts. Based on Kyocera turbocharger rotor experience, it appears that automated or semi-automated systems are feasible and can cost-effectively meet the inspection needs of automotive and other turbine components. Specifically, establish a program to evaluate the amorphous silicon detectors with CT to determine if an automated or semi-automated system, which incorporates computer decision making, is feasible. The program could be linked with component fabrication development such as the AlliedSignal rotor fabrication effort funded by DOE.
3. Establish a working team to pull existing data together and to plan future data acquisition/gathering in an effort to correlate flaws/inspection/performance of ceramic components. This team should be made up of a combination of individuals from the ceramic companies, the engine companies, and national laboratories. The team should prepare a master plan, which includes specific strategies and action items for (1) tying together and jointly analyzing prior data, (2) coordinating current program NDE activities and identifying needs, and (3) establishing cooperative programs to meet the needs not addressed in prior or current programs. A particularly key activity of the team might be to define a plan for achieving a database of overspeed proof test failures on the AlliedSignal rotors (and/or other turbine components) linked to pre-test NDE and post-test analysis.
4. Establish a program to gather a database of RI spectra for a statistically significant number of various turbine component configurations and test samples. Repeat the measurements after proof testing, rig testing, and engine testing. Analyze the data to determine if RI has potential as a screening tool, or for accept-reject decisions.

5. Review present procedures for dimensional inspection and identify needs to achieve production-viable, cost-effective systems for turbine components. This might also be addressed by the team listed in item 3.

ACKNOWLEDGEMENTS

The author thanks the Department of Energy for funding this study, members of the Ceramics Division of USAMP (Gary Crosbie, George DeBell, Bob Herbon, Fred Kennard, and Bob Powell) for their guidance and discussions, representatives of the automotive companies (especially Keith Carson) for their input on current NDE and other quality procedures in the automotive industry, representatives of AlliedSignal Ceramic Components and Kyocera Industrial Ceramics for their input on turbine component fabrication and NDE needs, Bill Ellingson of Argonne National Laboratory for his input on NDE, Gary M. Crosbie of Ford Motor Company for input on NDE for automotive electronics components, Dave Stinton of ORNL for his guidance and help in planning this study and in publication of the final report, and especially Bob Powell for his leadership through USAMP and help in obtaining information from the automotive companies.

Ceramics for turbine engines

Ceramic materials for gas-turbine engines have been in development for decades, but only now are those efforts showing signs of success. **By David W. Richerson**

SINCE THE MID-1940s, researchers have envisioned ceramic materials as a way to improve the performance of gas-turbine engines, lengthen their life span, and reduce their fuel consumption substantially. Yet ceramics are just now approaching their first commercial use in turbines.

The challenges involved have been considerable. The material in modern turbines must survive temperatures of more than 1,100°C for thousands of hours; high thermal stresses caused by rapid temperature changes and large temperature gradients; high mechanical stresses; isolated impact and contact stresses; low- and high-frequency vibrational loading; chemical reactions with adjacent components; oxidation; corrosion; and time- and stress-dependent effects such as creep, stress rupture, and cyclic fatigue. Early ceramic materials were not able to withstand these conditions, and early turbine-component designs were not compatible with brittle materials. Technological evolution has to be made over a broad front, and progress has been slow.

MATERIAL COMPOSITION

A variety of oxides, borides, carbides, and cermets were evaluated in the 1940s and 1950s for potential use as turbine components. Some ceramics had favorable strength and oxidation resistance, but none survived the thermal shock conditions imposed by an engine. Some cermets could survive thermal shock and impact conditions but did not have adequate oxidation resistance and stress rupture life.

Interest was renewed in ceramics for turbines when new materials in the silicon nitride and silicon carbide families of ceramics were developed during the 1960s.

David W. Richerson is president of Richerson and Associates in Salt Lake City.

These materials had better thermal shock resistance, largely due to a combination of low thermal expansion, high strength, and moderate thermal conductivity.

The first promising silicon nitride and silicon carbide materials were fabricated in Great Britain by reaction sintering. The silicon nitride was prepared by a reaction of a powder compact of silicon with nitrogen to form silicon nitride. This resulted in a reaction-bonded silicon nitride (RBSN) material, typically with room-temperature strength approaching 140 megapascals. The strength was retained to at least 1,400°C, but the material weakened over time when exposed at high temperature to an oxidizing atmosphere. The silicon carbide was prepared by reacting a mixed powder compact of silicon carbide plus carbon with molten silicon to form an SiC-bonded silicon carbide, with the pores filled with silicon. Early reaction-sintered silicon carbide materials had strength that was similar to RBSN and superior oxidation resistance.

A higher-strength pore-free silicon nitride was achieved in Britain in the late 1960s by hot pressing. This involved heating a mixture of silicon nitride powder plus a low percentage of oxides, such as calcium oxide or magnesium oxide, in a graphite die to about 1,700°C while applying a uniaxial pressure of about 13.8 to 34.5 megapascals. Room-temperature strength greater than 700 megapascals in three-point bending was demonstrated. However, additives to allow densification concentrated at the grain boundaries as a glass composition. This composition softened when the ceramic was heated to temperatures approaching 1,000°C, resulting in grain-boundary sliding when a stress was applied. This decreased strength.

Major efforts have been conducted worldwide since the early 1970s to improve the high-temperature properties of silicon nitride. Some have focused on finding a composition with a higher-temperature intergranular

glass phase; others have focused on compositions that can be heat-treated to crystallize the grain-boundary phase and avoid the glass phase. Only recently have the properties been adequate to consider long-life applications.

Ceramic turbine components are fabricated starting with powders of the raw materials. The quality of the final part depends on the quality of the starting powder and on each step in the fabrication process. Early powders were coarse and contained impurities, and they were not widely available until the mid-1970s. Around that time, researchers demonstrated that silicon nitride and silicon carbide could be densified by pressureless sintering if the starting powder was of very small particle size. Powder synthesis techniques were refined during the 1980s, making powders with a smaller particle size (sub-microns) and relatively high purity available.

Techniques to measure properties were critical to the evolution of ceramic materials for turbines. They provided both a means of comparing materials and the database needed for probabilistic design, and were an important part of quality control.

Initially, strength testing was conducted using a three-point bending procedure, with each company using a different test-bar size and fixture design. The data were impossible to compare, and a very small volume of material was exposed to the tensile load. Sometime around 1972, attempts were initiated to select a standard test-sample size and shape as well as conduct tests with four-point bending. More sample volume is exposed to tensile stress under four-point bending, so a reasonable tensile distribution can be established with 20 or 30 test bars. Four-point bend testing proved adequate to compare materials and to design small turbine components with localized regions of high stress, such as a turbine rotor blade inserted into a metal hub.

When components were designed with a larger volume under tensile stress, such as one-piece radial ceramic rotors, emphasis began to shift to tensile testing to simulate better the larger volume under stress and the increase in the proportion of internal flaws to surface flaws. Tensile testing is also important to gain an understanding of creep, stress-rupture life (slow crack growth), and cyclic fatigue, as well as to establish models for life prediction.

Earlier efforts at material characterization and database generation have continued under a project funded by the U.S. Department of Energy (DOE) through Oak Ridge National Laboratory in Oak Ridge, Tenn. The project addresses test standardization, a database for fast fracture and time-dependent fracture measured in uniaxial tension, correlations with nondestructive-evaluation (NDE)

techniques and life-prediction methods, and iterative increases in material and component reliability. Statistical tensile databases now exist for all key silicon nitride and silicon carbide turbine materials from room temperature to at least 1,370°C, including some stress-rupture and creep tests lasting more than 10,000 hours. A parallel program at Garrett Turbine Engine Co. in Phoenix—now part of AlliedSignal—has established a flexural-strength database on the same materials exposed for up to 3,500 hours in a dynamic test rig cycling between a diesel-fired burner and an air-blast quench.

THE FABRICATION PROCESS

One of the biggest challenges has been fabricating to achieve reliable properties in the required complex shape at acceptable cost. The primary focus has been on developing near-net-shape fabrication processes that can produce complex turbine-component shapes with a minimum of machining, and on optimizing and controlling each step in the fabrication process to minimize the size of microstructural flaws to within design limits.

Turbine programs in the 1970s had to rely on reaction-bonded and hot-pressed materials. Ford Motor Co. in Dearborn, Mich., conducted extensive development of injection molding. The automaker succeeded in injection-molding one-piece

stator-vane rings and rotor-blade rings.

Sintered silicon nitride and silicon carbide materials were developed in the late 1970s and became the primary candidates for turbine programs throughout the 1980s. These materials had intermediate strengths, between reaction-bonded and hot-pressed materials, but they had the potential to be fabricated to near-net shape at costs competitive with metal turbine components. Most of the effort focused on injection-molding and slip-casting radial turbine rotors, scrolls, and other turbine components. Given the properties of materials at that time, a microstructural flaw such as a pore, crack, or inclusion roughly 150 microns across in the high-stress region of the rotor was a critical flaw and would cause immediate failure. Early sintered materials had flaws substantially larger than this. Extensive development was required (and is still continuing) to fabricate turbine components free of these critical flaws.

The emphasis then shifted to hot isostatic pressing (HIP), either to achieve additional densification of a sintered part or to directly densify a powder compact encapsulated in a glass envelope. HIP achieved properties similar to uniaxial hot pressing but allowed complex shape fabrication. Turbine rotors and other components densified by HIP successfully operated at design condi-

The Allison AGT-5 engine is one components test bed that was used to develop ceramics for automotive applications.

tions in experimental turbines. HIP is expensive, however, and results in less strength near the surface than in the interior of the part.

Research during the 1990s has been directed toward improving the properties of sintered materials to minimize flaw size and refining the microstructure to increase fracture toughness. Higher fracture toughness means a larger critical flaw size for a given stress. Whereas the early materials had a critical flaw size around 150 microns for a 200-megapascal stress, the improved materials can withstand flaws several times larger.

Improvement in the Weibull modulus of the materials is a good gauge of progress. In 1980, the typical Weibull modulus of candidate turbine ceramics ranged from about 5 up to 8, with occasional values of 8 to 12. Now most of the materials have a Weibull modulus consistently above 20.

Fracture of a ceramic part in a turbine is likely to lead to fracture of adjacent ceramic parts and complete engine failure. Quality assurance must be rigorous enough to eliminate any ceramic parts with critical defects. This was a very difficult challenge when the critical defect size was about 75 to 150 microns. Materials with increased fracture toughness and improvements in processing have eased the challenge. In addition, NDE and proof-test procedures have improved, but these procedures are expensive.

Methods and tools for designing ceramic components have advanced dramatically since the 1960s. The field of probabilistic design for ceramics has been developed and validated in engine testing. The turbine-engine companies have established refined models and codes for preliminary design, detailed design, and life prediction. The first probabilistic life-prediction codes were based only on fast-fracture criteria, but improved databases have allowed time-dependent models to be incorporated in life-prediction codes.

ENGINE DEMONSTRATIONS

The first key program in the United States was the Brittle Material Design-High Temperature Turbine Program, initiated in 1971 by the Defense Advanced Research Projects Agency (DARPA) at Ford and Westinghouse Corp. in Pittsburgh. The program focused on demonstrating that ceramic components could be successfully designed using finite-element-analysis codes and could survive under severe engine-operating conditions. Ford's portion of the program culminated in a successful 25-hour test at 50,000 rpm and 1,200°C to 1,250°C of a single-shaft engine with all hot-section components (rotors, stators, combustor, casing, and two honeycomb rotary regenerators) fabricated from ceramics.

The Ford program was a pioneering effort, demonstrat-

The Garrett/Ford AGT 101 engine included ceramic components such as the radial rotor, the rotary regenerator, and even the attachment bolts.

ing that brittle ceramics could be designed with a probabilistic FEA approach and could survive the conditions of engine operation. However, the materials available at the time had marginal properties, and other issues such as contact stress, durability, reliability and cost-effective fabrication needed further attention.

The program at Ford did not focus on demonstrating increased performance. DARPA initiated a program in 1976 with AiResearch Manufacturing Co. in Phoenix—now AlliedSignal Engines—to retrofit ceramics into an existing turboprop engine, with the goal of demonstrating a 40-percent increase in power output and a 10-percent decrease in fuel consumption.

To achieve the goals, the turbine-inlet temperature (TIT) had to be increased from about 1,000°C to 1,200°C.

This was achieved by replacing the first two turbine stages with ceramics. The resulting design consisted of 104 ceramic parts. RBSN was selected for the stator vanes, shrouds, transition liners, and support structures. The rotors were a hybrid design that comprised Norton NC-132 hot-pressed silicon nitride blades with a single tang dovetail-inserted into a metallic disk. The blades and disk were separated by a thin metallic compliant layer. Performance improvement of 30 percent and fuel-consumption reduction of 7 percent were demonstrated in comparison with the baseline metallic engine.

An original goal of the program was to achieve 50 hours of successful engine operation. The team believed that this required a complete redesign of the static structure to avoid the source of biaxial contact stress, which had caused failures during engine testing. A U.S. Air Force program allowed redesign of the engine to avoid the contact-stress problems in the static structure. The structure was tested successfully in rigs and the rotor blades were validated by spin testing.

All engine testing in the Ford and AiResearch programs was conducted on test stands. The results were positive, but the question remained whether the ceramic components would be durable in an engine in an actual vehicle, especially a land-based vehicle subject to severe shock loading. DOE initiated a program in the mid-1970s at the Detroit Diesel Allison Division of General Motors Corp. in Detroit to design ceramic components into the Allison GT 404-4 truck engine. Testing included powering a truck on highways, city roads, and the GM proving grounds, where the engine was exposed to extreme vibrational and shock loading on the Belgian-block and truck-durability road courses. Testing clearly demonstrated that properly designed ceramic components could survive under the most severe conditions for a typical vehicle.

SYSTEM-DEVELOPMENT PROGRAMS

To take advantage of the higher temperature capability of ceramics, DOE funded programs from 1979 to 1987 to demonstrate proof of concept for a ceramic-based auto-

motive gas-turbine engine that could power a midsize automobile over a standard federal combined driving cycle (city and open road) at 42.8 miles per gallon. The Advanced Gas Turbine (AGT) Program included one program at Detroit Diesel and another at Garrett.

The Allison engine (AGT 100) was relatively conservative in design. It had a shaft for the gas-generator turbine and a shaft for the power turbine. This split the work and minimized the TIT (1,285°C maximum) and engine speed (85,000 rpm) required to achieve the mileage goal. However, the design required two radial ceramic turbine rotors and a complex shaped scroll (to transition the hot gases from the combustor to the nozzle guide vanes).

The Garrett engine (AGT 101) was an extension of the Ford 820 ceramic engine design. It had a single shaft, a radial rotor, and air bearings. It required a design speed of 100,000 rpm and a TIT of 1,370°C to meet the program goals. The AGT 101 design had the advantages of being symmetrical and simpler (fewer ceramic parts) than the AGT 100, but it posed a greater challenge to ceramic-materials technology because of the higher speed and temperature. For both projects, the TIT and the resulting engine performance remained short of program goals.

Many ceramic components fractured during testing, some due to design and assembly problems but most from material limitations. The desire was to use net-shape fabrication processes, but the state of technology in 1985 did not result in reliable ceramic turbine components.

The AGT program focus was changed to improving the materials processing of components, emphasizing use of the AGT rigs and engines as test beds to guide ceramic-component development. Substantial improvements in shape forming, properties, and reliability were achieved between about 1985 and 1993.

DOE automotive programs began to change in the early 1990s. Cost, producibility, and durability became key issues. Gas-mileage goals were increased to 80 miles per gallon; hybrid propulsion-system concepts became popular.

Allison, which had replaced its AGT 100 configuration with an axial turbine configuration designated AGT-5, modified its program to explore ceramic readiness for a hybrid turbine-electric concept. Several ceramic-component and engine failures have led to design or material modifications. The program recently achieved a 300-hour engine test with no sign of distress to any ceramic components.

Garrett discontinued the AGT 101 engine and switched to the AlliedSignal 331-200 auxiliary power engine, which was a better test bed for accumulating engine test time and field testing. More than 1,400 hours of engine testing have been performed with silicon nitride inlet guide nozzles, including more than 300

hours at design speed with a ceramic-bladed rotor.

AlliedSignal Engines, under a DARPA insertion program, is already conducting field tests of silicon nitride ceramic nozzles in its Model 85 auxiliary power unit. By April, more than 46,000 hours of engine testing had been successfully completed, including more than 7,500 hours on one engine. The primary concern has been cost, but AlliedSignal Ceramic Components has demonstrated 76-percent cost reduction over roughly the past year and predicts additional cost reduction.

A program at Solar Turbines Inc. in San Diego has reached the field-testing stage with ceramic components. DOE initiated this program in 1992 with the objective of retrofitting a Solar Centaur 50 industrial turbine engine with a ceramic combustor liner plus

first-stage nozzles and blades. A major challenge of this program was to achieve design stresses that were low enough to enable the ceramic to survive for 30,000 hours.

A series of full-scale engine tests have been conducted at Solar. Silicon nitride rotor blades from AlliedSignal Ceramic Components and silicon carbide composite combustor liners from DuPont Lanxide Composites in Newark, Del., have been qualified in these tests, and are

currently in a field test at an ARCO Western Energy oil field. As of this June, the engine had operated for more than 700 hours with no problems.

The recent field tests and other engine tests are encouraging. They indicate that the candidate ceramics, the component and engine designs, the manufacturing processes, and the life-prediction methods have simultaneously reached a level of maturity consistent with turbine-application needs. Concerns still exist, however, such as with contact stress as well as with foreign object damage and with cost.

Some significant successes already have been demonstrated in spin-off applications. Silicon nitride has been in high-volume production for high-speed cutting-tool inserts for machining cast iron and superalloys since the late 1970s. Silicon nitride turbocharger rotors have been used in Japan since the late 1980s, with no reported incidence of failure. Silicon nitride cam-follower rollers are in production at Detroit Diesel, and silicon nitride bearings are in production at St. Gobain Norton Industrial Ceramics. Silicon nitride seal runners have recently entered production for AlliedSignal propulsion engines for business aircraft.

Silicon nitride is also used in the paper industry, in sand-blast nozzles, and in many other commercial applications. Many applications also exist for silicon carbide materials developed primarily for turbine-engine programs. The substantial investments in advanced structural ceramics are clearly starting to pay off. ■

The Solar Centaur engine (shown with a schematic of its hot section) is retrofitted with ceramic components.

APPENDIX B Questions Posed to Automotive Representatives

David W. Richerson
2093 E. Delmont Drive
Salt Lake City, Utah 84117
Phone: 801-272-0436
Fax: 801-272-2423

May 28, 1997

Ref: 97026

Keith Carson
Ford Motor Company
Fax: 313-322-3763

Dear Keith:

I thought it might be useful for you to have something in writing. Here is a list of questions that have been running through my mind.

1. What are typical requirements and/or standard practices for inspection in the automotive industry to verify functionality? Durability/reliability/warranty life? Dimensional? Other factors? Let's consider each one separately.
2. Functionality
 - a. What parts currently require functional measurements or demonstrations as part of the quality assurance before an auto company will accept the part? Would any of these provide guidance to us regarding future requirements for turbine ceramics?
 - b. Are all spark plugs checked for electrical continuity and output?
 - c. Are oxygen sensors checked for functionality?
 - d. Is inspection 100% or partial?
 - e. Are there functionality checks specified for drivetrain components?
Does the inspection change if a new material or design is implemented?
Is there a transition period resulting in decreased inspection requirement as production experience and field test experience is accumulated?
Or do cost criteria demand that functional inspection be fully integrated at a production-capable level before the auto company will implement use of the part?
Or is the specific part first introduced in a luxury vehicle where a higher cost and lower quantity of production can allow more flexibility?
What scenario would most likely be the case for implementation of a turbine engine with ceramic components?
 - f. What functionality-based inspections might be required for ceramics?
 - g. Are any components currently proof-tested?

3. Durability/Reliability/Warranty life
 - a. Are there any specifications for inspection that relate to durability/reliability/warranty life?
If so, for what type of components?
How are these implemented?
How is the cost absorbed?
 - b. Will standards and quality assurance testing be required to verify durability/reliability/warranty life for ceramic turbine components?
 - c. Are there any existing cases that might be similar to that expected for ceramic turbine components?
 - d. Do any components currently have testing to verify resistance to shock loading, such as electronic control systems? Might this be required for any ceramic components?
 - e. Are any components currently proof-tested?
4. Dimensional
 - a. What current dimensional inspection criteria and technology are applicable to future needs with ceramic turbine components?
 - b. Are there any that are particularly relevant that should be discussed in detail? Are there individuals that I should meet with or that should attend our meeting?
5. Others
 - a. Are there other areas of inspection that we need to consider and discuss?
6. What is the role of nondestructive evaluation (NDE) for current automotive components?
7. Where can the answers to the above questions guide us in evaluating the feasibility of structural ceramics and defining inspection needs consistent with automotive requirements?

I hope these questions will provide you some guidance. Perhaps they can also be an outline for our discussions on June 10. The questions may also help you decide what other people you might want present in our meeting.

Sincerely,

David W. Richerson

Cc: Sue Hartfield-Wunsch
Bob Powell
Dave Stinton
George DeBell
Fred Kennard

APPENDIX C Questions Posed to Ceramics Companies

1. Assessment based on technical and cost criteria of the potential of current or projected MDE methods for use in qualifying ceramic turbine components for automotive use.
2. Assessment of alternative to NDE such as proof testing or SPC.
3. Assessment of the needs and state-of-the-art for dimensional inspection.
4. Above assessments regarding (1) verification of functional capability, (2) assurance of reliability/durability/warranty life.
5. Assessment of where NDE might be an important tool during development.
6. Recommendations for developments of inspection techniques that need to be established in parallel to ceramic manufacturing development programs.
7. Recommendations of how ceramic components and test samples fabricated under the manufacturing development programs can be used to (1) evaluate NDE and/or dimensional inspection techniques, (2) support development of improved NDE or dimensional inspection techniques, (3) establish a database correlating NDE/proof-test results with performance/survival.
8. Preparation of a "strawman" road map.

APPENDIX D Supplementary Report – Nondestructive Evaluation Technologies for Automotive Ceramic Applications

Submitted by: William A. Ellingson
Argonne National Laboratory
Argonne, Illinois

NONDESTRUCTIVE EVALUATION TECHNOLOGIES FOR AUTOMOTIVE CERAMIC APPLICATIONS

PURPOSE

It is the purpose of this supplemental report to provide information on the status of specific nondestructive evaluation (NDE) technologies that are likely to have application to ceramic components which may be used in automotive applications (or applications similar to automotive). It will not be the purpose of this report to review the status of the NDE technologies relative to application in the various stages of ceramic processing. Rather, the purpose of this report is to focus on current technologies, which are being used or can be used on densified components.

There are several publications that discuss the application of NDE in the various steps of ceramic processing.¹⁻⁴

BACKGROUND

NDE technological advances are heavily impacted by digital computing advances and advanced sensor developments. Thus, these technologies directly impact the status of NDE. With this in mind, this background section will be discussed in two areas: (1) brief, broad statements about overall past ceramic processing developments and (2) recent developments in technologies that will impact NDE methodologies.

(1) Broad Statements

The US Department of Energy (DOE) has had a significant program in place for a number of years to develop improved monolithic ceramic materials.⁸ As a part of that research, there have been efforts to develop NDE technologies for application to various steps of ceramic processing with the intent of impacting the reliability of use of these materials. While these efforts have focused mainly on Si_3N_4 materials, much of the NDE technology is quite generic requiring only minor modification for application to other ceramic materials such as SiC .

(2) New Technological Advances

In this section, four (4) NDE modalities will be discussed along with information that impacts their application to ceramics for automotive uses. These four technologies are:

- (a) Resonant Ultrasonic Inspection
- (b) Fluorescent Penetrants
- (c) 3D X-ray Computed Tomography (CAT Scan Imaging)
- (d) Elastic Optical Scattering

(a) Resonant Ultrasonic Inspection

Resonant Ultrasonic Inspection, also called Resonant Ultrasonic Spectroscopy (RUS), is commonly referred to as the "ping" test by ceramists. Fundamentally, the "ping" test refers to the process of lightly impacting (or tapping) a ceramic and "listening" to the resulting sound emitted. A schematic diagram of a "ping" test setup is shown in Fig. 1.

Fig. 1. Schematic diagram of a basic "ping" test setup for Resonant Ultrasonic Spectroscopy.

In Fig. 1, the instrumented impact hammer impacts the ceramic test specimen and a high-sensitivity microphone picks up the resulting sound. Both the signal from the instrumented impact hammer and the signal from the microphone are digitized by a high-speed digitizing system. Using advanced, digital-signal-processing technology, these data allow calculation of the bulk elastic modulus as well as the specific damping capacity. These two parameters allow

separation of "good" from "bad" components if a sufficient database exists from which the "good" and "bad" components can be selected. Recent work funded by the Department of Defense (DoD) through the Defense Advanced Research Projects Agency (DARPA) has shown that this technology can be used reliably to sort ceramic ball bearings.¹⁴ A limitation of this method is that it can be used for sorting "good" from "bad," but allows little detail to be established about what caused the part to be "bad."

(b) Fluorescent Penetrants

There are several liquid-penetrant methods and procedures that have been developed over the years but only fluorescent penetrants, that is, those penetrants that require ultraviolet (UV) light for activation, are applicable to ceramics because of the tight cracks involved. Recent advances in this technology primarily focus on the use of magnified imaging at 50-100 X for defect detection, and use of digital-imaging technology for important data archival.¹⁶ This technology is quite inexpensive to implement and is being routinely used today for ceramic applications.

(c) 3D X-ray Computed Tomographic Imaging

X-ray computed tomographic imaging is often referred to as CAT scanning in the popular press because of the familiarity with medical applications.¹⁷ Most medical CAT scanners provide so called 2-dimensional (2D) images or individual "slice" images of the human body. These images are familiar to most people because they are seen frequently in the newspaper or on TV. Three-dimensional (3D) CAT scan imaging, often also called cone-beam tomography or volumetric tomography, differs from 2D imaging in that the X-ray image data acquired allow the image to be directly presented on video screens in 3D representation. What makes this possible is the use of a 2D detector that is a plane rather than a single-line detector. A schematic diagram of a 3D-detector system is shown in Fig 2.

Drawbacks associated with CAT scan technology as an NDE method are the cost and limited throughput of such systems. This is, in general, a true assessment. However, new, 2-dimensional X-ray detector technology and advanced computers have the potential of making CT scanning a viable alternative. EG&G Amorphous Silicon in Santa Clara, California, for example, recently announced the beginning of a commercialization activity for a new, amorphous silicon detector. These detectors allow images to be acquired in less than 60 seconds with near-real-time image reconstruction, allowing 3-dimensional scans of ceramic objects to be completed in the 60 seconds it took for the data acquisition. Such technology is now under evaluation for automotive ceramics at Argonne National Laboratory through a Cooperative Research And Development Agreement (CRADA) with EG&G. Coupled with these new, fast X-ray area detectors, the developments in fast desktop computers with parallel processing capability are making the speed of image generation from the CAT scan data nearly real time. If this can be realized in the near future, that is, within the next 24-30 months, then this X-ray imaging technology can no longer be ignored because it is too slow and cannot handle throughput.

Indeed, it will likely become a method of choice because of the ability to not only detect defects but also perform dimensional analysis on complex-shaped parts with internal structure.

Block Diagram of 3-D X-Ray CT System

Fig. 2. Schematic diagram of a 3-Dimensional X-ray "CAT" scan system showing the components of the system.

(d) Elastic Optical Scatter

Work over the past 5 years has shown that low-power, optical-wave-length lasers can be used to detect both surface and subsurface defects in structural ceramics such as Si_3N_4 and SiC .²⁰⁻²³ In this technology, see Fig. 3, a low-power laser is used together with special polarizing

Fig. 3. Schematic diagram of elastic optical scatter system used to detect surface and subsurface defects.

Optics. Both illuminate the object under investigation and detect the reflected light. Research has shown that visible light can, in fact, penetrate sintered ceramic materials to depths of several hundred microns. Figure 4 is a diagram that shows percent of light transmitted by several ceramic materials as a function of the thickness of the material. By careful analysis of the reflected light, that is, analysis of the polarization angles that have been preset to either emphasize surface or subsurface defects, defects can be detected and correlated with known surface and subsurface defects. The data acquired by such a laser-scattering system are displayed on a computer screen in a similar way that any other type of image is displayed. Current work has focused on detection of machining-induced damage such that the laser system could be used for an on-line, real-time process control system. While no ASTM standards have yet been developed, research is under way to establish correlation with various defects.

SUMMARY

In summary, four advanced NDE technologies have been briefly presented which offer the potential for fast, low-cost ceramic component assessment with which decisions on accept-reject could be based. With further research into these systems, correlation to remaining life may

be possible. If this situation could be fulfilled, remaining life could, for the first time, be predicted.

Fig. 4. Optical transmission of several ceramic materials.

REFERENCES

Overviews

1. R. W. McClung, "Overview of NDE Development," pp. 433-441 in Proceedings of the Annual Automotive Technology Development Contractors' Coordination Meeting, P-265, Dearborn, Mich., November 2-5, 1992, SAE, Warrendale, Pa., 1993.
2. W. A. Ellingson et al., "Nondestructive Evaluation Development for Process Control of Ceramics," pp. 433-437 in Proceedings of the Annual Automotive Technology Development Contractors' Coordination Meeting, P-256, Dearborn, Mich., October 28-31, 1991, SAE, Warrendale, Pa., 1992.
3. K. E. Amin, "Radiograph and Ultrasound Detect Defects Early," Ceram. Bull. 74(1): 76-81 (1995).

4. W. A. Ellingson et al., "Development of Nondestructive Characterization Technologies for Process Control of Ceramics," pp. 637-649 in Proc. 5th Int. Sym. on Nondestr. Char. of Mat., Karuizawa, Japan, eds. T. Kishi et al., Iketani Sci. Foundation, Japan, 1991.
5. W. A. Ellingson et al., "Nondestructive Characterization of Structural Ceramic Components," pp. 156-161 in 5th International Symposium on Ceramic Materials and Components for Engines, eds. D. S. Yan, X. R. Fu, and S. X. Shi, World Scientific, Singapore, Shanghai, China, 1994.
6. D. R. Johnson et al., "Needs Assessment for Nondestructive Testing and Materials Characterization for Improved Reliability in Structural Ceramics for Heat Engines," ORNL/TM-10354, Martin Marietta Energy Systems, Inc., Oak Ridge Natl. Lab., August 1987.
7. "New Structural Materials Technologies/Opportunities for the Use of Advanced Ceramics and Composites," US Government/Office of Technology Assessment, Technical Memorandum OTA-TM-E-32, 1986.
8. "Ceramic Technology for Advanced Heat Engines Program Plan," ORNL/TM-8896, Martin Marietta Energy Systems, Inc., Oak Ridge Natl. Lab., June 1984.
9. D. R. Johnson, "Advanced Materials Development Program Ceramic Technology for Advanced Heat Engines Program Plan 1983-1993," ORNL/M-1248, Martin Marietta Energy Systems, Inc., Oak Ridge Natl. Lab., July 1990.
9. Johnson, D. R., "Materials Development Program Ceramic Technology Project Addendum to Program Plan Cost Effective Ceramics for Heat Engines," ORNL/M-2309, Martin Marietta Energy Systems, Inc., Oak Ridge Natl. Lab., August 1992.

Acoustic Resonance

11. S. Yang et al., "Nondestructive Characterization of Thermal Cycling-Induced Microstructural Changes in Ceramics and Ceramic Matrix Composites," pp. 131-141 in NCA. Vol. 20/ AMD Vol. 211 Proceedings of Conference on Innovative Processing and Characterization of Composite Materials, Anaheim, Ca., ASME, 1995.
12. R. A. Bemis, K. Shish, and W. A. Ellingson, "Nondestructive Evaluation of Thermally Shocked Silicon Carbide by Impact-Acoustic Resonance," ASME J. of Engr. for Gas Turbines and Power, 118(3):491-494 (1996).
13. S. Yang, R. F. Gibson, and G. M. Crosbie, "Vibrational Characterization and Comparisons of Automotive Engine Valves Made from Conventional and Unconventional Materials," J. Sound Vib. 191(5):986-992 (1996).
14. J. Maynard, "Resonant Ultrasonic Spectroscopy," Physics Today, pp. 26-31, January 1996.

Penetrant

15. R. G. Sherwin and P. DuBosc, "Trends Affecting Penetrant Specifications and Standards: Beyond 2000," *Mat. Eval.*, pp. 864-870, August 1997.
16. G. A. Forster and W. A. Ellingson, "An Investigation of Penetrant Techniques for Detection of Machining-Induced Surface Breaking Cracks on Monolithic Ceramics," ORNL/Sub/93-SN893/1, Martin Marietta Energy Research Corporation, Oak Ridge Natl. Lab., February 1996.

X-ray Tomographic Imaging

17. W. A. Ellingson and M. W. Vannier, "X-Ray Computed Tomography for Nondestructive Evaluation of Advanced Structural Ceramics," Argonne National Laboratory report, ANL-87-52, Argonne National Laboratory, Argonne, Ill., 1987.
18. W. A. Ellingson, "Three-Dimensional Radiography," in *Encyclopedia of Mater. Sci. and Eng.*, ed. Michael B. Bever, Pergamon Press, Oxford, England, 1986.
19. 1991 Industrial Computed Tomography Conference II, Published by the Amer. Soc. for Nondestructive Testing, Columbus, Ohio, USA.

Elastic Optical Scattering

20. J. S. Steckenrider and W. A. Ellingson, "Application of Laser Scattering to the Detection of Surface and Subsurface Defects in Si_3N_4 Components," *Ceram. Eng. Sci. Proc.* 15(4):382-389 (1994).
21. J. G. Sun, M. H. Haselkorn, and W. A. Ellingson, "Laser Scattering Detection of Machining-Induced Damage in Si_3N_4 Components," presented at the Eighth International Symposium on Nondestructive Characterization of Materials, June 15-20, 1997, Boulder, Colorado.
22. J. G. Sun et al., "Application of Optical Scattering Methods to Detect Damage in Ceramics," in *Machining of Ceramics and Composites, Part IV: Chapter 5*, in press (1998).
23. J. G. Sun, M. Shirber, and W. A. Ellingson, "Laser-Based Optical Scattering Detection of Surface and Subsurface Defects in Machined Si_3N_4 Components," *Ceram. Eng. Sci. Proc.* 18(4):273-280 (1997).

Internal Distribution

P. F. Becher, 4515, MS-6068
T. M. Besmann, 4515, MS-6063
R. A. Bradley, 4508, MS-6161
S. A. David, 4508, MS-6095
M. K. Ferber, 4515, MS-6069
J. A. Haynes, 4515, MS-6063
M. A. Janney, 4508, MS-6087
D. R. Johnson, 4515, MS-6066
M. A. Karnitz, 4515, MS-6065
E. Lara-Curzio, 4515, MS-6069
R. J. Lauf, 4508, MS-6085
H. T. Lin, 4515, MS-6068
R. A. Lowden, 4515, MS-6063
P. J. Maziasz, 4500S, MS-6115
S. B. McSpadden, 4515, MS-6069
K. L. More, 4515, MS-6064
S. D. Nunn, 4508, MS-6087
O. O. Omatete, 4508, MS-6087
A. E. Pasto, 4515, MS-6062
B. A. Pint, 4500S, MS-6156
A. C. Schaffhauser, 4500N, MS-6186
W. A. Simpson, 4500S, MS-6158
D. P. Stinton, 4515, MS-6064
R. W. Swindeman, 4500S, MS-6155
P. F. Tortorelli, 4500S, MS-6156
A. A. Wereszczak, 4515, MS-6069

External Distribution

AlliedSignal Ceramic Components, 2525 W. 190th St., Torrance, CA 90504

D. Foley
D. Newson
J. Pollinger
D. Twait
J. Wimmer

AlliedSignal Composites, 1300 Marrows Rd., PO Box 9559, Newark, DE 19714-9559

P. Craig
R. Matsumoto

AlliedSignal Engines, 2739 E. Washington St., PO Box 5227, Phoenix, AZ 85010
B. Schenk

AlliedSignal Power Systems, 8725 Pan American Freeway NE, Albuquerque, NM 87113
H. A. Pursley

Argonne National Laboratory, 9700 S. Cass Ave., Argonne, IL 60439-4838
W. A. Ellingson
J. G. Sun

Capstone Turbine Corp., 6430 Independence Ave., Woodland Hills, CA 91367
K. Duggan
M. Stewart

Caterpillar Inc., Advanced Materials Technology, PO Box 1875, Peoria, IL 61656-1875
M. Readey

Cummins Engine Co., Inc., 1900 McKinley Ave., MS: 50183, Columbus, IN 47201
T. Yonushonis

DaimlerChrysler AG, CIMS 481-00-64, 800 DaimlerChrysler Dr., Auburn Hills, MI 48236-2757
P. Rosa

George C. DeBell, Consultant, 40960 Jill Ct., Plymouth, MI 48170-2752

Delphi Energy and Engine Management Systems, Mail Code 485-220-130, 1300 N. Dort Hwy.,
Flint, MI 48556
F. Kennard

DOE Chicago Operations Office, 9800 S. Cass Ave., Argonne, IL 60439
J. E. Jonkouski
J. Mavec
J. Livingood
S. Waslo

DOE-HQ, 1000 Independence Ave., SW, Washington, DC 20585
D. Haught, EE-21
P. Hoffman, EE-22
T. King, EE-22
W. P. Parks, EE-22
S. L. Richlen, EE-21
M. Smith, EE-23
C. A. Sorrell, EE-23

DOE-HQ, 19901 Germantown Road, Germantown MD 20874-1290
H. Kerch, SC-13

Energetics, Inc., 501 School St., SW, Suite 500, Washington, DC 20024
R. Scheer

Energy Technologies Applications, 5064 Caminito Vista Lujo, San Diego, CA 92130-2849
T. Bornemisza

Freedman, Steven I., Engineering Consultant, 410 Carlisle Ave., Deerfield, IL 60015

Ford Motor Co., 20000 Rotunda Dr., Dearborn, MI 48121-2053
R. L. Allor, MD3535 SRL Bldg.
G. M. Crosbie, MD3182 SRL Bldg.
P. C. Killgoar, Jr., MD3182 SRL Bldg.
J. M. Nicholson, MD3135 SRL Bldg.
R. F. Novak, MD3182 SRL Bldg.

Gas Turbine Association, 1050 Thomas Jefferson St., NW, 5th Fl, Washington, DC 20007
J. Abboud

General Electric CR&D, PO Box 8, Schenectady, NY 12301
G. S. Corman
K. L. Luthra
J. Lynch

General Motors NAO R&D, Mail Code 480-106-212, PO Box 9055, Warren, MI 48090-9055
R. Powell

Kyocera Industrial Ceramics Corp., 5713 E 4th Plain Blvd., Vancouver, WA 98661
D. Carruthers

NASA Glenn Research Center, 21000 Brookpark Road, MS 106-5, Cleveland, OH 44135
J. DiCarlo

NREC, 39 Olympia Ave., Woburn, MA 01801-2073
P. Baldwin
K. Ginwala

NREC, 32 Exeter St., Portsmouth, NH 03801
J. Johnson
J. Kesseli

Richerson and Assoc., 2093 E. Delmont Dr., Salt Lake City, UT 84117
D. Richerson

Rolls-Royce Allison, 2925 W. Minnesota St., Indianapolis, IN 46241

S. Berenyi

P. Heitman

L. Groseclose

T. SEBESTYEN, Consultant, 1309 Park Garden Lane, Reston, VA 20194-1998

SOLAR TURBINES, INC., 2200 Pacific Hwy., PO Box 85376, MZ R, San Diego, CA 92186-5376

P. Browning

M. D. Fitzpatrick

V. S. Parthasarathy

M. Van Roode

Teledyne Continental Motors, 1330 W. Laskey Rd., PO Box 6971, Toledo, OH 43612-0971

J. T. Exley

Turbogen AB, Stora Varvsgatan 19, Malmö, S-20122, Sweden

L. Malmrup

UCI Combustion Laboratory, U. of CA, Irvine, Irvine, CA 92697-3550

V. McDonell

UDRI, Ceramic & Glass Laboratories, 300 College Park Ave., Dayton, OH 45469-0172

G. Graves

N. Osborne

United Technologies, Pratt & Whitney, 400 Main Street, MS 165-22, East Hartford, CT 06108

W. H. Day

United Technologies, Research Center, 411 Silver Lane, MS 129-24, East Hartford, CT 06108

H. E. Eaton

J. Holowczak

U. S. Advanced Ceramics Association, 1600 Wilson Blvd., Suite 1008, Arlington, VA 22209

B. Werst

Williams International, 2280 West Maple Rd., PO Box 200, Walled Lake, MI 48390-0200

R. Oates

C. Schiller