ANL/ET/CP --85-328
CONF-950129--10

AUTOMATED LASER SCATTER DETECTION OF SURFACE AND SUBSURFACE DEFECTS IN Si_3N_4 COMPONENTS*

by

J. S. Steckenrider
Energy Technology Division
ARGONNE NATIONAL LABORATORY
Argonne, IL 60439

DISCLAIMER

The submitted manuscript has been authored by a contractor of the U. S. Government under contract No. W-31-109-ENG-38. Accordingly, the U. S. Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or allow others to do so, for U. S. Government purposes.

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 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.

To be presented at the American Ceramics Society's 19th Annual Conference
on Composites, Advanced Ceramics, Materials and Structures

January 8-12, 1995

*Research performed as a subcontractor to Solar Turbines, Inc. as part of the Ceramic Stationary Gas Turbines program under DOE contract DE-AC02-92CE40960.

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED ^{10/}

MASTER

DISCLAIMER

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

AUTOMATED LASER SCATTER DETECTION OF SURFACE AND SUBSURFACE
DEFECTS IN Si_3N_4 COMPONENTS

J. Scott Steckenrider

Energy Technology Division

Argonne National Laboratory

9700 South Cass Avenue

Argonne, IL 60439

ABSTRACT

Silicon Nitride (Si_3N_4) ceramics are currently a primary material of choice to replace conventional materials in many structural applications because of their oxidation resistance and desirable mechanical and thermal properties at elevated temperatures. However, surface or near-subsurface defects, such as cracks, voids, or inclusions, significantly affect component lifetimes. These defects are currently difficult to detect, so a technique is desired for the rapid automated detection and quantification of both surface and subsurface defects. To address this issue, we have developed an automated system based on the detection of scattered laser light which provides a 2-D map of surface or subsurface defects. This system has been used for the analysis of flexure bars

and button-head tensile rods of several Si_3N_4 materials. Mechanical properties of these bars have also been determined and compared with the laser scatter results.

INTRODUCTION

The need to achieve higher gas firing temperatures in new stationary gas turbines has led to the requirement for stronger, longer-lasting materials capable of functioning in environments more severe than traditional materials permit. Such a higher operating temperature ($>1300^\circ\text{C}$) would provide a substantial increase in energy efficiency and reduced emissions. For these new turbines, conventional materials will not do, so a number of alternate material configurations are being considered. Included among these is Si_3N_4 ceramics. In an effort to improve and extend the working lifetimes of these components, improvement in the detection of critical defects (such as cracks, voids, inclusions, or microstructural variations) in their surface and near-subsurface regions is being investigated. While all of these defects can be created by either the manufacturing process or the in-service application, the specific type of defect most likely to occur in a given component depends on a variety of factors, including material system, processing and machining parameters, component geometry, and application environment.

The defects generated in the near-subsurface region must be detected as early as possible to minimize cost [1] or to reduce or eliminate in-service failure of the ceramic component. Although a number of techniques exist for the characterization of surface condition and defects [2,3,4], nondestructive characterization of the near-subsurface is a relatively complicated endeavor, particularly for 2-D surfaces. Traditional methods of nondestructive evaluation (NDE) such as radiography and ultrasonics are not well suited for subsurface defect detection. Radiography is primarily sensitive to interior (bulk) defects and is therefore relatively insensitive to surface/near-surface defects[5]. Ultrasonics, on the other hand, can be highly sensitive to the surface/near-

surface region. However, in order to obtain sensitivity to the near-subsurface region, higher frequencies must be used, and these require the use of a coupling agent (i.e., water, oil, etc.), which is often undesirable in ceramic applications where the ceramic may not be 100% dense. Ultrasonic methods are also generally best suited for flat surfaces, so the complex geometries associated with turbine components can be problematic[6].

A technique is therefore desired for the rapid characterization and defect detection and quantification of the near-subsurface (and surface) region of an array of Si_3N_4 ceramics in multiple geometries. Previous researchers have focused considerable effort into the noncontact characterization of machined surfaces of metals by elastic-optical-scattering (predominantly laser-based) techniques [3,7,8]. However, these techniques do not directly provide information on the subsurface condition of these materials.

The ability to characterize the subsurface material in some ceramics (e.g., zirconia) for density variations has been shown[9]. For many Si_3N_4 (and other) ceramics, the optical penetration depth can be $\geq 100 \mu\text{m}$ in the visible spectrum, depending on grain size, second-phase composition, and material absorption[10]. Because Si_3N_4 components can partially transmit visible (and IR) light, we have previously applied static polarization based laser scattering to the analysis of various Si_3N_4 materials fabricated into several component shapes, detecting defects as deep as $100 \mu\text{m}$ below the surface [11]. In an effort to apply this technique to the inspection of gas turbine components, a number of candidate Si_3N_4 materials systems have been investigated using an automated 2-D scanning laser scatter imaging system which provides a 2-D map of defects indicating their relative position and severity. To determine applicability to multiple geometries, and to correlate laser scatter results with actual mechanical properties, both flat (flexure bars) and curved (button-head tensile rods) specimens were examined.

EXPERIMENTAL PROCEDURE

Surface Analysis

The most important features in the inspection of ceramic surfaces are the size and orientation of surface discrepancies. Detection of such defects is somewhat simplified in materials whose "normal" surface condition is uniform and well defined. Detection and quantification of surface irregularities can then be accomplished by optical Fourier filtering. In this technique, a spatial Fourier transform of a surface is recorded using precision optics and coherent (i.e., laser) illumination. For a well defined uniform surface, the Fourier pattern of an undamaged surface can be recorded or computed and then subtracted from the actual pattern scattered from the surface. In regions where no surface defect is present, the difference is essentially zero. However, when a defect enters the inspection system's field of view, additional spatial frequencies appear in the Fourier plane. Since these frequencies are not present in the undamaged surface, they produce an increase in signal in the pattern difference. Furthermore, if the distribution of spatial frequencies is analyzed, the specific shape and orientation of the surface defect can be determined.

Subsurface Analysis

Subsurface laser scatter analysis is a relatively straightforward technique in which a specimen is directly illuminated by a laser beam (either focused or unfocused, depending on the specific application). The only material requirement is that the specimen of interest be translucent (e.g., able to diffusely transmit or scatter light), as is the case with most polycrystalline ceramics. The degree of scatter is dependent upon the difference in index of refraction between the grain boundary region and the bulk crystal. Si_3N_4 materials are highly scattering (so much so that they generally appear opaque) because in order to achieve maximum densification, a small percentage

(usually $\approx 5\%$) of second phase material such as Y_2O_3 , Al_2O_3 , or MgO is added to the Si_3N_4 . This material, which has a significantly different index of refraction than does the Si_3N_4 , and may even absorb light, collects at the grain boundaries. Thus, the Si_3N_4 's opacity results from the absorption and/or relatively high reflection which occurs at these grain boundaries. When a relatively thick specimen of Si_3N_4 is illuminated by a beam of light, whatever light is not absorbed is back-scattered in an almost purely diffuse pattern (e.g., an equal intensity is scattered in all directions). If a defect (i.e., a pore, crack, inclusion, etc.) is now placed in the subsurface material, the amount of back-scattered light will be changed. If the defect is, for instance, an iron inclusion, it will absorb significantly more light than would the Si_3N_4 material it displaced giving a decrease in back-scattered intensity. If, on the other hand, the defect were a lateral crack or pore, the difference in index of refraction between the air which would fill such a defect ($n \approx 1$) and the Si_3N_4 itself ($n \approx 1.9$) is significantly higher than that between the Si_3N_4 and the densification aiding material, so the back-scattered intensity would increase for such a defect.

The relative depth of a given defect can also be approximated by determining the ratio of the scattered intensity in the region of the defect to that of the normal material. The more this ratio differs from unity, the closer the defect is to the surface. While determination of an exact relationship between scattered intensity and defect depth is not possible, the general trend does permit an approximation to be made with regard to a defect's depth. (In addition, any depth determination requires that the beam spot size on the surface be smaller than the detected defect. Otherwise the intensity change will be moderated by undamaged material surrounding the defect.) Thus, by measuring the type, degree, size, and shape of a defect's laser scatter signature, a general description of the defect can be derived along with its location on the specimen.

System Design and Operation

The experimental arrangement used for the analyses presented herein is illustrated in Fig.

1. A vertically polarized laser beam is directed through a polarizing beam-splitter (PBS) cube onto the specimen surface by means of some focusing optics so that light specularly reflected from the surface will be collimated. Any light reflected from the component surface will undergo no change in polarization (unless the surface is extremely rough), so all surface scattered light will again be reflected in the PBS and directed back toward the laser. However, the polarization of any light which is scattered from the subsurface material will be randomized (and the scattered light will be completely diffuse). Thus, half of the subsurface scattered light will also be reflected by the PBS and directed back to the laser. However, the other half of the subsurface scattered light will be transmitted by the PBS into the detection train.

If surface scattered light is of interest, a properly oriented quarter-wave ($\lambda/4$) plate is inserted between the PBS and component surface. This serves to rotate the light's polarization by 90° on each pass so that all of the surface scattered light and half of the subsurface scattered light are transmitted to the detection train. Since in most cases the surface scattered light is much more intense than the subsurface scattered light, it dominates the detected signal so that the subsurface scatter can be neglected.

Figure 1. Illustration of the basic setup for the laser scatter inspection technique.

In both arrangements, the detection train is the same. A linear sheet analyzing polarizer (with a polarization efficiency higher than that of the PBS) is placed behind the PBS to maximize signal-to-noise ratio. A high quality positive lens with focal length f_T (the transforming lens) is then placed just beyond the polarizer. A CCD camera is focused at a distance behind the transforming lens of f_T . What is imaged on the CCD array is in fact the power spectrum of the spatial Fourier transform of the surface or subsurface of the component. The specific system used in this effort records the full 2-D optical Fourier power spectrum of back-scattered laser light from a single point on the component including spatial frequencies up to $0.2 \mu\text{m}^{-1}$. This Fourier scatter pattern is then digitized for further analysis using a 486/66 MHz PC. Thus by examining the intensity distribution in the camera's image one can determine the character of roughness on the component's surface or subsurface. In order to improve acquisition speed, the system initially only computes the average magnitude of all such frequencies which is then mapped in a gray scale value

as a function of specimen position. The result is scatter "images" of the surface or subsurface material. In cases where specific defects were detected using this scanning approach, more detailed inspection of the actual scattered light's Fourier power spectrum was performed.

Machined Step-Wedges

The first specimens examined were machined step-wedges 4mm wide and 25 mm long with nominal step thicknesses of 800, 400, 200, 100, and 50 μm . Wedges were made from a variety of different Si_3N_4 materials, and each wedge was polished on its top (flat) surface. These wedges were used for two purposes. First was the determination of optical transmittance at a wavelength of 632.8 nm using an integrating sphere for a number of different Si_3N_4 ceramics. The results of these measurements are shown in Fig. 2. Note that the transmission characteristics vary across the several Si_3N_4 materials shown by more than an order of magnitude. This means that the total depth of penetration (i.e. the depth to which defects can be detected) will also vary between materials.

Figure 2. Plot of optical transmission as a function of specimen thickness at a wavelength of 632.8 nm. Lines are best-fit approximations to:

$$T = I_T/I_I = e^{-\alpha t}, \text{ where } t \text{ is thickness and } \alpha\text{'s are given parenthetically in the legend.}$$

The step-wedges were also used to simulate subsurface lateral cracks. In this case, the wedge was again illuminated normal to its polished surface, and the intensity of back-scattered light was monitored. Thus, while the front surface appeared uniform across the entire wedge, the steps machined into the back surface provided a ceramic-air interface in the subsurface material to simulate a subsurface lateral crack. The back-scattered intensity at each point was digitized and recorded. These scatter intensities were normalized into gray scale values and assembled into a composite image of the step-wedge. Examples of such images are presented in Fig. 3, with the thinnest steps on the left. If these steps had been actual cracks, the back-scattered intensity would have been even greater, owing to additional scatter from the material beneath the defect.

Figure 3. Subsurface laser scatter images of three Si_3N_4 step-wedges. (a) GS44, (b) NT164, and (c) SiAlON.

RESULTS AND DISCUSSION

Examined in this study were 80 flexure bars from various manufacturers with both machined and as-fired surface conditions, and 8 tensile rods of four different materials. Both surface sensitive and subsurface sensitive analyses were performed. While presentation and discussion of all results would be excessive, several noteworthy examples will be presented.

A wide variety of response from one material type to another was seen. Three examples are shown in Figs. 4 and 5. Figure 4(a,b) shows the laser scatter images for both surface and subsurface scans of 10 GN-10 flexure bars. (All flexure bars presented herein were 4mm wide, and a gauge section of 20 mm length was examined in each bar at 200 μm resolution. Defects smaller than 200 μm were detectable, though the intensity of their signatures was reduced.) Two features are clearly evident from the figure. First, there is ink written on all 10 bars which is visible in both the surface and subsurface images. Although numerous attempts were made to remove this ink (used to label each specimen), all were unsuccessful. Thus, a defect beneath the ink would not be detected. The second feature seen in the laser scatter data is an overall mottling of the subsurface image which is not seen in the surface image, and is therefore entirely a subsurface

phenomenon. In addition, since the same mottled appearance is evident in x-ray radiographic images it would appear that the mottling is actually a variation in local density. (Note that a similar phenomenon has been seen in bearing components made from another ASCC ceramic, GS44. In that case, destructive analysis indicated a locally high porosity in the regions of reduced laser scatter[11].)

Figure 4(c,d) shows the laser scatter images for both surface and subsurface scans of 10 NT-164 flexure bars. The 5 bars on the left were machined, while the 5 on the right were in the as-fired condition. The most severe of the defects seen in this group is the dark spot in the lower extreme of bar #11's subsurface image. Based on previous experience (through the visual inspection methodology currently being applied for flexure bar qualification) this appears to be an iron inclusion of some kind, and it is readily seen using high resolution radiography. However, several other similar, though less severe, occurrences were seen with the laser scatter technique in other NT164 bars, as shown with the black circles in Fig. 4d, which were not detected using either visual inspection or x-ray radiography. Initial mechanical testing has revealed that these iron inclusions act as stress concentration points where failure initiates, but the specimens shown in Fig. 4 have not yet been mechanically tested.

Figure 5 shows subsurface and surface laser scatter images for an NT-164 tensile rod. These images were acquired with a 100 μm resolution and are 40 mm long and 20 mm wide (circumference). As with the GN-10 flexure bars presented above, this tensile rod was written on with a "permanent" ink pen in the gage region. Fortunately, we were able to remove all of the ink from the surface (as seen in Fig. 6a). Although all of the surface ink was removed using acetone and alcohol, some of the ink remained in the subsurface after cleaning, as can be seen in the subsurface image (with boxes drawn around the ink marks to delineate them from actual defects).

Despite the presence of this ink, two defects are still evident in this rod. First, there is a significant number of distributed defects in the NT164 rods which appear as dark spots. Again, as in the flexure bar of the same material, these are likely iron inclusions which are common in the NT-164 material. Furthermore, because they are not visible in the surface scan, they are completely enclosed within the rod. The second feature is a linear defect on the left hand side of the image about halfway up its length which cannot be attributed to residual ink. Because it appears in both the surface and subsurface images as a bright and dark indication, respectively, this is likely a surface scratch which is sufficiently severe as to dominate the laser scatter even in subsurface sensitive analysis.

Figure 4. Images of (a,b) AlliedSignal Ceramic Components' GN-10 flexure bars and (c,d) Norton's NT-164 flexure bars showing (a,c) surface sensitive laser scatter images and (b,d) subsurface sensitive laser scatter images.

Figure 5. Images of a Norton NT-164 tensile rod showing (a) surface and (b) subsurface sensitive laser scatter images.

CONCLUSIONS

By taking advantage of Si_3N_4 's ability to partially transmit visible (and IR) light, we have developed a Laser Scatter Inspection technique and applied it to the detection of defects and/or damage in several different Si_3N_4 materials and in multiple component geometries. The type of defects detected include iron inclusions, surface scratches, subsurface density variations, and even subsurface residual ink. Using polarization techniques, separation of the effects surface and subsurface defects was possible to depths of several hundred microns. Furthermore, through the application of a two dimensional scanning system, conformally mapped images of the scattering surface or subsurface were generated for flat and cylindrical specimens with a resolution of from 10 μm to 1 mm. Mechanical testing of all components inspected is currently underway, but these results are not yet available. However, limited results from previous analyses indicate that the type of defects detected (especially iron inclusions and density variations) are those which would

cause premature failure. Thus, the laser scatter method holds promise for the automated inspection and qualification of ceramic components for gas turbine components.

ACKNOWLEDGMENTS

The author would like to acknowledge Steven Hester for his diligence in data acquisition and Vijay Parthasarathy of Solar Turbines for preliminary mechanical testing. The work was performed under a subcontract to Solar Turbines, Inc. as part of the Advanced Turbine Systems program under DOE contract number DE-AC02-92CE40960.

REFERENCES

1. Konig, W., and Popp, M. "High Precision Machining of Advanced Ceramics," in Third International Symposium on Ceramic Materials and Components for Engines, Las Vegas, NV, Nov. 27-30, 1988, V.J. Tennery, ed., published by Am. Cer. Soc., pp. 1159-1224.
2. Françon, M., Laser Speckle and Applications in Optics. New York, Academic Press (1979).
3. Stover, J.C., Optical Scattering: Measurement and Analysis, New York, McGraw-Hill (1990).
4. Church, E.L., J.C. Dainty, D.M. Gale, and P.Z. Takacs, "Comparison of Optical and Mechanical Measurements of Surface Finish," *Proc. SPIE*, 954-27 (1988).
5. Sivers, E.A., D.L. Holloway, W.A. Ellingson, and J. Ling, "Development and Application of Local 3-D CT Reconstruction Software for Imaging Critical Regions in Large Ceramic Turbine Rotors," in *Review of Progress in QNDE*, Vol. 12, eds. D.O. Thompson and D.E. Chimenti (Plenum, New York, 1993), pp. 357-364.

6. Chou, C.-H., P. Parent, and B.T. Khuri-Yakub, "Acoustic Microscopy with Mixed Mode Transducers," in *Review of Progress in QNDE*, Vol. 8, eds. D.O. Thompson and D.E. Chimenti (Plenum, New York, 1989), pp. 865-871.
7. Vorburger, T.V. and E.C. Teague, Optical Techniques for On-Line Measurement of Surface Topography, *Prec. Eng.* 3(2) (1981), pp. 61-83.
8. Bennett, J.M. and L. Mattson, *Introduction to Surface Roughness and Scattering* (Optical Society of America, Washington, DC 1989).
9. Cielo, P., X. Maldague, S. Johar, and B. Lauzon, "Some Laser-Based Techniques for the Characterization of Sintered Ceramics," *Mat. Eval.*, **44** (May 1986).
10. Ellingson, W.A., D.M. Ayaz, M.P. Brada, and W. O'Connell, "Detection of Subsurface Defects in Machined Silicon Nitride Ceramics by Optical Scattering Methods," in *Machining of Advanced Materials*, NIST Special Publication 847, Washington, DC (1993), pp 147-157.
11. Steckenrider, J.S. and W.A. Ellingson, "Application of Laser Scattering to Detection of Surface and Subsurface Defects in Si_3N_4 Components", *Ceramic Engineering and Science Proceedings Vol. 15: 18th Annual Conference on Composites and Advanced Ceramic Materials*, (Cocoa Beach, FL, January 9-14, 1994), American Ceramics Society, 382-389 (1994).