D. NDE Development for Ceramic Valves for Diesel Engines

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Objectives

- Develop a laser scattering nondestructive evaluation (NDE) method for detection and characterization of sub-surface defect/damage in silicon-nitride ceramic valves so NDE data can be used to determine cost-effectiveness and reliability of ceramics valves for diesel engines.
- Identify strength-limiting flaws and failure mechanisms in cylindrical silicon-nitride specimens due to inherent material defects or machining damage.

Approach

- Continue optimization of an automated laser scattering NDE system for fast scanning of entire valves.
- Inspect as-processed valves to determine the level of machining damage and evaluate bench-tested valves for accumulated damages.
- Correlate NDE data with fracture test results for machined cylindrical silicon nitride specimens to identify fracture initiation defects and defect type, depth, and severity.

Accomplishments

- Successfully tested a photomultiply tube (PMT) optical detector in a laser scattering system. The PMT detector is more sensitive and >6 times faster than the current semiconductor optical detector.
- Evaluated ten NT551 silicon-nitride valves that were bench tested for 100, 500, and 1000 hours. Analyzed processing and accumulated damages in the valve head sections.
- Inspected 48 as-processed SN235P silicon nitride valves. Determined levels of machining damage in various sections of the valves and identified severe damage that caused valve failure.
- Demonstrated laser scattering detection sensitivity for fracture initiation defects in machined cylindrical SN235P silicon nitride specimens.

Future Direction

- Implement the PMT detector in the automated NDE valve-scan system and use optical fibers for collecting and delivering scatter light to the detector. Further investigate other detectors that may have higher speed for this application.
- Inspect re-machined SN235P valves for initial damage level due to machining. Characterize accumulated damage by periodic inspection of these valves during planned rig/engine tests of various operating conditions and durations.
- Develop new theoretical, experimental, and quantitative image-processing methods for optimized characterization of strength-limiting subsurface defects/damage (size and depth) due to corrosion, fatigue, impact, and wear in silicon nitride ceramics.

Introduction

Advanced ceramics are leading candidates for high-temperature engine applications that offer improved fuel efficiency and engine performance. Among them, silicon nitrides are being evaluated as valve train materials for diesel and natural gas engines. However, material defects and process/operation-induced damage in the subsurface may significantly degrade the reliability and performance of these ceramics. These defects are typically less than 100 microns in size and within 200 microns under the surface. To detect and characterize these defects, Argonne National Laboratory (ANL) developed a laser scattering NDE method to measure detailed subsurface microstructures for ceramics. The objective of this research is to demonstrate that this method can be used to assess/evaluate the cost-effectiveness and reliability of ceramic valves for diesel engines. The primary effort in FY 2004 was to evaluate accumulated impact/wear damages in 10 NT551 silicon nitride valves tested in a bench rig for 100, 500, and 1000 hours and to inspect 48 as-processed (machined) SN235P silicon nitride valves for initial machining quality. Another effort was focused on NDE identification of fracture initiation defects in machined cylindrical SN235P silicon nitride specimens. This research is collaborated with Caterpillar, Inc.

Approach

The critical region of ceramic components for structural applications is near-surface (usually to a depth of <200 μm). The common types of defects in this region are mechanical ones, such as cracks, spalls, inclusions, and voids. Because ceramics are partially translucent to light, a laser scattering method based on cross-polarization detection of optical scattering originated from the subsurface can be used for noncontact, nondestructive measurement of subsurface microstructure in such materials. By scanning the entire surface (flat or curved) of a ceramic component and constructing a two-dimensional (2D) scatter image, subsurface defects can be readily identified, as they exhibit excessive scattering over the background, and their type and severity may be analyzed. To apply this technology for NDE of ceramic valves, an automated laser scattering system was developed by ANL for scanning entire valve surfaces. This system utilizes two rotation and two translation stages to align and focus the laser beam on the valve surface during the scan; and the resulting 2D scattering image data are used to identify the location, size, and relative severity of subsurface defects/damage. This system can therefore be used for inspection of the quality of as-processed valves and for evaluation of accumulated damage in ceramic valves from rig/engine tests.

Results

Evaluation of Bench-Tested NT551 Valves

Ten NT551 silicon nitride valves (Figure 1) were tested for 1000 h in a valve bench rig at Caterpillar. These valves were seated with metallic seat inserts at matched or mismatched angles, or with eccentric seat inserts. The bench test matrix is listed in Table 1.[1] These valves were examined by the automated laser scattering system at accumulated durations of 100, 500, and/or 1000 h as shown in Table 1. NDE data for each valve at different test durations were compared and analyzed to determine

Table 1. Bench test matrix and NDE evaluation intervals for NT551 valves

Valve number	100-h valve-seat	400-h valve-seat	500-h valve-seat	Total hours	NDE at accumulated bench-test hours
CV04, CV12	30-30	30-30	30-30	1000	500
CV19, CV20	45-45	45-45	45-45	1000	500, 1000
CV18, CV25	45-30	45-30	45-30	1000	500, 1000
CV08, CV15	30-45	30-ECC	30-ECC	1000	100, 500, 1000
CV14, CV16	30-ECC	30-ECC	30-ECC	1000	100

Notes: numbers for valve-seat are their respective angles; ECC = eccentric seat insert.

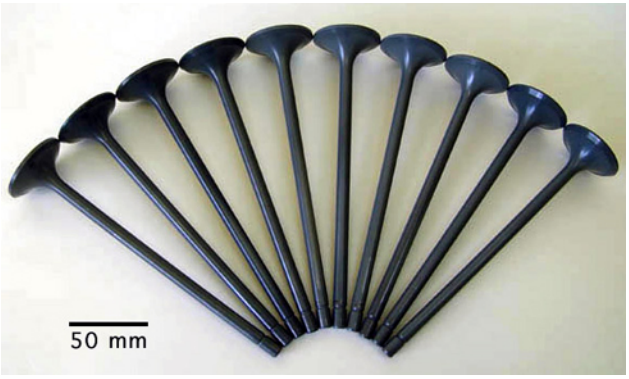

Figure 1. Ten NT551 silicon nitride valves.

any new damage or growth of existing defects in the contact and fillet radius surfaces.

Figure 2 shows the laser scattering image of the valve-head section of NT551 valve CV19 after a 1000-h bench test. The scanned axial length was 30 mm (the top 4-mm region is the contact area) and is aligned in the vertical direction. The scan resolution was 10 microns in the axial direction and 11 microns in the circumferential direction. Typical of all NT551 valves, NDE data for CV19 indicated no significant accumulated damage from the bench tests, except surface wear within the contact surface. Figure 3 shows the detailed image of the contact surface. The reduction of scattering intensity within the wear scars (two darker horizontal stripes) was due to surface damage of increased roughness and contamination (embedded metal particles from the metal seat insert). Several segmented subsurface flaws of high optical-scattering intensity near the top

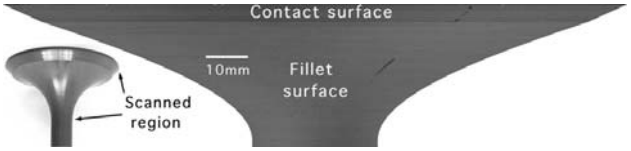

Figure 2. Laser scattering scan image for NT551 valve CV19 after 1000-h bench test.

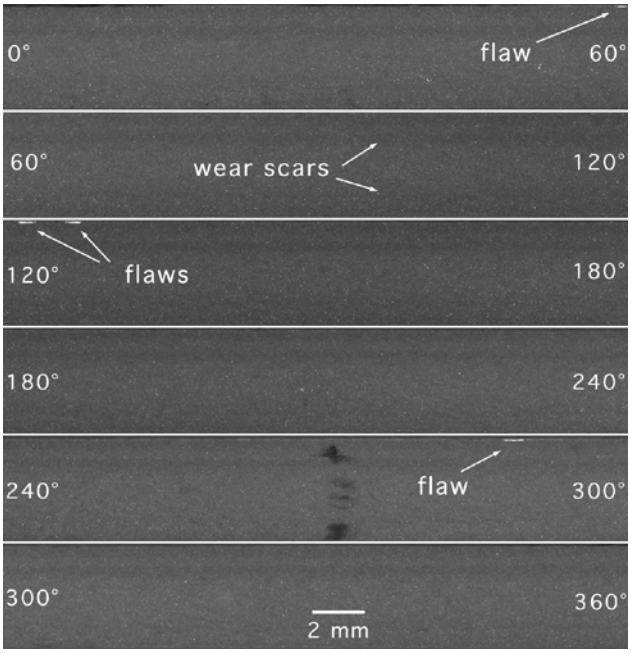

Figure 3. Laser scattering image of the contact surface of NT551 valve CV19.

edge are indicated in Figure 3. One of the detected edge flaws is shown in Figure 4 and compared with the surface photomicrograph. This flaw is pre-spall damage that was probably generated during valve processing. Other flaws within the subsurface are mostly material porosity of <80 μm in size. Valve CV19 also has several machining-damage marks in

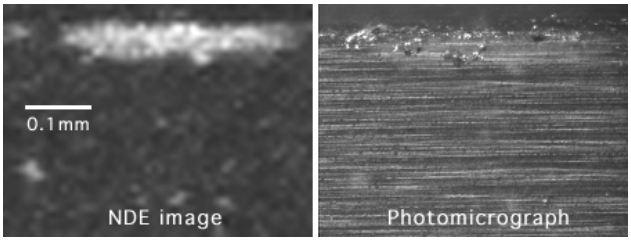

Figure 4. Detailed laser scattering image and photomicrograph of a subsurface edge flaw.

the fillet radius surface. In addition, subsurface damage, near a scratch, that was not present after a 500-h test was seen after a 1000-h test (Figure 5).

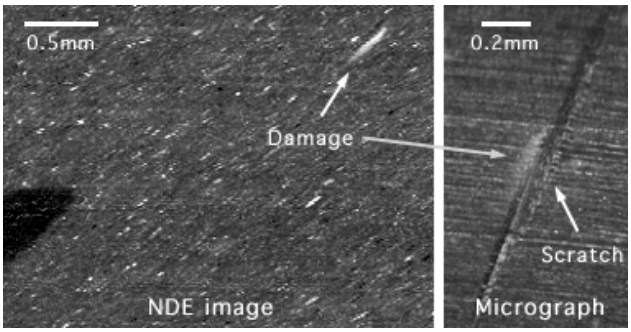

Figure 5. Detailed laser scattering image and photomicrograph of a subsurface scratch damage.

Inspection of As-Processed SN235P Valves

Forty-eight as-processed (machined) SN235P silicon nitride valves were inspected by the valve-scan NDE system. These were diesel and natural-gas intake and outlet valves, each with a slightly different axial profile. Figure 6 shows a laser scattering image of a typical SN235P diesel intake valve. Spatial resolution in both scan directions is ~10 micron. Significant machining damage was found in the fillet radius and nearby stem regions, as shown in detailed images in Figure 7. The damage is seen as lines of high optical-scattering intensities along the machining direction. This result is consistent with the machining conditions for these valves. As indicated in ref. 1, because of an error in the machining process, the valve fillet and keeper notch regions were only rough-machined, while the contact surface and the majority of the stem regions were fine-machined. The high damage level in the fillet region as detected by the NDE would likely reduce valve strength.

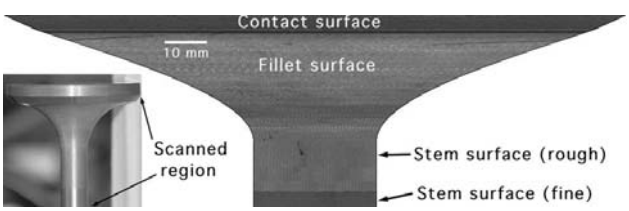

Figure 6. Laser scattering image of a typical SN235P diesel intake valve.

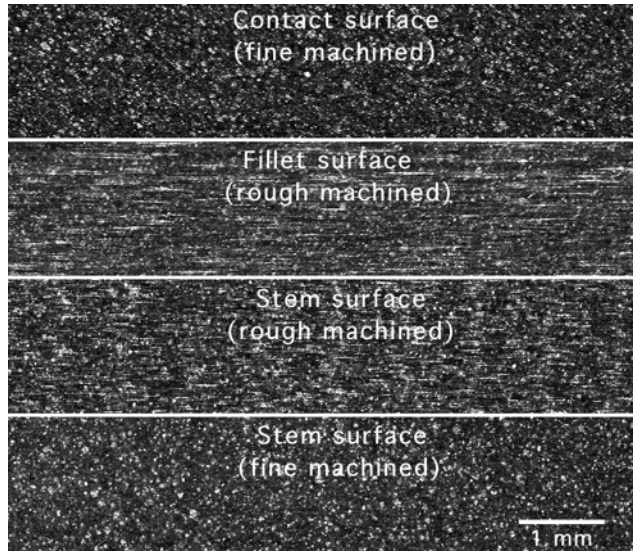

Figure 7. Detailed laser scattering images in various regions, as indicated in Figure 6.

Machining damage in the valve keeper notch region was not anticipated to be significant. However, when a diesel valve was tested in the bench rig at Caterpillar, it was soon broken near the upper edge of the keeper notch. In comparison, ten NT551 valves were tested in the same rig for 1000 h without a single failure. From scanning electron microscopy analysis, Caterpillar determined that the fracture origin was machining damage. NDE image data for a typical SN235P valve notch are shown in Figure 8. Very severe machining damage was found around the entire circumference of the upper edge, which is at the same location of the notch failure. The damage appears to be segmented; each segment is similar to the edge flaw of NT551 valve, as shown in Figure 4. The intensity of this damage is much higher than that within the notch which, however, is already considered to be more significant than typical rough machining damage. Because all machining damage is likely to be median cracks, these results indicate that laser scattering intensity may be correlated with crack depth and may predict failure.

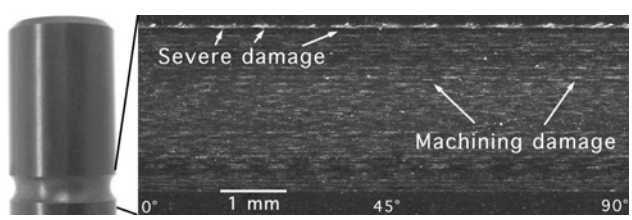

Figure 8. Laser scattering image in keeper notch region of a typical SN235P valve.

Detection of Machining Damage and Fracture Origins

Three sets of cylindrical SN235P silicon nitride specimens were examined by the laser scattering method to determine machining damage. Similar to the results for SN235P valves (Figures 6–7), NDE data showed damage level and distribution at different machining conditions. In addition, a color variation was observed on some rod surfaces, as seen in Figure 9. This coloration change is probably due to non-uniform mixing of powder materials during material processing, and it may have some effect on the mechanical property of the rods.

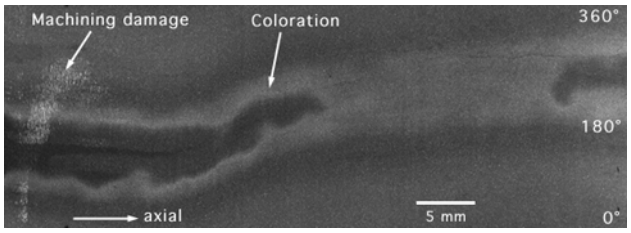

Figure 9. Color variation in a machined rod.

NDE data were correlated with destructive test results to determine the fracture initiation defect for these cylindrical SN235P specimens. The fracture surfaces were first examined to locate the fracture flaw site. This site was then mapped in the tensile (or cylinder) surface. By comparing the flaw location with a previously acquired laser scattering image for the surface, the fracture initiation defect can be identified in the NDE image. Figure 10 shows the fracture surfaces of a SN235P rod in which the fracture initiation defect can be easily observed. The correlation of the surface photomicrograph with the laser scattering image is seen in Figure 11. As indicated, the fracture initiation defect is not the largest in the region. This is because the strength test used a four-point bending fixture. The maximum stress is therefore localized within an axial line in a particular azimuth angle. Only those defects positioned within the maximum stress line will grow and cause fracture. The fracture initiation defect in this specimen appears to be a high-porosity material defect.

Conclusions

An automated laser scattering system developed by ANL was used to evaluate ten NT551 valves at accumulated bench-test durations of 100, 500,

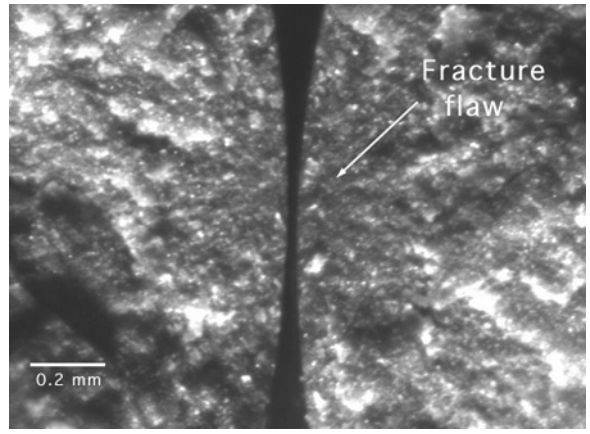

Figure 10. Fracture surfaces of a fractured SN235P rod.

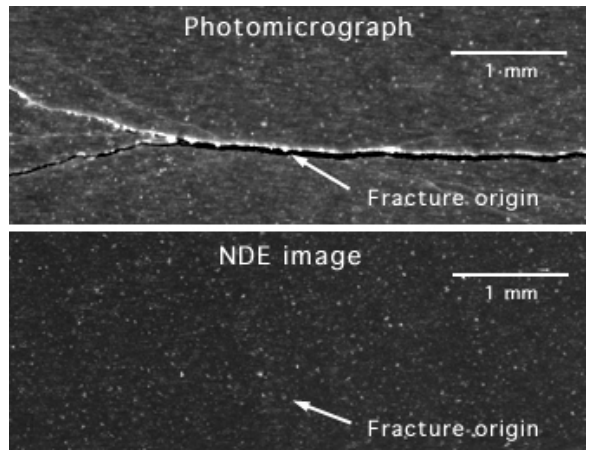

Figure 11. Photomicrograph and laser scattering image on tensile surface of a fractured SN235P rod.

and/or 1000 h. NDE data for each valve at different test durations were analyzed to determine any new damage or growth of existing defects. In general, no significant subsurface damage from the bench tests was detected. Existing flaws, such as machining damage and inherent material defects, did not grow under the various test conditions and durations. Damage from the bench tests was mainly in the valve contact surface, including wear scars and an unknown deposit for some valves.[1] The NDE data will be correlated with the retained strength from destructive tests of these valves.

Forty-eight as-processed (machined) SN235P valves were inspected by the valve-scan system. Because of an error in the machining process, the valve fillet and keeper notch regions were only rough-machined, while the contact and stem surfaces were fine-machined. The NDE data showed significant

machining damage in the rough-machined fillet radius and nearby stem regions. More severe damage was found at the upper edge of the keeper notch. The severe damage at the keeper notch was responsible for a valve failure, and the high damage level in the fillet region would likely reduce valve strength. Based on these NDE results, it was recommended that all SN235P valves be re-processed to remove all rough machining damage before being tested in an engine or a bench rig.

The laser scattering method was also used to characterize defects/damage in cylindrical SN235P silicon nitride specimens that were machined by different vendors at various machining conditions. NDE data identified damage level and distribution at different machining conditions, and a color variation in base material that was probably due to material processing. In addition, NDE data were demonstrated for identification of fracture initiation defects for these cylindrical SN235P specimens.

References

1. M. J. Andrews, "Lightweight Valve Train Materials," quarterly progress report for January 1 through March 31, 2004, Heavy Vehicle Propulsion Materials Program, U.S. Department of Energy.

Publications/Presentations

J. G. Sun, J. Zhang, and M. J. Andrews, "Laser Scattering Characterization of Subsurface Defect/Damage in Silicon Nitride Ceramic Valves," to be published in *Proceedings of the American Ceramic Society's Annual Cocoa Beach Conference*, Cocoa Beach, FL, Janury 25-30, 2004.

Patents

J. G. Sun, "Device and Nondestructive Method to Determine Subsurface Microstructure in Dense Materials," invention report ANL-IN-02-086, DOE case no. S-101,670, U.S. patent filed in December 2003.