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K. Goebbels
Fraunhofer-Institut für zerstörungsfreie Prüfverfahren

H. Reiter
Fraunhofer-Institut für zerstörungsfreie Prüfverfahren

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Characterization of Defects and Heterogeneities in Silicon Nitride and Silicon Carbide by Different NDE Methods

Abstract
The brittleness of ceramic materials like silicon nitride and silicon carbide makes it necessary to fabricate homogeneous structures and to detect small defects in the region of 10 to 100 microns diameter. In the German program on NDE for the gas turbine therefore a study was made to compare different NDE methods and to develop new techniques. Tests were made with ultrasonics, microradiography, vibration analysis, acoustic emission and optical-holographical interferometry on test samples and real components of the gas turbine (rotor, stator, combustor). The results show that especially

- microradiography with projection technique and X-ray focus of ≈ 10 µm diameter,
- ultrasonics with different kind of transducers, equipment and wave modes in the frequency range until about 150 MHz

are well suited to detect the small defects and to characterize structure heterogeneities.

- Vibration analysis seems to be a good method to compare many samples of the same kind and to detect matter of the fabrication process data.

The comparison between UT, vibration analysis, acoustic emission and destructive tests (fracture strength) indicates that there are more or less correlations between NDE and the destructive analysis.

Keywords
Nondestructive Evaluation

Disciplines
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CHARACTERIZATION OF DEFECTS AND HETEROGENEITIES IN SILICON NITRIDE AND SILICON CARBIDE BY DIFFERENT NDE METHODS

K. Goebbels, H. Reiter
Fraunhofer-Institut für zerstörungsfreie Prüfverfahren
D-6600 Saarbrücken 11, Germany

ABSTRACT

The brittleness of ceramic materials like silicon nitride and silicon carbide makes it necessary to fabricate homogeneous structures and to detect small defects in the region of 10 to 100 microns diameter. In the German program on NDE for the gas turbine therefore a study was made to compare different NDE methods and to develop new techniques. Tests were made with ultrasonics, micro-CT, vibrational analysis, acoustic emission and optical-holographical interferometry on test samples and real components of the gas turbine (rotor, stator, combustor). The results show that especially

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- Vibration analysis seems to be a good method to compare many samples of the same kind and to detect scatter of the fabrication process data.

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INTRODUCTION

Two problems have to be solved according to the nondestructive evaluation of ceramics like SiC and SiC for gas turbine components: characterization of materials structure (i.e. description of homogeneity, analysis of heterogeneities) and detection of smallest defects (dimensions 10 µm for surface regions, 10 µm below the surface). Two methods primarily are suited to solve these problems: high frequency ultrasonics and microfocus radiography. Both will be discussed in detail below. Several other methods were studied in the German program for the NDE of ceramics. From these the vibration analysis (determination of the main resonance frequency) seems to be the best way to characterize by an integral way a lot of components with the same symmetry (e.g. turbine blades). In Fig. 1 two series of one hundred 4-point-bending-test specimens (RBSN) were analyzed by ultrasonic Longitudinal-Wave Velocity ($v_L$) measurements and the resonance frequency (measured parameter: 2T = two lengths in the time domain). It is easily to that for the second series the fabrication process resulted in a more uniform structure (for the first series). For the first series measurements were completed by shear wave velocity ($v_T$) measurements, density ($\rho$) measurements and acoustic emission during the destructive strength determination (4-point-bending fracture). The correlation between these data and vibrations is shown in Fig. 2. Drawing the data row with increasing fracture strength shows the result to be expected: there is no clear correlation. But with further analysis of these a linear regression analysis points out that increasing strength the Poisson ratio $\nu$ and wavelength of resonance vibration $\lambda$ are decreasing while the ultrasonic wave velocities $v_L$ and the elastic modulus $E$ are increasing, too. We think that this should be analyzed further in new experimental series. For the acoustic emission measurement (by Motoren-Turbine-Union, Munich) no significant results could be obtained. Additional destructive tests together with acoustic emission energy measurements on 58 RBSN specimens with and without Knoop-Indent microcracks did not show any promising results.

Studies with optical-holographical interferometry from homogeneous and inhomogeneous (microcracks) RBSN samples were able to distinguish between the types of specimens (Institute for Applied Ray Technique, Bremen) but the ultrasonic and radiographic detection and location of the defects were more effective [11].
Analysis of 100 RBSN 4-point-bending-test specimens with ultrasonic waves, vibration analysis, acoustic emission in correlation to the fracture strength.

Fig. 2

ULTRASONICS

Structure heterogeneities can be detected with ultrasonic attenuation, scattering and velocity measurements. Because of the high accuracy of velocity determinations - e.g. by pulse-echo-overlap techniques - they were used for the analysis of many turbine components and samples of hot pressed and reaction bonded silicon carbide and silicon nitride /1/. In Fig. 3 two pictures are reproduced from a HPSN disc with 36 mm thickness and 136 mm diameter. In the outer region residual stresses could be detected with 5 MHz polarized shear waves (Fig. 3 above): double reflection arising for two directions of vibration (45° to the radial direction) and vanishing for the other two (radial and tangential) is a strong reference to residual stresses in circumferential orientation. The quantitative stress determination needs the knowledge of the elastic higher order constants l, m, n /2/. The inner region of the disc shows a sharp boundary of change of density (Fig. 3 below): three high damped backwall echoes (10 MHz longitudinal waves) followed by 20 µs without any echo and then many echoes created by a sequence of mode conversion processes are obtained if the pulse-echo transducer position is exactly on the boundary region.

Single defects generally are easier to detect with high frequency ultrasonic waves /2,4/ than with low frequencies. In special cases low frequencies can be used, too. Figure 4 shows the detection of small saw cuts (width 150 µm) of different depths with 17 MHz shear waves. The 5 µm cut also can be resolved with the 300 µm wavelength. In Fig. 5 natural surface defects in turbine blades are analyzed with 8 MHz Rayleigh waves: the amplitude of the signal reflected at the rotor ring is a measure for the influence of the defects on the wave propagation. For high frequency ultrasonic waves (10 MHz to 100 MHz) a new technique was adopted, recommended by Arnold /5/. LiTaO3 single crystals (p = 7.5 g/cm³, vL = 5.5 mm/µs, 5 mm diameter, 10 mm length) excited with high frequency bursts (cf. Fig. 6) can be used for a wide frequency region. The piezoelectric crystals do not need any evaporated thin films of metallic (e.g. Au) and piezoelectric (e.g. CdS) materials. Frequency and pulse width are given by the electric excitation. Figure 6 shows (left) more than 600 backwall echoes for 90 MHz and the first backwall echo followed by three backwall echoes from a 3.5 mm thick HPSN bending test specimen (right) for 20 MHz.

Fig. 3 Detection of residual stresses (above) and inhomogeneity regions (below) with 5 MHz shear and 10 MHz longitudinal waves, respectively in a disc of HPSN.

Fig. 4 Detection of 150 µm width slots of different depths in RBSN with 17 MHz shear waves.
Detection of surface defects in turbine blades with 8 MHz Rayleigh waves.

High frequency ultrasonic waves from a LiTaO\(_3\)-transducer (5 mm diameter, 10 mm length), left: backwall echoes for 90 MHz. Right: backwall echoes from 3.5 mm thick 4-point-bending-test specimen of HPSN (20 MHz).

MICRORADIOGRAPHY

Microfocus X-ray units enable the imaging of water sample areas (\(\approx 25 \times 25 \text{ mm}^2\)), for curved surfaces, with microscopic resolution (\(\approx 25 \mu\text{m}\)). The projection technique used (WARDRAY E12 Unit, developed by the NDT Centre Harwell) is sketched in Fig. 7. Electrostatic focussing of the electron beam results in a \(\approx 15 \mu\text{m}\) diameter X-ray focus inside the tube. The specimen directly is attached to the window (distance to the focal spot \(\approx 130 \text{ mm}\)) and the film has a distance of several meters to the specimen. The X-rays (\(\geq 80 \text{ kV} \text{age}, \approx 0.5 \text{ mA} \text{ current}) are propagating with a diverging beam of \(18^\circ\). By separating the sample film in projection radiography first a natural magnification of about x10 to x20 is achieved. Only a very significant 'clean up' in the microradiograph is effected because a very large portion of the secondary radiation dissipates itself by attenuation before it reaches the film. Heterogeneities as well as single defects are reproduced on the film. The exposure time depending on the kind of film and the sample thickness varies between \(< 1 \text{ sec}\) and \(\geq 1 \text{ hour}\). Density variations in HPSN and HPSC are shown in Fig. 8a and Fig. 8b. The disc described in Fig. 3 is reproduced by microradiography in Fig. 9, showing clearly the inner inhomogeneous zone. The slots in HPSN (150 \(\mu\text{m}\) width) analysed with ultrasonic waves in Fig. 4 are imaged in Fig. 10. The resolution of the X-ray technique allows to see the 21 \(\mu\text{m}\) slot. The smaller ones (17 and 6 \(\mu\text{m}\) deep) cannot be resolved. Surface defects in original turbine blades (Fig. 5 for ultrasonic waves) and pores in an original stator are shown in Fig. 11a and Fig. 11b, respectively. Some seeded defects (inclusions of Fe and C) in HPSN and HPSC specimens are imaged in Fig. 12.

Fig. 5 Detection of surface defects in turbine blades with 8 MHz Rayleigh waves.

Fig. 6 High frequency ultrasonic waves from a LiTaO\(_3\)-transducer (5 mm diameter, 10 mm length), left: backwall echoes for 90 MHz. Right: backwall echoes from 3.5 mm thick 4-point-bending-test specimen of HPSN (20 MHz).

Fig. 7 Microfocus X-ray equipment with projection technique

Fig. 8a Microradiography image of a HPSN specimen with density variation. Irradiated thickness: 9.5 mm.
Fig. 8b Microradiography image of a HPSC specimen with density variations. Irradiated thickness: 4 mm

Fig. 9 Microradiograph of a 36 mm thick HPSN disc with density variation.

Fig. 10 Detection of 150 μm width slots of different depths in 5 mm thick RBSN with microradiography. Greatest depth: 412 μm, lowest depth detected: 21 μm (cf. Fig. 4)

Fig. 11a Surface defects in RBSN turbine blades

Fig. 11b Pores in a RBSN stator, diameter of the marked wire: .30 μm

Fig. 12 Microradiographs of seeded defects in HPSN (Fe, C) and HPSC (Fe) (from left to right). Sample thickness = 3.5 mm.
DISCUSSION

For the analysis of structure heterogeneities and single defects in ceramics like Si₃N₄ and SiC different NDE methods are well suited. Original parts of the gas turbine (e.g. stator, rotor, combustor) with partly complicated shapes and varying thicknesses create difficulties for the application in practice. Ultrasonic free and guided waves and especially microfocus X-rays have advantages over other methods. At the moment the most advanced technique with at the same time high resolution and greater specimen volume analysed in a relatively short time seems to be the micro-radiography. High resolution image intensifier instead of films should enable an easier imaging of defect areas. Additionally, stereo-microradiography (with two photographs from two different positions) should enable to locate the defect inside the component.

Some advantages can be seen, too, for the characterization of the homogeneity of the fabrication process if lots of specimens are analysed with vibration measurements. For one of the most interesting points, the correlation between strength and NDE results, more experiments have to be done.

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SUMMARY DISCUSSION  
(K. Goebbels)

John Schuldies (Airresearch): Could you comment on why you think vibrational resonance testing of a rod is better than just absolute velocity measurements?

Klaus Goebbels: It is better. I think only it is easy if you have several hundred samples from the same propagation process from the same material out from the beginning, and then you are making easy impact testing and measurements, and if there are some samples that are lying outside the scatter of most of the data, I think that you can then take them away and concentrate further on the others.

John Schuldies: I guess the purpose of my comment was that you still got to make a translation later on from the test bar to the real piece. If so, if you came up with a means of velocity very accurately, you could immediately make that transition to the actual piece and confirm that the properties of the test bar are the same as the components that you are using. That was the reason for my comment.

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