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FLAW CHARACTERIZATION IN STRUCTURAL CERAMICS USING SCANNING LASER ACOUSTIC MICROSCOPY

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ABSTRACT

The ability of scanning laser acoustic microscopy (SLAM) to characterize artificially seeded voids in sintered silicon nitride structural ceramic specimens was investigated. The voids ranged from 20 to 430 μm in diameter and were embedded up to 2 mm beneath the surface of the specimens. Probability of detection was determined as a function of void depth and size. Trigonometric relationships and Airy's diffraction theory were used to obtain predictions of void depth and size from acoustic diffraction patterns produced by the voids. Agreement was observed between actual and predicted void depths. However, predicted void diameters were generally much greater than actual diameters. Precise diameter predictions are difficult to obtain because of measurement uncertainty and the limitations of the 100-MHz SLAM applied to typical ceramic specimens.

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OVERVIEW

FLAW CHARACTERIZATION IN STRUCTURAL CERAMICS USING SCANNING LASER ACOUSTIC MICROSCOPY

OBJECTIVE: DETERMINE ABILITY OF SLAM TO CHARACTERIZE (SIZE, DEPTH) INTERNAL FLAWS IN CERAMIC SPECIMENS

EXPERIMENTAL APPROACH: SINTERED SILICON NITRIDE SPECIMENS WITH ARTIFICIALLY SEEDED INTERNAL VOIDS

RESULTS AND CONCLUSION:

(1) LARGE MEASUREMENT UNCERTAINTY DUE TO EXPERIMENTAL CONFIGURATION

(2) UNCERTAINTY SEVERELY AFFECTED VOID SIZE PREDICTIONS WHICH DEVIATED MORE THAN 100% FROM ACTUAL SIZES

(3) REASONABLE AGREEMENT BETWEEN PREDICTED AND ACTUAL VOID DEPTHS

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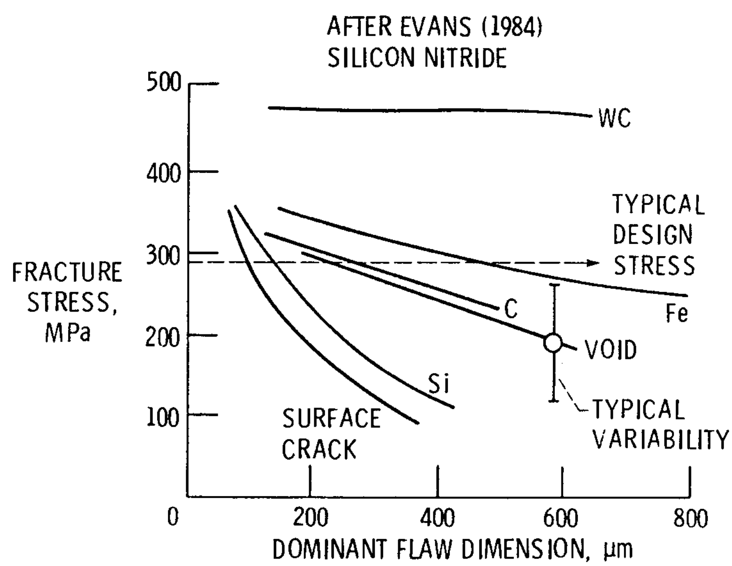
FRACTURE STRENGTH VERSUS FLAW TYPE AND SIZE

Experiment Rationale: Relationships between fracture strengths and flaw types, sizes, shapes, and locations are being actively investigated to promote understanding of structural ceramic mechanical behavior. Therefore, it is essential to develop accurate flaw characterization techniques for use on as-fabricated specimens.



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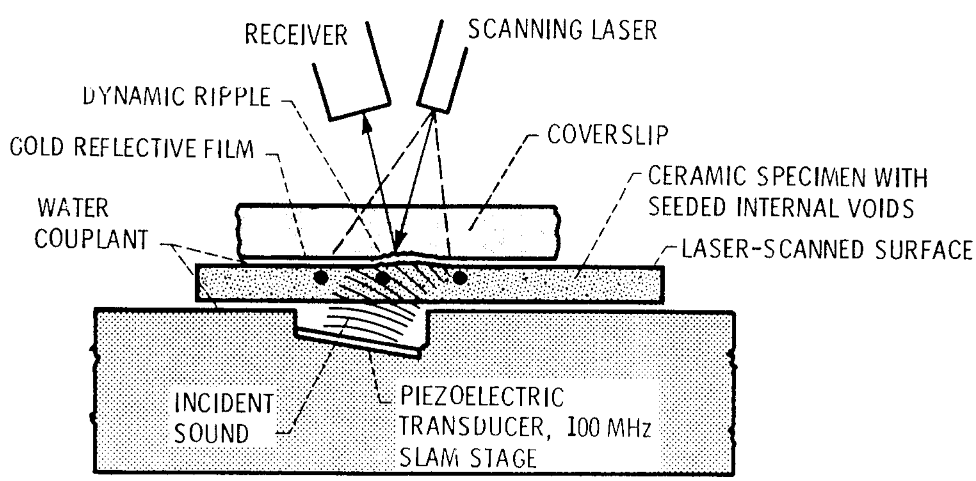
SCANNING LASER ACOUSTIC MICROSCOPY (SLAM)

The experimental setup for scanning laser acoustic microscopy of ceramic specimens is shown below.



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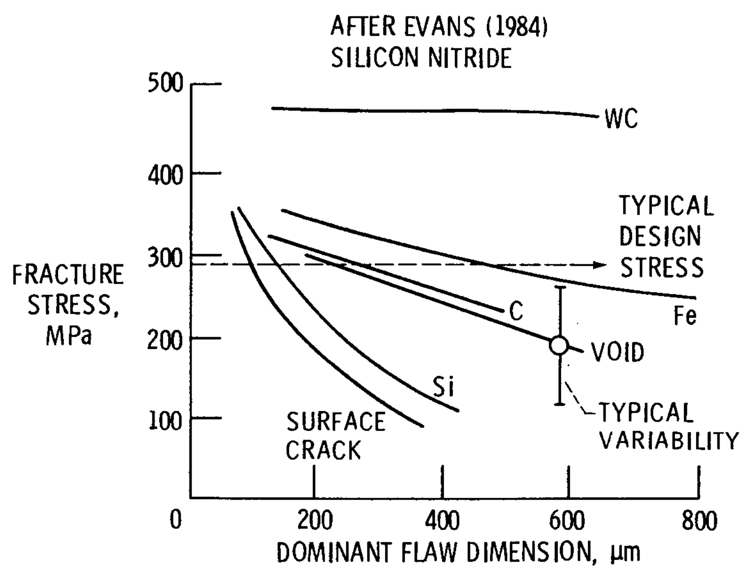
POSTER PRESENTATION

PURPOSE OF STUDY

Structural ceramics exhibit wide variability in strength and low fracture toughness because of their brittle nature (Lenoe, 1983; Shannon, 1981; and Salem, 1985). Generally, failure is attributed to discrete flaws such as microcracks, voids, impurities, and oversized grains (Evans, 1984; Heitman, 1983; and Sanders, 1986). The relationships between fracture strengths and flaw types, sizes, shapes, and locations are being actively investigated for structural ceramics as indicated in the figure. Therefore, the ability to accurately characterize existing flaws in these materials by nondestructive evaluation (NDE) techniques has become extremely important.



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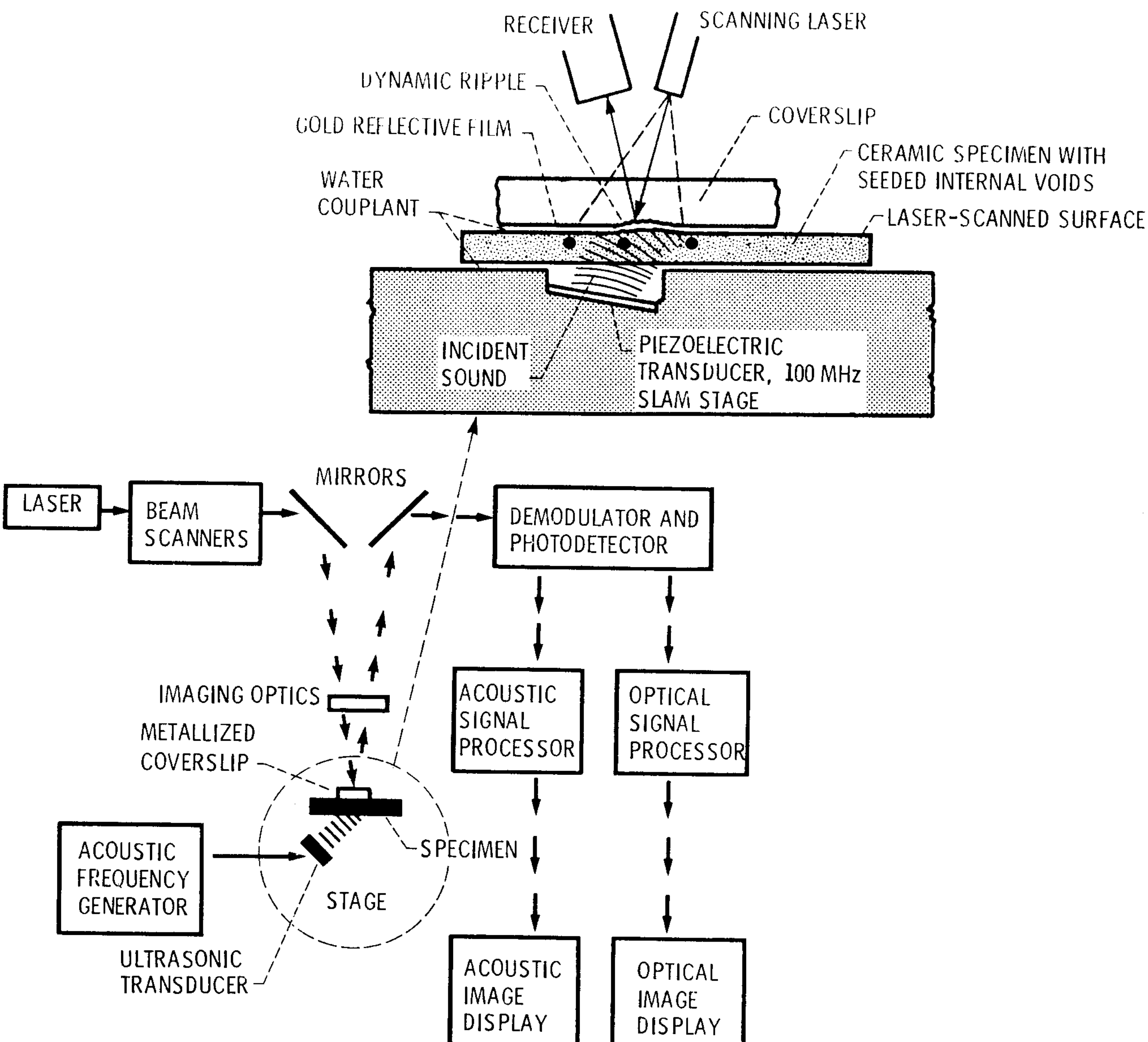
SCANNING LASER ACOUSTIC MICROSCOPY (SLAM)

The following is a brief description of the principles of operation of SLAM. Continuous 100-MHz ultrasonic waves traveling through a specimen produce micro-distortions on the specimen surface farthest from the transducer. The distortion pattern is determined by the microstructural, bulk, and surface features of the material. A laser beam constantly raster scans a small area of the specimen. The laser beam, angularly modulated by the distortion pattern, is reflected to a photodetector and converted to an electronic signal. In this manner, an "acoustic" image of the specimen, including surface and internal flaws such as voids, inclusions, and cracks, is obtained and displayed on a video monitor in real time at approximately 100x (Roth, 1986a and b; Roth 1987; and Generazio, 1986).



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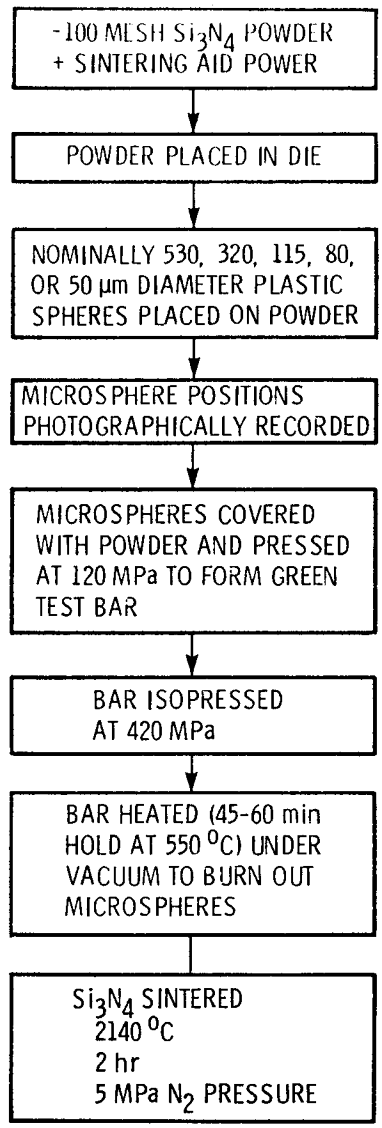
SPECIMEN PREPARATION

Sintered Si_3N_4 specimens containing seeded internal voids were fabricated by using the processing steps shown in the figure and described in detail by Baaklini (1986). Briefly, plastic microspheres of various sizes were embedded in green specimens and later burned out to create voids within sintered specimens.



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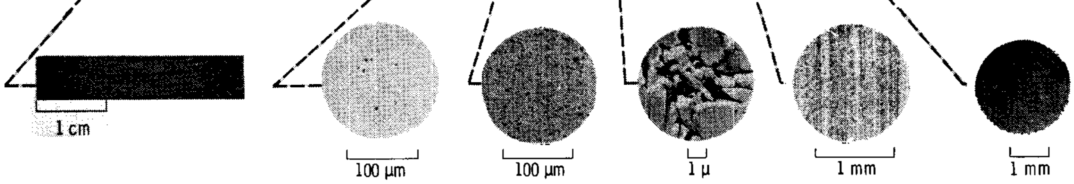
CHARACTERIZATION OF SPECIMENS

The seven sintered silicon nitride specimens were microstructurally characterized as shown in the table. The 12 internal voids seeded in the specimens ranged from 20 to 430 μm in diameter and from 0 to 2 mm in depth (Roth, 1987).

CHARACTERIZATION OF SPECIMENS

TEST BAR MATERIAL	NUMBER OF TEST BARS USED	LENGTH X WIDTH, (mm)	THICKNESS, (mm)	DENSITY		POROSITY DISTRIBUTION	AVERAGE GRAIN SIZE, (μm)	PEAK-TO-VALLEY ROUGHNESS, (μm), OF -		SEEDED INTERNAL VOIDS[g]		
				(g/cc)	% THEORETICAL			GROUND SURFACE	AS-FIRED SURFACE	TOTAL NUMBER	DIAMETER, (μm)	DEPTH BELOW SPECIMEN SURFACE (mm)
SINTERED Si_3N_4 (SSN)	7	30x6	2-4	3.230	~100	LESS AT EDGE THAN AT CENTER	0.5-1.5[c]	RELATIVELY ORDERED 0.5-3.5[e]	RANDOMLY ORIENTED 3.0-7.5[e]	12	20-430	0-2



[a] OPTICAL PHOTOGRAPH (GROUND SURFACE SHOWN).
[b] OPTICAL MICROGRAPH OF METALLOGRAPHICALLY POLISHED SECTION (BLACK SPOTS INDICATE POROSITY).
[c] AVERAGE GRAIN SIZES OBTAINED USING THE HEYN INTERCEPT (MEAN FREE PATH) METHOD GIVEN IN ASTM E112-81.
[d] TRANSMISSION ELECTRON MICROGRAPH OF REPLICA OF METALLOGRAPHICALLY POLISHED AND ETCHED SECTION
[e] SURFACE PROFILE (USING A 12.5 μm DIAMETER DIAMOND STYLUS) (PERPENDICULAR TO GRINDING MARKS FOR SPECIMEN WITH GROUND SURFACE).
[f] OPTICAL MICROGRAPH.
[g] CHARACTERIZED AFTER SINTERING SPECIMEN AND EXPOSING VOIDS TO SURFACE.

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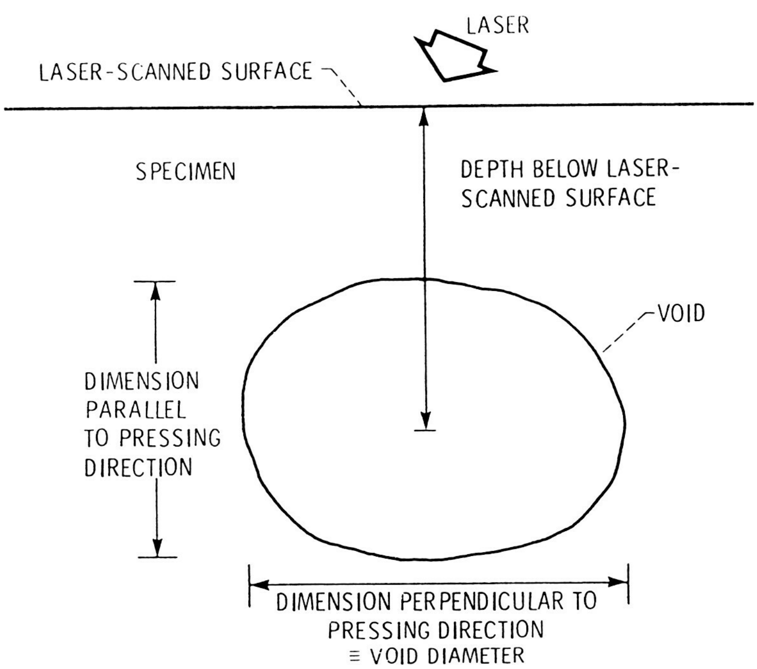
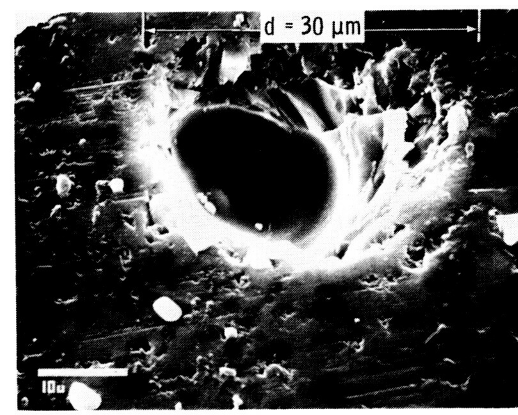
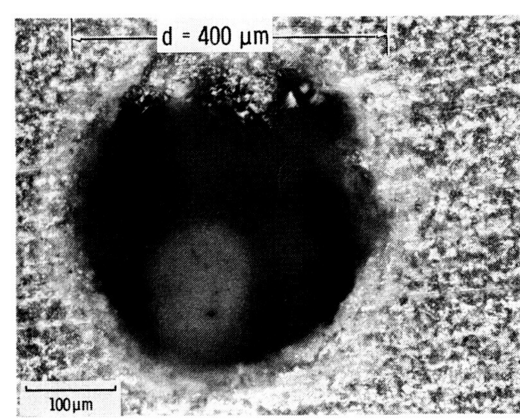
SEEDED INTERNAL VOIDS

The seeded internal voids were exposed to the surface by grinding. At this point, the void dimensions were measured optically, and the void depths at the various SLAM inspections were determined (Roth, 1987).





SEM MICROGRAPH (SILICON NITRIDE)



OPTICAL MICROGRAPH (SILICON NITRIDE)

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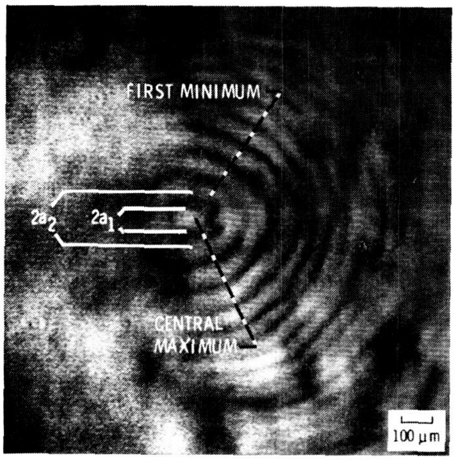
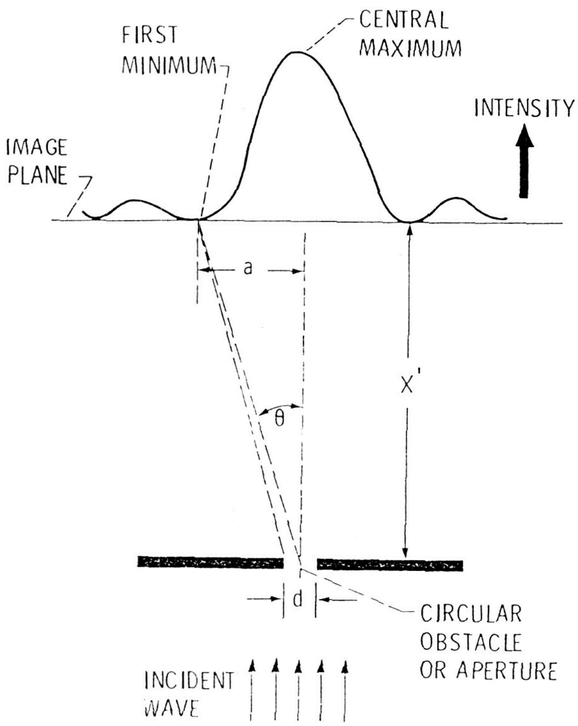
ACOUSTIC IMAGE OF INTERNAL VOID

The acoustic image of an internal flaw often consists of a diffraction pattern rather than a facsimile image of the flaw. In this case, it is especially difficult to characterize the flaw. However, techniques have been investigated from which it is theoretically possible to predict flaw shape, size, and depth by using acoustic diffraction patterns (Generazio, 1986; Roth, 1986b and 1987). Measurements obtained from the acoustic images were used to obtain predictions of void diameter and depth (Roth, 1987).





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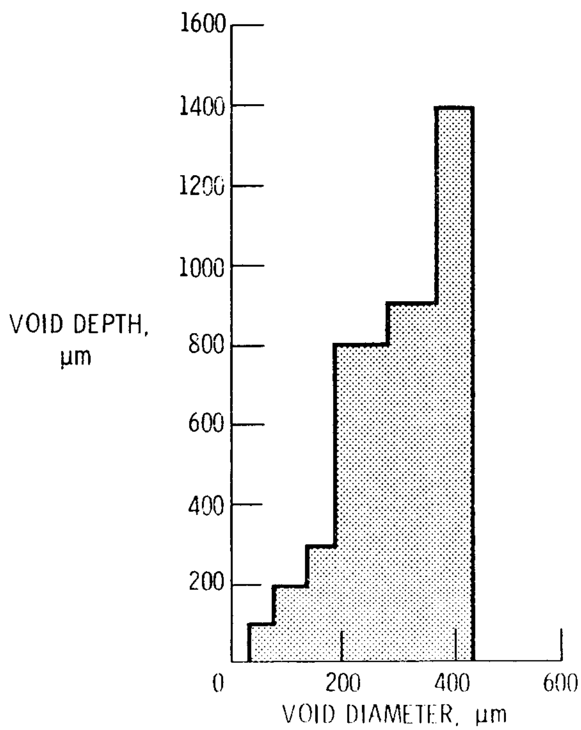
PROBABILITY OF DETECTION OF INTERNAL VOIDS

Probability-of-detection data for the seeded internal voids as a function of void diameter and void depth is shown (Roth, 1986b). The range of depths and diameters for which 90 percent or higher probability of detection (at a 95 percent confidence level) was achieved is indicated by the outlined region for sintered silicon nitride specimens (Roth, 1986).



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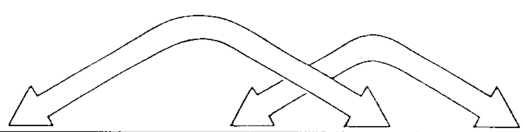
PREDICTED VOID DEPTHS AND DIAMETERS USING SLAM

Predictions of depth and diameter were determined for 4 voids at 23 depths. The 23 predicted void depths deviated less than 70 percent from actual depths and 17 were within 20 percent of actual depths. Predicted void diameters deviated more than 100 percent from actual values in all cases but one (Roth, 1987). Examples are shown in the table.



ACTUAL VOID DIAMETER, µm	AT	ACTUAL VOID DEPTH, µm	PREDICTED DIAMETER, µm	PREDICTED DEPTH, µm
403	AT	1897	1113	1917
252	AT	568	790	616
139	AT	197	571	153
30	AT	79	135	54

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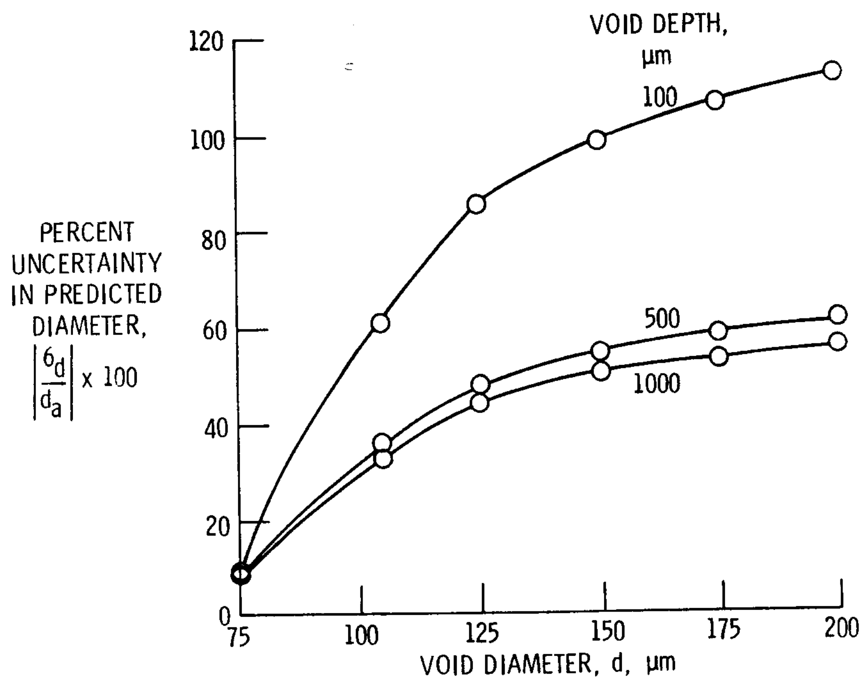
MEASUREMENT UNCERTAINTY

Measurements obtained from the acoustic images were used to obtain predictions of void diameter and depth. Precise measurements were not possible. Void diameter predictions were more severely affected by the measurement uncertainty than were void depth predictions. Measurement uncertainty was expected to increase with increasing void diameter and decreasing void depth (Roth, 1987).



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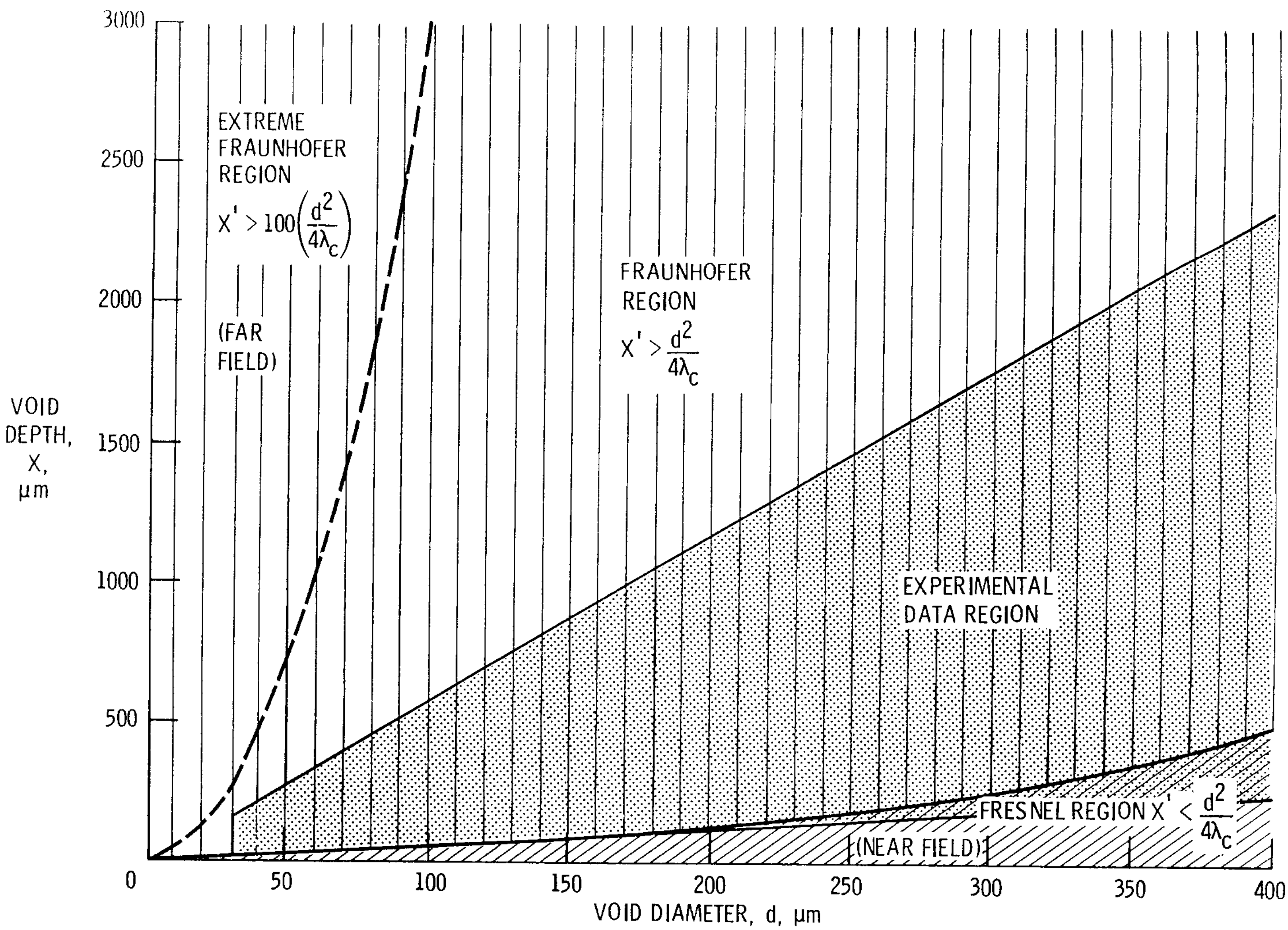
VALIDITY OF RELATION USED TO PREDICT VOID DIAMETER

The range of void depths and diameters that produced discernible diffraction patterns is shown in the figure below in the area labeled "experimental data region." It is expected that for voids of these diameters and depths, the relation used to predict void diameter is of questionable validity. The relation is most valid for void depths and diameters in the "extreme Fraunhofer region" (far field). Extreme Fraunhofer conditions are difficult to approach with the 100-MHz SLAM configuration applied to typical ceramic specimens (Roth, 1987).



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CONCLUSION

Probability of detection was determined for seeded internal voids in sintered silicon nitride specimens as a function of void size and depth. The acoustic images produced by the voids were used to obtain predictions of void size and depth.

The measurements taken from the acoustic images of the internal voids had large uncertainty associated with them (Roth, 1987). The measurement uncertainty severely affected the prediction of void diameter. Additionally, the relation used to predict void diameter may be of questionable validity for the conditions of this experiment. As a result, predicted void diameters were generally much larger than actual void diameters (Roth, 1987). The measurement uncertainty affected the prediction of void depth less severely. Hence, reasonable agreement was observed between predicted and actual void depths (Roth, 1987).

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