Nondestructive Microwave Evaluation of Ceramics

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Abstract—The ability of microwave energy to penetrate some kinds of ceramic materials at frequencies of 100 GHz and above suggests that microwave techniques may be useful for the nondestructive evaluation (NDE) of such materials. This paper presents experimental results that demonstrate the basic feasibility of using these techniques to detect and locate various types of inclusions (including voids) in silicon nitride Si_3N_4. Inclusions as small as 0.005 in in diameter have been detected.

I. Introduction

IN RECENT years a great deal of effort has gone into the development of ceramic materials for high-performance applications such as gas turbines. The primary materials in this category are silicon nitride (Si_3N_4), silicon carbide (SiC), and lithium-aluminum silicate (L-A-S). Such ceramics are brittle, and their strength varies with the presence of defects such as cracks, voids, and inclusions in the material. Since it is impossible to completely eliminate such defects, the economic feasibility of using these materials will depend on the availability of nondestructive evaluation (NDE) techniques for locating these defects and determining if the defects that are present can be tolerated.

The critical size of a flaw in a typical ceramic material such as Si_3N_4 is considered to be about 100–200 μm at room temperature. However, at 1400°C this critical dimension reduces to about 25 μm for some applications [1]. This latter dimension represents a severe challenge for any NDE technique, especially since the grain size in high-density hot-pressed material can be as large as 5 μm, and as large as 100 μm in cold-formed material. In addition, the criticality of a flaw depends upon its type, so a good NDE technique should also be capable of classifying the flaws that it detects. Besides voids, cracks, and density laminations, there can occur inclusions of various materials such as tungsten carbide, iron, tungsten, silicon, and carbon.

It would seem that, in principle, a combination of X-ray radiography and very high-frequency (100-MHz or greater) ultrasonics [2] can probably meet this demand for high resolution, high sensitivity, and defect classification. However, in practice, there are several problems. X-ray radiography is expensive and time consuming. The highest frequency that is practical for ultrasonic techniques is limited by surface roughness and grain-boundary scattering. Even for smooth surfaces, great care must be taken in coupling a high-frequency ultrasonic transducer to the ceramic. This requirement makes it difficult to move the transducer (or sample) for scanning purposes and limits application of the technique to simple geometries.

In view of these limitations, there is a need to develop other NDE techniques to complement the X-ray and ultrasonic techniques already in use. In this paper, the possibility of using millimeter-wavelength electromagnetic waves for this purpose is explored. That such a study should be conducted is suggested by the following considerations.

1) Microwaves can penetrate low-loss dielectric materials, and will interact with local variations in the dielectric properties of these materials.

2) Microwave transducers (antennas) do not need to be in physical contact with the material to be inspected, nor do they require a coupling medium other than air.

3) Millimeter-wave technology has improved considerably in recent years in the areas of availability, performance, and cost.

The application of microwave-frequency electromagnetic techniques to various NDE problems has been discussed and studied extensively during the last 15 years [3]–[5]. Microwaves have been used to probe dielectric materials to measure changes in thickness and material composition, and to detect and locate interior flaws such as voids and inclusions. In the case of ceramic turbine parts, the finding and characterization of flaws is of most interest, although there is also a need for quality control in material processing. In the past, the use of microwave frequencies for the detection of small flaws has suffered from the limited sensitivity and resolution capabilities that are dictated by the relatively long wavelengths involved. The use of millimeter-wave frequencies, however, should alleviate this situation, and for this reason it appears worthwhile to take a fresh look at the use of high-frequency electromagnetic techniques for the NDE of ceramics.

Thus the purpose of this paper is to present some results of an experimental study that was undertaken to determine the feasibility of using millimeter waves to detect, localize, and characterize small defects in ceramics such as Si_3N_4. First, some general considerations involved in

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microwave NDE for ceramics are discussed in Section II. Then, experimental results that were obtained using seeded plates of Si_3N_4 are presented in Section III. Finally, the conclusions derived from the work are summarized in Section IV.

II. General Considerations in the Microwave NDE of Ceramics

Microwave nondestructive evaluation techniques can be applied to dielectric materials to measure either gross material and geometrical properties (e.g., dielectric constant, dissipation factor, and plate thickness) or localized properties associated with defects [5]. In the evaluation of ceramic materials such as Si_3N_4, one is primarily concerned with the detection of small defects. In this case, the basic physical effect involved is the scattering of electromagnetic waves by the defect.

For detection of an interior flaw to be possible, the microwave energy must be able to both enter and leave the ceramic material. First of all, this requirement means that the propagation loss inside the material must not be too large. Secondly, the microwave energy must not be totally reflected from the surface of the material, nor must it be trapped inside the material. The theory for the reflection of a plane wave at an air/dielectric interface [6] shows that some energy will always enter the dielectric material except at grazing incidence. Use of the Brewster angle when the electric-field vector is polarized parallel to the plane of incidence will maximize the transmission of energy through a plane parallel-sided dielectric plate. However, it is often simplest in practice to choose normal incidence even though this procedure results in some reflection loss. Typically, however, this loss is on the order of 20 percent and can be tolerated.

Since there is always some reflection at a dielectric/air interface (except under Brewster conditions), in general there will be standing waves inside the dielectric material. These standing waves will be particularly enhanced if the dielectric specimen is a plate with plane, parallel sides. The scattering from a defect depends on the relative position of the defect in the standing wave. Hence, since the positions of the maxima and minima in a standing wave vary with frequency, the scattering will exhibit a position-related frequency dependence that may mask any intrinsic defect-related frequency dependence.

Scattering measurements are usually classified according to the arrangement of the transmitting and receiving antennas, and according to the operating frequency. There are three basic antenna arrangements that can be considered for NDE: backscatter-monostatic, backscatter-bistatic, and forward-scatter-bistatic. In the latter two of these arrangements the transmitting and receiving antennas can be either copolarized or cross-polarized.

Microwave antennas for NDE applications can be characterized as either predominantly radiating or predominantly nonradiating. An example of the radiating type would be a horn or open-ended waveguide. A small aperture in the side of a waveguide or resonant cavity is an example of an antenna that radiates very little. In this case a defect must be close to the aperture so that interaction can take place between the defect and the reactive near fields around the aperture.

In the microwave NDE (as in ultrasonic NDE), sensitivity (the ability to detect a defect) depends mainly on two factors: 1) the amount of scattering engendered by the defect, and 2) the ability of the detection technique to separate the desired scattered wave from undesired scattered waves (clutter). Electronic noise is also a factor in determining sensitivity, but is usually less important than clutter.

In general, the theoretical determination of the scattering from an arbitrary shape and type of defect is a difficult problem requiring computer analysis [7]. However, if it is assumed that the defects can be approximated as either metal or dielectric spheres, some rather simple formulas can be used if the wavelength of the electromagnetic radiation is much larger than the sphere radius (Rayleigh region). Since the defects of most interest in the NDE of ceramics have maximum dimensions between 25 and 125 μm, one finds that this assumption is justified even for a frequency of 100 GHz. A few calculations using the Rayleigh formulas for scattering cross section [8] show that a major consideration in the microwave NDE of ceramics will be the detection of relatively small signals.

If the transmit and receive antennas are copolarized, one generally finds that the detected signal suffers from poor contrast; that is, the small scattered signal is buried in a large specularly reflected or transmitted signal. Thus one is forced to use a "bridge" technique to improve contrast.[1]

There are several different kinds of bridge techniques. One simple kind of bridge would be one where a portion of the transmitted signal with the proper amplitude and phase is fed directly to the receiver so that a signal received in the absence of a defect is canceled. Another technique uses mode conversion—e.g., the detector can be made to be responsive only to a wave whose field configuration is orthogonal to that of the incident wave [9]. A third technique imparts a modulation to the wave scattered by a defect by appropriately vibrating [10] or rotating the sample being inspected. The modulated wave can then be separated from any large unmodulated scattered waves that are also received by using appropriate filtering.

The use of a feed-forward canceller is not very practical at high microwave frequencies because of the sensitivity of the null condition to the position of the specular scatterer. In this case it is difficult to maintain a null as

[1]The use of side scattering would seem to be an obvious choice for the purpose of improving contrast. However, in practice, there can be side scattering from the boundaries of the test object that is of the same order of magnitude as the sidescattering from the defect. This situation makes it difficult to identify a bona fide defect. In addition, there can be nulls in the side-scattering pattern that could cause a defect to be overlooked.

the ceramic part is scanned in front of the antennas, particularly if the part has nonplanar surfaces.

Of the remaining two contrast-improving techniques, it is simplest to implement the mode-conversion technique by using separate receiving and transmitting antennas and orienting them with their polarizations orthogonal to one another. Although a monostatic-backscatter scheme appears attractive because only one sensor is required for both illumination and detection, this advantage is offset slightly by the circuit complexity required to separate the orthogonal transmitting and receiving modes. Therefore, the bistatic-forward-scatter scheme (using cross-polarized antennas) was used to obtain the experimental results described in this paper. Although scatterers with certain geometrical symmetries will not couple orthogonal modes, there is very little probability that these symmetries will occur for real defects in ceramics.

The above discussion has implicitly assumed the use of continuous-wave (CW) measurements. For the NDE of relatively thin ceramic parts (a few wavelengths thick or less), there is no advantage to be gained from the use of pulsed measurements (or, equivalently, wideband swept-frequency measurements). Normally, pulsed measurements would be used to separate reflections (in the time domain) from various scattering centers along the direction of propagation (range gating). However, the shortest practical pulse that can be produced and radiated results in a spatial extent for the pulse of the order of 10 wavelengths in the ceramic material. Thus, for a part that is only a few wavelengths thick, range gating cannot be accomplished.

An important criterion for evaluating the effectiveness of any defect-locating NDE system is that of spatial resolution, namely, the ability to differentiate between two closely spaced defects. As discussed previously, for thin ceramic parts microwave NDE does not provide any spatial resolution in the nominal direction of propagation of the illuminating wave. In the scanning plane (defined as a plane containing all the positions of a reference point on the sample that are occupied as the sample undergoes rectilinear translation), spatial resolution is determined by the active areas of the transmitting and receiving antennas. At distances sufficiently far removed from the test piece, the "active" area is essentially determined by the far-field beamwidth of the radiating antenna.

The best spatial resolution using a far-field antenna is obtained by incorporating a lens into a microwave horn [11]. Such a lens can be fabricated from polystyrene, and produces a 3-dB spot size having a diameter approximately equal to one free-space wavelength at the operating frequency. Focal lengths of such lenses are typically 10 wavelengths.[2]

[2]In principle, a "synthetic array" technique could be used to improve resolution along the line of scan. In this technique, the amplitude and phase of the scattered waves received at a number of successive sample points along the scan line are stored and then the data are processed by computer to form a high-resolution image. This technique works fine in a radar application, but probably could not be used in the special geometries associated with ceramics NDE.

An alternative approach, and one that can yield comparable magnitudes of resolution, is to move the test piece into the near fields of the antennas. The size of the active area depends on the nature of the fringing fields around the aperture and the distance between the aperture plane and the scanning plane.

One of the simplest types of antenna is an open-ended waveguide. Typical aperture dimensions for such an antenna at 100 GHz are about 0.4 by 0.8 free-space wavelengths, or 1.25 by 2.5 mm. Higher resolutions can be obtained by reducing the aperture size [10] (i.e., by using an electrically small antenna). However, this approach generally results in a tradeoff between sensitivity and resolution. In addition, an electrically small antenna radiates relatively very little energy, and the near fields associated with it fall off very rapidly with distance from the antenna. Thus, when using such an antenna, one runs the risk of not detecting a defect inside the ceramic material.

An important advantage of obtaining a good spatial resolution is the minimization of spurious scattering caused by variations in the gross geometry of the ceramic part. Signals due to slow variations in geometry can be removed by filtering, but a small, active sensor area is the only solution if one wishes to inspect a part very close to a sharp change in boundary surface (such as an edge).

Because of their simplicity and convenience, open-ended waveguides were used as antennas in the experiments to be reported here. In general, the spatial resolution achievable in the scanning plane with these antennas depended on the thickness of the ceramic part that was inspected. For example, for a scanning plane located 4 mm from the aperture plane of the open-ended waveguide, the 3-dB beamwidth [12] in the scanning plane at 100 GHz is about 14 mm in the E plane, and 6 mm in the H plane. This resolution was adequate for the purposes of this feasibility study.

III. Experimental Detection and Location of Defects in Si_3N_4

In accordance with the general considerations discussed in Section II, the feasibility of detecting and locating inclusions in Si_3N_4 was studied using measurements of cross-polarized forward scattering in W band (75 to 110 GHz). Usually both of the open-ended waveguide apertures were placed as close to the surface of the material under test as possible in order to obtain the best spatial resolution.

A block diagram of the experimental arrangement is shown in Fig. 1. Physically, the transmitter and receiver antenna apertures were placed directly opposite one another, with the feed waveguides aligned along a vertical axis and cross-polarized. The ceramic sample was then placed between the transmitter and receiver and moved in a horizontal plane. The horizontal scanning mechanism that held the sample provided a continuous motor-driven scan in one direction and movement in precise steps in the orthogonal direction. The detected signal was sampled at

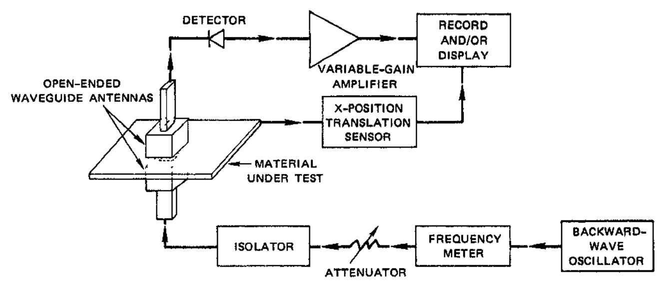


Fig. 1. Experimental arrangement for cross-polarized transmission measurements.

a set of points in the scan area, and then recorded in an FM format on magnetic tape. These data were then subsequently digitized and processed with a computer to produce a display of the resulting *C*-scan[3] image. Typically, about 250 points were sampled over a 10-cm scan length, giving a spatial sampling resolution of 0.4 mm. This degree of spatial resolution was more than adequate, since the beamwidth of the open-ended-waveguide antenna in the defect plane was at least 10 times larger than the scan increment. The Si_3N_4 samples that were examined using this system were all in the form of flat plates. Most of them were made of hot-pressed material[4] that had been seeded with various types and sizes of inclusions at specific locations. The other ceramic material of major interest, SiC, could be examined internally with microwaves because of its excessive loss.

Some preliminary measurements were conducted prior to attempting the detection and imaging of the inclusions. First, simple measurements of the power reflected and transmitted by the plates were made to assure that the reflection and propagation losses were not so large as to preclude detection of the inclusions. It was found that the fraction of incident power reflected was what would be expected for a material having a relative dielectric constant of about 7 (about 20 percent), and that the propagation loss of Si_3N_4 in *W* band was negligible.

Second, swept-frequency scattering measurements were made to determine the best operating frequency for each type of inclusion. Typical frequency responses for four different types of inclusions in one of the seeded plates are shown in Fig. 2. The nominal diameter of these particular inclusions was 0.020 in. It can be seen from the figure that there are significant variations in these frequency responses, and this fact may prove useful for flaw identification. However, as mentioned before, these frequency responses are determined in part by the reflections from the surfaces of the ceramic plate containing the inclusions, and thus it may be difficult to separate the flaw-dependent information from these responses.

On the basis of these swept-frequency measurements, a compromise frequency of 94 GHz was chosen and a *C* scan of the portion of a hot-pressed plate containing both 0.020- and 0.005-in diameter inclusions was made. Fig. 3(a) shows the area covered by the scan and the intended flaw locations. Fig. 3(b) shows the portions of the scan area that produce a scattered signal greater than an arbitrarily selected threshold value. Finally, Fig. 3(c) shows the amplitude of the scattered signal as a function of position within the scan area.

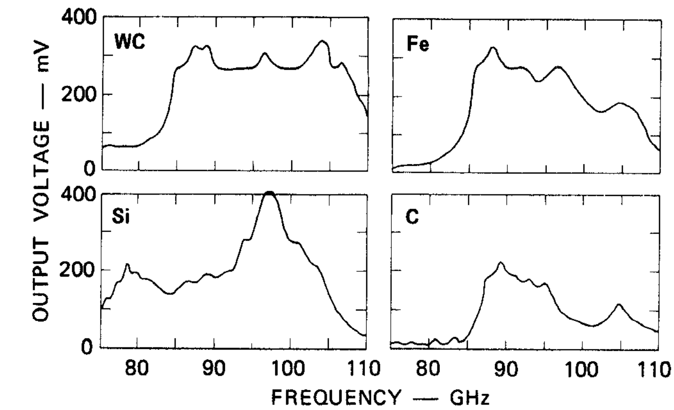


Fig. 2. Frequency dependence of the cross-polarized forward scattering from four types of inclusions in a hot-pressed Si_3N_4 plate.

Several features of this *C* scan are noteworthy. First, all of the 0.020-in diameter flaws were detected. Iron provides the strongest signal, and is the only 0.005-in diameter flaw that is clearly detected in this figure (the other small flaws become more apparent if the frequency is changed). Second, X-rays show that diffusion of the iron inclusion during hot pressing produces an irregularly shaped scatterer that causes the spatial extent for this flaw to appear overly large in the microwave *C* scan. Finally, it appears that a crack-like flaw is present between the 0.020-in diameter iron and silicon inclusions. Previous examinations using X-ray, ultrasonic, and dye penetrant techniques did not reveal the presence of such a flaw. If this flaw is indeed real, it would indicate the superior sensitivity of the microwave technique for detecting this type of flaw.

Fig. 4 shows a similar microwave *C* scan, but for a surface-ground hot-pressed Si_3N_4 plate that contained different types and densities of inclusions. The dots in the figure are a purely schematic indication of the intended flaw locations and density. All of the 0.005-in diameter inclusions were detected in this scan, but the closer spacing between the inclusions in this plate may have enhanced this detection.

Another scan of the same plate as in Fig. 4 is presented in Fig. 5, but of the area containing the 0.001- through 0.010-in diameter silicon inclusions. The sensitivity of the microwave technique for the detection of unreacted silicon appears to be good, and may be better for this purpose than other techniques. This feature could be important in a process-control application.

These results were all obtained using seeded billets of hot-pressed material. Since reaction-sintered material usually has a larger grain size and more porosity than hot-pressed material, it is of interest to see how these material characteristics might affect the microwave detection of flaws in reaction-sintered material. To this end, a plate of

[3]The term "*C* scan" is commonly used to describe the imagine of material variations in the scanning plane.

[4]NC132, a designation of the Norton Company, Worcester, MA.

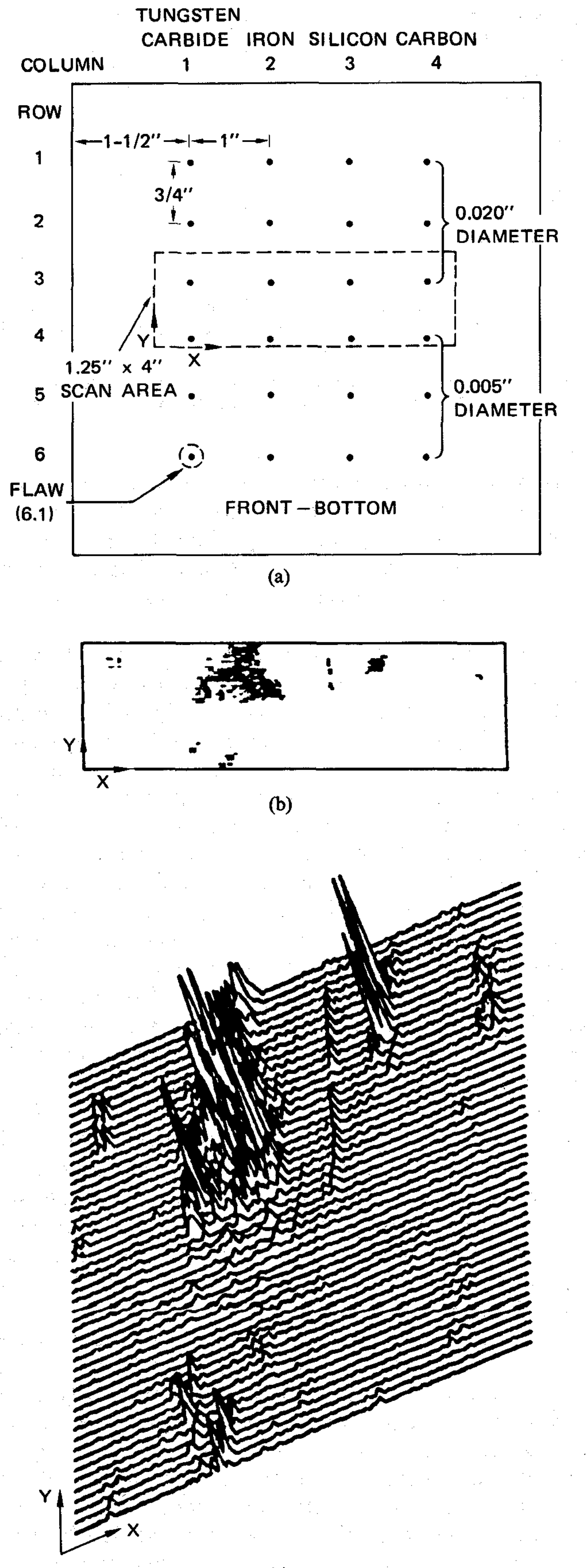


Fig. 3. Microwave cross-polarized transmission *C* scan of four types of inclusions in hot-pressed Si_3N_4. Frequency = 94 GHz. (a) Map of intended flaw locations showing scan area. (b) Excess signal versus position. (c) Amplitude versus position.

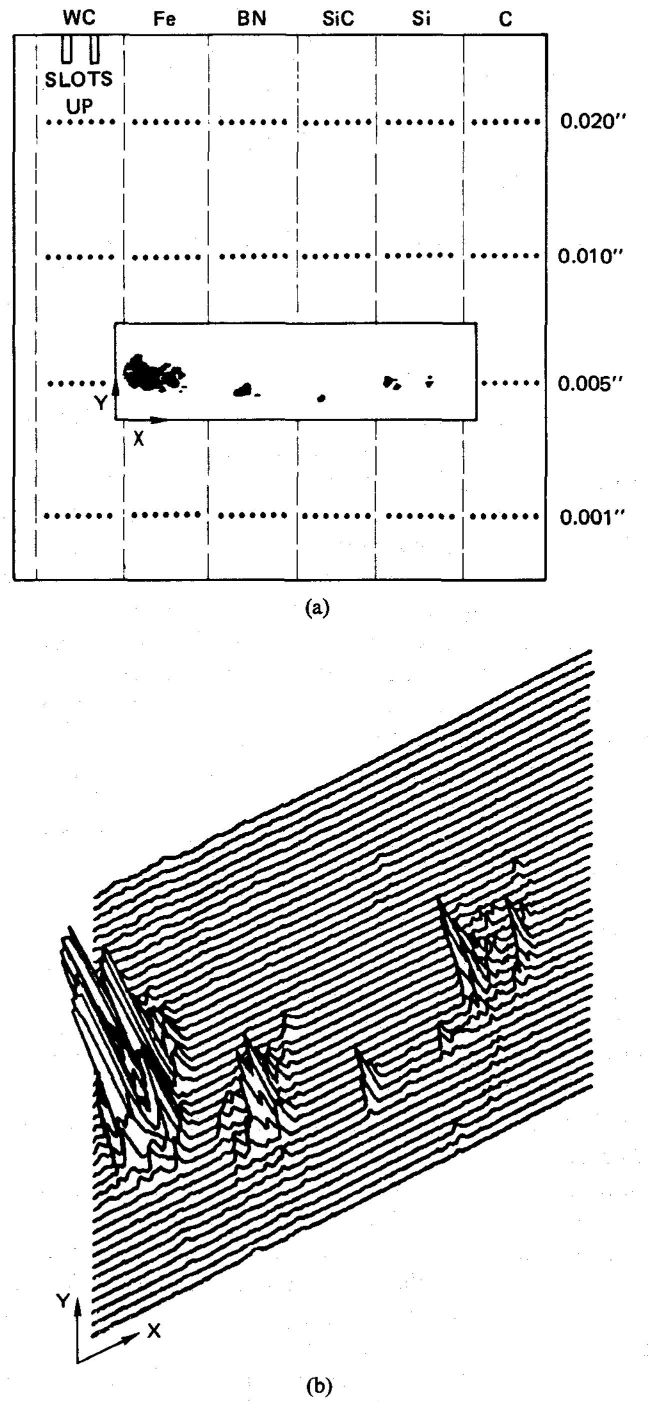
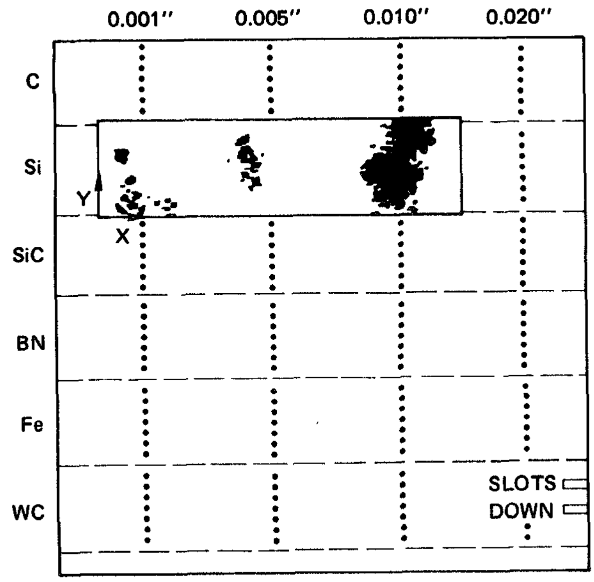


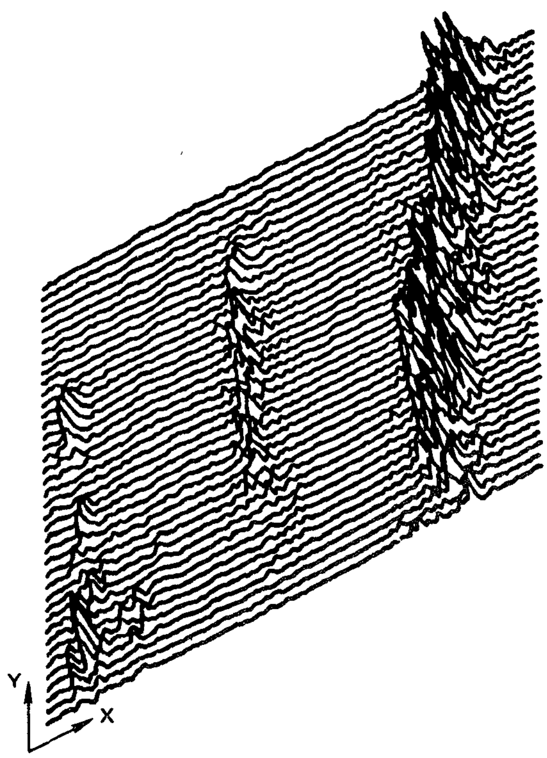
Fig. 4. Microwave cross-polarized transmission *C* scan of four types of inclusions in hot-pressed Si_3N_4. Frequency = 91 GHz. (a) Excess signal versus position. (b) Amplitude versus position.

reaction-sintered Si_3N_4 was obtained having the types, sizes, and intended locations of seeded inclusions shown in Fig. 6. The location of the area scanned using the microwave *C*-scan system is also indicated in the figure.

Fig. 7 shows data obtained for this scan area. Compared with Figs. 3–5, an increase in clutter level can be noticed in this figure. Generally speaking, it was found that the reaction-sintered material produced a higher clutter level than did hot-pressed material. However, the clutter level does not appear to be large enough to mask detection of 0.010-in diameter inclusions. Smaller inclusions were not examined in this billet because of mechani-



(a)



(b)

Fig. 5. Microwave cross-polarized transmission *C* scan of silicon inclusions in hot-pressed Si_3N_4. Frequency=98 GHz. (a) Excess signal versus position. (b) Amplitude versus position.

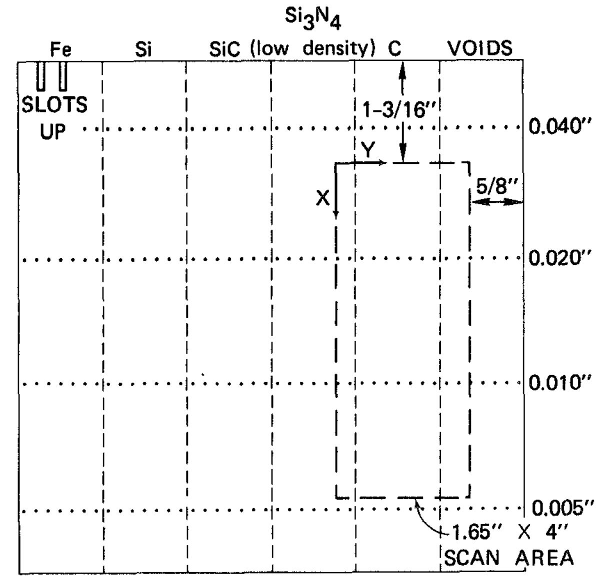
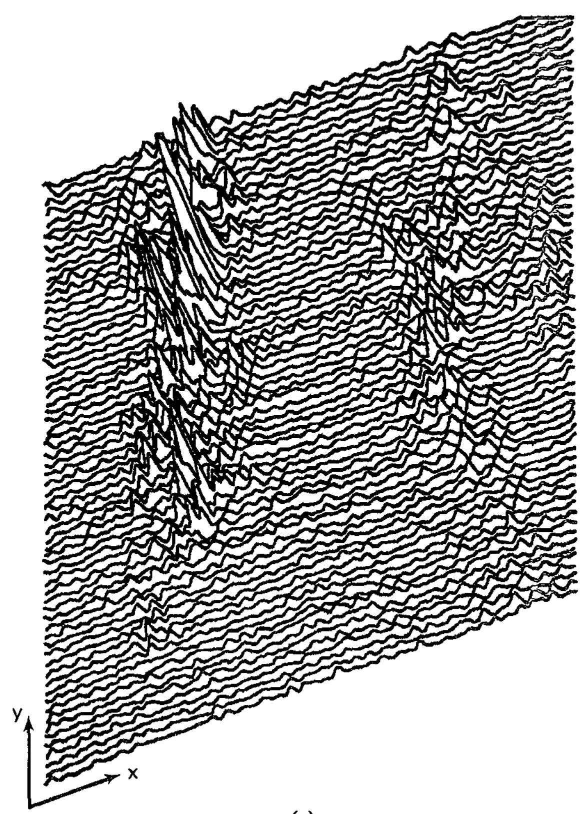


Fig. 6. Intended flaw locations and scan area in reaction-sintered Si_3N_4.



(a)

(b)

Fig. 7. Microwave cross-polarized transmission *C* scan of carbon inclusions and voids in reaction-sintered Si_3N_4. Frequency=92.6 GHz. (a) Amplitude versus position. (b) Excess signal versus position.

cal limitations in the scanning system, but it is clear that inclusions having a size equal to, or less than, the grain or pore size in the material will not be distinguishable from the background.

The same conclusion holds, of course, with regard to the effect of surface roughness. During the program, a relative comparison was made of the scattering from a disk of hot-pressed Si_3N_4 having an as-pressed, grit-blasted surface, and the scattering from the same disk after surface grinding. Qualitatively, the difference in clutter level for the two surface conditions was about the

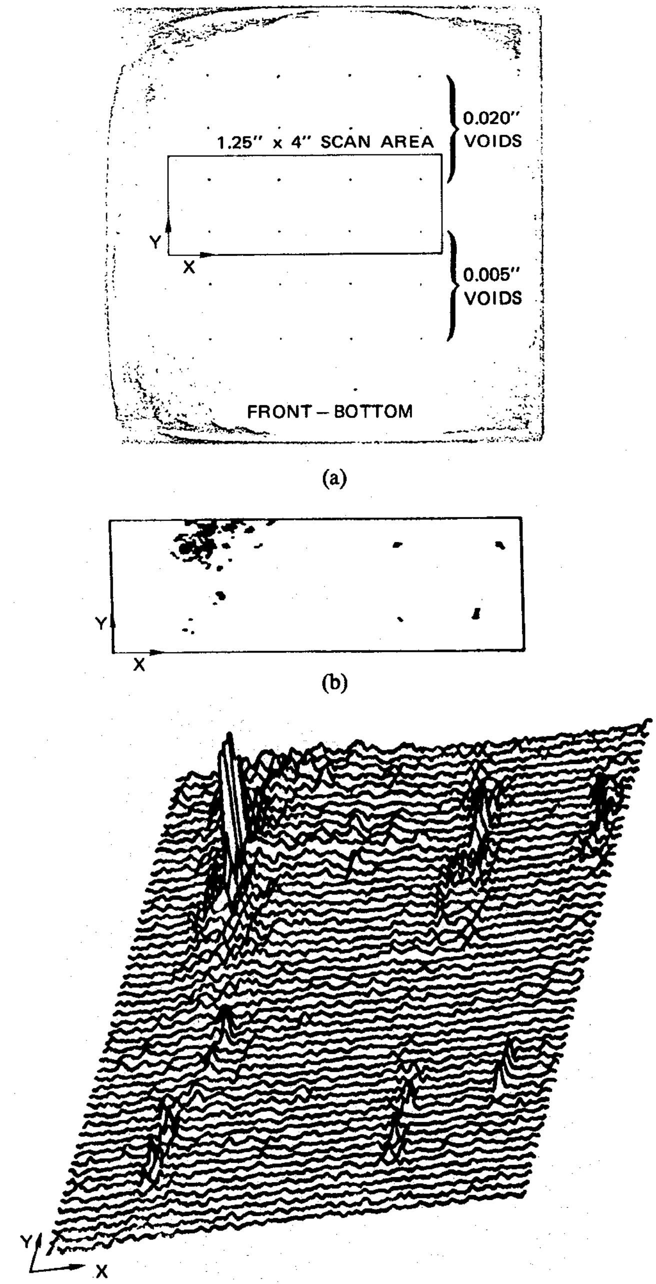


Fig. 8. Microwave cross-polarized transmission C scan showing voids in hot-pressed Si_3N_4. Frequency=94 GHz. (a) Ultrasonic C-scan (focused 25 MHz) map showing void locations and scan area. (b) Excess signal versus position. (c) Amplitude versus position.

same as that observed for hot-pressed and reaction-sintered material.

It appears from Fig. 7 that voids as well as inclusions can be detected in Si_3N_4. In order to study this possibility in more detail, measurements were conducted on a hot-pressed surface-ground Si_3N_4 plate that contained seeded voids. The voids were formed in the interior of this plate by first drilling small holes in a 0.125-in thick plate, and then diffusion bonding this plate to a second 0.125-in thick plate. The data shown in Fig. 8 further illustrate the ability of the microwave system to detect small voids in Si_3N_4. It can be seen that not all the voids were detected by microwaves, but these particular voids were also weakly imaged in an X-ray, indicating that they probably contained some kind of material, rather than being empty as was intended.

It is interesting to note the partial diffraction pattern produced by the uppermost holes on the left of the scan area. In principle, such a diffraction patttern could provide information about the geometry of the scatterer. The microwave C scan apparently also shows some inclusions that are not detected in the ultrasonic C scan.

Finally, to show the effects of edge scattering, the entire surface of a 4-in diameter hot-pressed Si_3N_4 disk was scanned with the W-band system. These measurements were made after both surfaces of the disk had been ground smooth. The results are shown in Fig. 9. X-ray inspection of the disk showed the presence of an unintended high-density inclusion about 0.020 in in diameter. This flaw is clearly detected in the microwave C scan, and the location of the flaw (see Fig. 9(b)) coincides very closely with the location indicated by the X-ray. It can be seen from the figure, however, that the flaw would not have been detectable if it had been near an edge, because the cross-polarized scattering from an edge is quite strong and contributes to the detected signal at a significant distance from the edge. As discussed previously, the region of influence of edge scattering depends on the beamwidth of the antennas, and is minimized when the beamwidth is smallest.

IV. Conclusions

The results of this study have established the definite feasibility of using millimeter-wave scattering to detect and locate small internal flaws in Si_3N_4. Specific conclusions drawn from this work can be summarized as:

1) 0.005-in diameter defects of various types (including voids) have been successfully detected using frequencies near 100 GHz. Si_3N_4 was found to exhibit very little dissipation loss at these frequencies.

2) Microwave scattering appears to be more sensitive to certain types of defects (e.g., unreacted silicon and surface cracks) than are ultrasonic or X-ray techniques. Generally speaking, metallic inclusions were found to produce the largest microwave scattering.

3) Using cross-polarized open-ended waveguides as antennas, spatial resolutions of a few millimeters were obtained at 100 GHz. Slightly better resolution should be obtainable using lenses.

4) A frequency dependence has been observed in the microwave scattering from different types of flaws. This frequency dependence may prove useful for flaw characterization. The usefulness of being able to vary the frequency of the illuminating wave in order to optimize detection and characterization suggests that the microwave system should be broadband.

5) The usable upper frequency of operation for the microwave NDE of ceramics appears to be determined by the background clutter associated with surface roughness and grain boundaries or porosity. This clutter begins to be

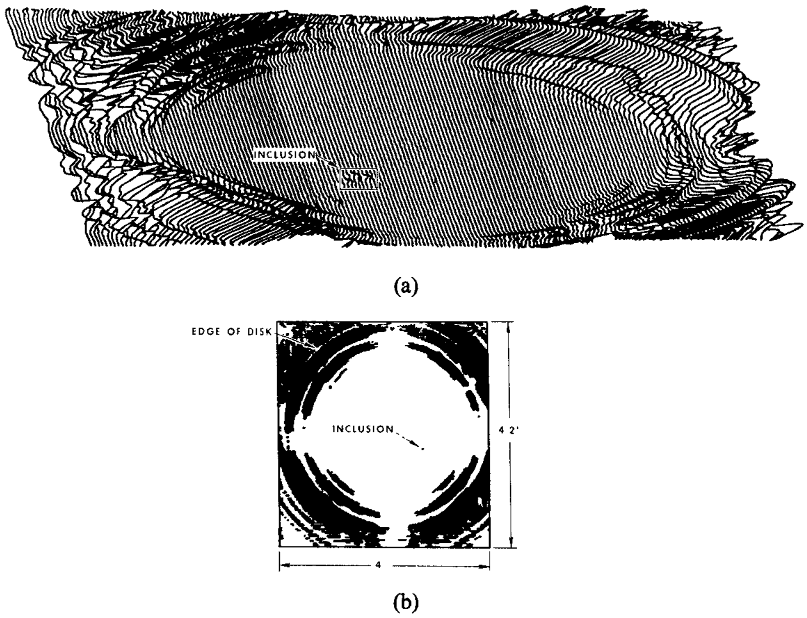


Fig. 9. Microwave cross-polarized transmission C scan of 4-in diameter hot-pressed Si_3N_4 disk. Frequency=94.4 GHz. (a) Amplitude versus position. (b) Excess signal versus position.

noticeable at 100 GHz for grit-blasted surfaces and/or reaction-sintered material.

6) A limitation of the microwave technique stems from the large amount of cross-polarized scattering that is produced by a sharp geometrical corner. This spurious scattering will mask the weaker scattering produced by any defects in the neighborhood of such a corner.

In general, the most promising microwave technique for the NDE of ceramics appears to be one that makes use of cross-polarized scattering from a defect, either in a forward-scatter or a backscatter mode. Direct incoherent detection of the scattered waves appears feasible, but other detection techniques should be studied. In addition, the quantitative capabilities of microwave NDE need further assessment.

The use of microwave techniques for the nondestructive evaluation of Si_3N_4 (and any other ceramic material having similar dielectric properties) appears very promising from a technical standpoint. Of course, acceptance of this technique by the NDE community will depend not only on technical performance, but also on the cost of equipment acquisition. This cost is relatively high at present, but it can be expected to decrease in the future because of the demand that is building in other application areas such as communications, radar, and radiometry.

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