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NONDESTRUCTIVE EVALUATION TECHNIQUES FOR SILICON CARBIDE HEAT-EXCHANGER TUBES

Annual Report
October 1977—September 1978

by

D. S. Kupperman, D. Yuhas, C. Sciammarella,
N. P. Lapinski, and N. Fiore

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ARGONNE NATIONAL LABORATORY, ARGONNE, ILLINOIS

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Materials Science Division

March 1979

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ABSTRACT

This report discusses the adequacy of several nondestructive evaluation techniques for the detection of flaws in silicon carbide heat-exchanger tubing. Experimental results have been obtained for conventional ultrasonic testing, acoustic microscopy, conventional and dye-enhanced radiography, holographic interferometry, infrared scanning and internal-friction measurements. Calculation of critical flaw size and the advantages and disadvantages of overload proof testing are also discussed. Suggestions regarding preservice as well as inservice inspection of silicon carbide tubing are given, along with a comparison of the effectiveness of the various nondestructive evaluation methods evaluated in this study.

I. INTRODUCTION

The application of ceramic components is of particular interest because they are lighter than their metallic counterparts, can operate at high temperatures, have good corrosion resistance, and can be fabricated from inexpensive and abundant elements. As a result, the use of ceramics can lead to more efficient energy-conversion systems. If ceramics were used for heat exchangers, for example, working fluids could attain temperatures of 1200 to 1600°C, whereas if superalloys were employed, temperatures would be limited to 800°C.

In recent years, significant progress has been made in the use of ceramics for structural applications. Silicon carbide (SiC), for example, is currently being used for heat-exchanger tubing because of its excellent thermal-shock resistance, low coefficient of expansion, high thermal conductivity and strength at high temperature.

Several organizations are engaged in the evaluation and development of ceramic heat exchangers. For example, Garrett AiResearch Casting Co. (Torrance, CA) has a program to develop and evaluate SiC recuperators. Their

results indicate that chemical vapor deposit (CVD), sintered and siliconized SiC are suitable for high-temperature heat-exchanger applications. Los Alamos Scientific Laboratory (Los Alamos, NM) has a program to develop heat pipes for use in recuperators for high-temperature industrial-process applications. Solar Turbines International is developing joining techniques for ceramic heat exchangers, including ceramic/ceramic and ceramic/metal joints, and Oak Ridge National Laboratory has a program to evaluate the potential of certain ceramics for heat-exchanger applications.¹

The reliable use of ceramics as structural components, however, requires effective failure prediction and thus effective flaw-detection capabilities. The lifetime of SiC components is affected by cracks, porosity, inclusions and free silicon. The size of critical cracks leading to fracture can be relatively small (an order of magnitude or more smaller than in comparable metallic parts) and related to microstructural features such as grain size and inclusions. Many fracture origins are adjacent to the surface,² indicating that surface cracks are an important cause of failure. Thus, nondestructive evaluation (NDE) techniques that are satisfactory for metals may not be for ceramics. It may be necessary to develop or advance conventional NDE techniques for ceramic applications. Currently, the techniques most widely employed by industry for ceramics are x-radiography and fluorescent dye penetrant testing. However, efforts are under way at several institutions to advance NDE techniques for structural ceramics. These techniques include high-frequency (>50 MHz) ultrasonic testing,³ microfocus x-radiography,³ microwave NDE,⁴ acoustical surface-wave testing,⁵ photoacoustic spectroscopy,⁶ and acoustic-emission detection.⁷

The purpose of the present ceramic NDE program is to compare the effectiveness of conventional and unconventional NDE techniques for specific high-temperature ceramic components. After an investigation of many NDE techniques and failure modes, one or more NDE methods will be developed further for the specific ceramic components of interest. The current effort involves SiC heat exchangers; previous efforts have involved silicon nitride (SiN) gas-turbine rotors.⁸ The present investigation encompasses many NDE techniques, particularly those not under extensive evaluation at other institutions. These include dye-enhanced radiography, acoustic microscopy, conventional ultrasonic testing, acoustic-emission detection, acoustic impact testing, holographic interferometry, infrared scanning, internal-friction measurements and overload proof testing. No single technique is expected to serve as a universal detection method; several techniques will be required to thoroughly assess SiC tubing. The limitations of several common techniques are discussed below.^{3,8}

Detection and characterization of critically sized defects in ceramic components via ultrasonic methods appear feasible, even though numerous obstacles must be overcome. Because of the necessity for detection of small flaws, frequencies much higher than those in current use may have to be

employed (i.e., up to ~400 MHz versus <50 MHz). The practical application of high-frequency ultrasonics still may be limited by transducer design, sample geometry, and difficulties in producing good acoustic coupling to the interrogated sample. Ultrasonic in-service evaluation of heat-exchanger tubing from the bore side would be particularly difficult if high frequencies are used.

The important variables in establishing the sensitivity of x-ray methods for flaw detection are the resolution and image contrast. The resolution of conventional x-ray systems is ~100 μm , but more sophisticated systems using electron-focusing techniques have greater resolving power. The ultimate limitation of x-ray systems, however, is in the image contrast,³ determined by the relative values of the absorption coefficients of the flaw and the ceramic material. In addition, tight cracks are generally difficult to detect, since the x-ray beam must be parallel to the crack plane.

Penetrants yield information on surface defects, mainly cracks, but are probably not capable of resolving bore-side cracks in tubing, or any flaws with critical dimensions less than 300 μm .² Also, information on flaw depth cannot be obtained with dye-penetrant techniques.

Overload proof testing is designed to eliminate weak components that would not survive in service. The limitations here are that slow crack growth may occur during testing, and the flaw population may not be constant while the part is in service. Furthermore, it may not be easy to reproduce large thermal stresses produced by transients in a complex system.

The limitation of acoustic-emission detection arises from the difficulty of interpreting the acoustic-emission signals to identify, localize and characterize flaws.

II. SAMPLES

The 14 SiC tubes used in the present study were obtained from the Garrett AiResearch Manufacturing Division's high-temperature heat-exchanger program. They included the following types: Super KT (Carborundum); sintered α -SiC (Carborundum); NC430 (Norton); and CVD [Materials Technology Corp. (MTC) and Deposits and Composites, Inc. (DCI)]. The tubes are nominally 200 mm long and 25 mm in diameter, and have wall thicknesses ranging from 1.5 to 3 mm. Three of the tubes are shown in Fig. 1.

Three hot-pressed SiC bars (square cross section) were purchased from Norton Co. They are nominally 150 x 6 x 6 mm in size, with the surface ground smooth. A photograph of one of the bars is shown in Fig. 2.

Fig. 1. SiC Tubes Used In the Present Study. Neg. No. MSD-66093.

Fig. 2

SiC Bar Used in the Present Study.
Neg. No. MSD-66094.

III. OVERLOAD PROOF TESTING AND CRITICAL FLAW SIZE

A. Background

Fracture mechanics is a useful discipline for the evaluation of structural reliability. An assumption in fracture mechanics analysis is the existence of a crack of finite size. Therefore, a fundamental step in life prediction is the detection of cracks and the establishment of a minimum detectable crack size. One method for crack detection is overload proof testing, a procedure that can be applied to ceramic heat-exchanger tubing.

The lifetime, t_f , of a structural component is the time required to nucleate a crack by, for example, fatigue, corrosion or a combination of both, and to propagate it to critical size a_c . For a preexisting crack, the nucleation time does not enter into the problem and the only relevant quantity is the propagation time of the crack from the initial crack size a_i to critical size a_c . The purpose of proof testing is to provide an estimate of a_i by the application of fracture mechanics analysis.

If one starts from the basic equation of linear fracture mechanics, and considers a crack under mode-I crack opening, one can write

$$K_I = \sigma_a Y \sqrt{a}, \quad (3.1)$$

where σ_a is the applied tensile stress, Y is a constant that depends on the geometry of the problem under consideration and a is the crack length. By differentiating Eq. 3.1 we obtain

$$\frac{dK_I}{dt} = \frac{\sigma_a^2 Y^2 v}{2K_I}, \quad (3.2)$$

where $v = da/dt$, the velocity of crack growth. By integrating Eq. 3.2 we obtain

$$t_f = \frac{2}{\sigma_a^2 Y^2} \int_{K_{Ii}}^{K_{Ic}} \frac{K_I}{v} dK_I, \quad (3.3)$$

where K_{Ii} = the initial stress-intensity factor at the most serious flaw and K_{Ic} = fracture toughness of the material.

The integration of Eq. 3.3 requires a knowledge of $v = f(K_I)$. A log-log plot of v versus K for environmentally induced slow crack growth in many ceramics shows three characteristic regions. The region where most of the crack growth takes place can be represented by the equation

$$\frac{da}{dt} = AK_I^n, \quad (3.4)$$

where A and n are constants to be experimentally determined. Substituting Eq. 3.4 into Eq. 3.3 and integrating, we obtain

$$t_f = \frac{2}{AY^2(n-2)\sigma_a^2} [K_{Ii}^{2-n} - K_{Ic}^{2-n}]. \quad (3.5)$$

Equation 3.5 can be written in an alternative form if we take into consideration the following relationships:

$$K_{Ii} = \sigma_w Y \sqrt{a_i}, \quad (3.6)$$

where σ_w is the working stress, and

$$K_{Ic} = \sigma_w Y \sqrt{a_c}. \quad (3.7)$$

Substituting into Eq. 3.5, we obtain

$$t_f = \frac{2}{A(n-2)\sigma_w^n Y^n} [a_i^{(2-n)/2} - a_c^{(2-n)/2}], \quad (3.8)$$

which can also be written

$$t_f = \frac{2}{(n-2)AY^n\sigma_w^n} \left[\frac{1}{a_i^{(n-2)/2}} - \frac{1}{a_c^{(n-2)/2}} \right]. \quad (3.9)$$

Equations derived from Eq. 3.5 have been successfully utilized in applications to glasses.⁹⁻¹¹ The same techniques have been applied to other ceramics^{12,13} and to ceramic turbine components⁶ with the assumption that significant damage by cyclic loading is absent. In the work cited above, the damage produced by cyclic loading is not taken into consideration. There is, however, considerable experimental evidence that in metals and some other materials, cyclic-load fatigue crack-growth rates can be correlated with the range of the stress-intensity factor. A log-log representation gives three regions which are similar to the ones observed in plots of the static fatigue of glasses versus the stress-intensity factor. The propagation of cracks takes place in region II and is characterized by an equation of the form

$$\frac{da}{dN} = B\Delta K^m, \quad (3.10)$$

where N is the number of cycles and B and m are constants to be experimentally determined. The integration of Eq. 3.10 gives

$$N = \frac{2}{(m-2)BY^m\Delta\sigma^m} \left[\frac{1}{a_i^{(m-2)/2}} - \frac{1}{a_c^{(m-2)/2}} \right]. \quad (3.11)$$

Equations 3.8 and 3.11 give similar results if $\Delta\sigma = \sigma_w$, that is, if the stress increases from 0 to a maximum value, the case in many structures. Of course, Eq. 3.8 gives the result in time, Eq. 3.11 in number of cycles.

The term $\frac{1}{a_c^{(n-2)/2}}$ can be neglected if $a_i \ll a_c$. For that case,

$$t_f = \frac{2}{(n-2)AY^n\sigma_w^n} \frac{1}{a_i^{(n-2)/2}}. \quad (3.12)$$

We can see that in both cases the life of the component is inversely proportional to a power of the minimum crack size. The life is controlled by the two constants A and n . In general, $n > 2$, and in many cases $n > 10$.

We can now consider proof testing for both static fatigue and cyclic loading. Proof testing puts an upper limit on the flaw size present in the specimen after testing. If we let σ_p be the stress level applied during proof testing, flaws of size leading to the condition $\sigma_c = \sigma_p$ will cause failure. Crack sizes leading to $\sigma_p < \sigma_c$ will not cause failure. We can then write

$$K_{Ic} > K_p = \sigma_p Y \sqrt{a_i}; \quad (3.13)$$

from Eq. 3.13,

$$a_i \leq \frac{K_{Ic}^2}{\sigma_p^2 Y^2}; \quad (3.14)$$

and

$$\frac{1}{a_i^{(n-2)/2}} \geq \frac{\sigma_p^{n-2} Y^{n-2}}{K_{Ic}^{n-2}}. \quad (3.15)$$

Substituting into Eq. 3.12, we obtain

$$t_f \geq \frac{2\sigma_p^{n-2}}{(n-2)AY^2\sigma_w^n K_{Ic}^{n-2}}. \quad (3.16)$$

The above equation gives accurate values for t_f if $a_i \ll a_c$ for $\sigma_c = \sigma_w$, but if a_i becomes close to a_c we have to consider the complete Eq. 3.9.

B. Proof Testing and Critical Flaw Size for Ceramic Heat-exchanger Tubing

From Eq. 3.16 it is evident that accurate life prediction requires an accurate evaluation of the parameters A , n and K_{Ic} , which control the crack propagation. It also requires that the selected model for the fracture process be a good representation of the phenomena taking place. The stresses applied to heat-exchanger tubing depend on the design temperature gradient and on the stresses originated by the temperature gradient. The stresses depend on the selected geometric configuration and on the end conditions of the tubing. For reasons of simplicity let us consider only the bending stresses introduced by the thermal gradient. These stresses can be computed by the equation

$$\sigma = \frac{E\alpha\Delta T}{2(1-\nu)}, \quad (3.17)$$

where, for NC430,¹⁵

E = modulus of elasticity = 49×10^6 psi,

α = coefficient of linear expansion = $2.8 \times 10^{-6}/^\circ\text{F}$,

ΔT = the difference between the temperatures at the inner and outer surfaces of the tube wall,

and

ν = Poisson's ratio = 0.22.

Assuming $\Delta T = 150^\circ\text{F}$ (Ref. 15), $\sigma = 13 \text{ ksi} = 91(\text{MN}/\text{m}^2)$. For a long axial crack of depth a ,

$$a_{\text{critical}} = \left[\frac{K_{I\text{critical}}}{Y\sigma} \right]^2, \quad (3.18)$$

where $K_{I\text{critical}}$ is the critical stress-intensity factor. For SiC, $K_{I\text{critical}} \sim 3.5(\text{MN}/\text{m}^{3/2})$ and $Y = 1.99$ (see discussion below). Thus $a_{\text{critical}} = 370 \mu\text{m}$.

To simplify the initial calculations, the tube is assumed to be in perfect thermal contact with the steam and combustion gases and the crack is assumed to be in a uniform stress field. More sophisticated calculations could be carried out to obtain a more reliable result. Nevertheless, one may conclude that the critical crack size for SiC heat-exchanger tubing is, conservatively, on the order of $100 \mu\text{m}$.

Proof testing requires that the test samples be subjected to stresses that exceed the service stresses by specified amounts. For tubing, a proof-testing procedure utilizing internal pressure would satisfy the above condition. Internal pressure introduces almost uniform tensile stress in the hoop direction throughout the tube thickness.

For Carborundum KT, $K_{IC} = 3.53 \pm 0.4(\text{MN}/\text{m}^2)\sqrt{\text{m}}$, while the modulus of rupture at room temperature is $138(\text{MN}/\text{m}^2)$. For a tube, the most serious flaw is a long surface flaw. The value of Y for a surface flaw can be obtained by assuming the flaw to be an edge crack on an infinite plate. This assumption is valid if the crack depth can be neglected when compared to the thickness of the tube. For an edge crack, $Y = 1.99$; the critical size then can be computed from Eq. 3.1 with $\sigma_w = 138(\text{MN}/\text{m}^2)$, $K_{IC} = 3.53(\text{MN}/\text{m}^{3/2})\sqrt{\text{m}}$, and $Y = 1.99$. We obtain

$$a = 165 \mu\text{m}.$$

That is, $165 \mu\text{m}$ is the crack size that corresponds to the modulus of rupture of the material. This value is comparable to the estimated critical flaw size. Thus, for Carborundum KT, proof testing can only detect cracks longer than $165 \mu\text{m}$.

Although the data on cyclic-loading damage for Carborundum KT consist of only a few points, the following equation can be obtained from it:

$$\frac{da}{dN} = 0.37 \times 10^{-6} \Delta K^{2.19},$$

where da/dN is in m/cycle and ΔK in $\text{MN}/\text{m}^{3/2}$.

Table I shows the testing pressure required to insure different numbers of loading cycles for tubing subjected to a radial gradient of $\Delta T = 150^\circ\text{F}$.

TABLE I. Testing Pressure Required to Insure Various Numbers of Loading Cycles

Loading Cycles	a, μm	σ		P, ksi
		MPa	ksi	
300	200	126	18	6
420	100	178	a	a
650	50	250	a	a
845	25	353	a	a

^aThe necessary σ exceeds the modulus of rupture.

The last column of Table I gives the pressure necessary to detect cracks of the dimensions given in the second column. We can see that to detect a crack of 200 μm , an applied pressure of 6000 psi is required. For Norton NC430, the crack-growth relationship is

$$\frac{da}{dN} = 6.36 \times 10^{-11} \Delta K^{7.47},$$

where da/dN is in m/cycle and ΔK in $\text{MN}/\text{m}^{3/2}$.

Table II shows the relationship between required testing pressure, detectable crack size and life in cycles for NC430.

TABLE II. Testing Pressure vs Detectable Crack Length in NC430 Tubing

Crack length, a μm	σ_{critical}		P, ksi	Life, cycles
	MPa	ksi		
50	250	36.4	12.1	4.8×10^4
100	177	26.7	8.6	7.1×10^3
200	126	18.2	6.1	2.7×10^3
300	102	14.8	4.9	1.5×10^3
400	89	12.8	4.3	8.9×10^2
500	79	11.5	3.8	5.3×10^2
600	72	10.5	3.5	3×10
700	67	9.7	3.2	-

We can see that even for cracks close to 1 mm in length, the necessary pressures are high. Consequently, although internal pressure fulfills one of the basic requirements of proof testing, since it subjects all the elements of the volume to the necessary level of stress, it poses experimental difficulties. Of course, the computed stresses correspond to the hoop direction. In the axial direction, the stresses will be one half of the hoop stresses. Internal pressures of a magnitude that can be easily applied permit detection of only very large cracks. For example, with an internal pressure of $P = 2$ ksi, the minimum detectable crack size is 1.8 mm, whereas cracks on the order of several hundred μm in length are sufficient to cause failure.

It might be possible to utilize a temperature gradient as a means of testing the tubes. However, a thermal load will pose problems. A propagating crack will modify the geometrical constraints that caused the original stresses; this may change the stress distribution and lead to crack arrest. Consequently, it will be difficult to obtain a clear picture of the crack population distribution after testing.

Inadequate reproduction of the stresses (thermal or mechanical) in the tubes and the presence of crack-growth instabilities during unloading also limit the effectiveness of overload proof testing.⁷ Furthermore, of course, overload proof testing cannot detect cracks initiated after the component is put into service.

IV. ACOUSTIC MICROSCOPY

A. Background

Traditional methods of microstructural characterization employ a variety of instruments and techniques, such as optical and electron microscopy and x-microradiography. The common denominator of these methods is that the images are produced by the interaction of electromagnetic radiation with the specimen. Thus, structures are visualized only as a result of variations in electromagnetic properties, such as dielectric constant or conductivity. In contrast, the acoustic microscope is an imaging system based upon acoustic rather than electromagnetic waves. Thus, variations in the elastic properties are primarily responsible for visualization of structures in acoustic micrographs.

Acoustic waves are a form of mechanical energy; therefore, they are reflected, refracted, and scattered at interfaces, structures and inclusions. The propagation and attenuation of acoustic waves are governed by physical properties, such as density, elastic modulus, viscosity, and viscoelasticity of the material under investigation. Thus, the physical properties responsible for the acoustic image are different from those responsible for images obtained using waves from the electromagnetic spectrum. The acoustic-microscopic analysis of the elastic properties may provide a means for the nondestructive detection of defects which may be important in determining the performance of SiC heat-exchanger tubing components.

The instrument used in these investigations is the Sonomicroscope 100 developed by Sonoscan, Inc., a scanning-laser acoustic microscope (SLAM)

operated at an ultrasonic frequency of approximately 100 MHz (see Fig. 3). To facilitate interpretation of the acoustic micrographs presented in the report, a brief description of the important features of the microscope is given below.

Fig. 3. Acoustic Microscope Used in the Present Study. Neg. No. MSD-66095.

The irradiation geometry used to produce the acoustic micrographs of tube sections studied in this work is illustrated in Fig. 4. Ultrasonic energy is incident on the sample from below, at an angle of 10° . The transmitted acoustic energy imparts a slight oscillatory mechanical motion to the sample's top surface. These oscillations have the same frequency

as the incident wave, but vary in amplitude depending on the acoustic attenuation and absorption properties of the underlying material. These disturbances

Fig. 4. Irradiation Geometry for Production of Acoustic Micrographs.
Neg. No. MSD-66096.

are detected, point by point, by a rapidly scanning focussed laser beam (40,000 image points per micrograph) which drives an opto-acoustic receiver. The acoustic image is then displayed on the TV monitor; the white regions correspond to areas of the sample with good acoustic-transmission properties, while the darker areas of the micrographs are regions of higher ultrasonic attenuation. If the sample has a good surface finish, an acoustic image can be obtained from the light specularly reflected directly from the surface. When this is not the case, a mirrored coverlip is placed in acoustic contact with the top surface of the sample and light reflected from the coverlip is used to form the acoustic image.

The acoustic energy is coupled to the sample through the acoustic-microscope stage. Two basic types of stages are commonly used. In the glass cell, illustrated in Fig. 5a, the transducer is bonded to a glass block which serves to transport the acoustic energy from the transducer to the sample. The liquid cell is illustrated in Fig. 5b; here, the sound is conducted from transducer to sample by a liquid which fills the chamber. In the present study, the liquid cell was used almost exclusively and the angle of insonification was 10° from the normal. The special fixturing and transducer required to obtain results on intact tubes are described in Section D.

Fig. 5. Types of Stages Used for Generation of Acoustic Micrographs. Neg. No. MSD-66097.

B. Acoustic-microscope Data

Data are presented in the form of photomicrographs and super-8 movies. The four image modes available with the SLAM are described below. Switching between any of the visualization modes is accomplished electronically; thus, no repositioning of the sample is required.

1. Single-frequency Acoustic-amplitude Micrographs

Micrographs of this type are made at a single ultrasonic frequency. They are generally characterized by a substantial amount of contrast and are often subject to coherent speckle. The amount of speckle is related to the degree of scattering in the specimen and provides some indication of both the mode of acoustic loss and the elastic microstructure of the material under investigation.

2. Frequency-modulation Acoustic-amplitude Micrographs

These micrographs are obtained by sweeping the frequency over a variable bandwidth around 100 MHz. This eliminates coherent speckle and thus reveals features that would otherwise be masked.

3. Acoustic Interferograms

In addition to displaying the acoustic amplitude distribution throughout the field of view, the SLAM provides an acoustic interference mode of operation. Acoustic interferograms show a series of alternating light and dark stripes. For acoustically homogeneous samples, these bands (interference fringes) are parallel to one another and are equally spaced. For samples that are elastically inhomogeneous, the interference lines will be distorted by localized variations in the sound velocity and/or sample thickness. All of the interferograms in the present report are oriented so that fringe shifts to the left correspond to areas of lower ultrasonic velocity and shifts to the right indicate regions of higher ultrasonic velocity. For samples with controlled thicknesses, the character of the interferogram fringes is determined solely by localized variations in the velocity of sound. These variations are related to variations in either the bulk density or elastic modulus of the sample constituents.

4. Optical Micrographs

For samples which are not optically opaque, good optical transmission images can be obtained. In some cases the use of coherent laser illumination and special optical detection schemes lead to enhanced image contrast.

C. Acoustic Characteristics of Fractures in SiC Test Bars

1. Background

The acoustic characteristics of fractures detected by the SLAM depends on such parameters as the crack orientation relative to the direction of sonic propagation, the size of the crack opening, the texture of the fracture interface, and the extension of the fracture beneath the surface. In the sample described below, the acoustic transmission level across the fracture interfaces varied from almost total attenuation to only slight variations in acoustic contrast. Similarly, the perturbation of the interferogram fringe spacing by the fractures ranged from complete disruption through refraction of the sound beam to almost a nonperceptible fringe shift. In this section, micrographs illustrating the acoustic characteristics of a variety of fractures are presented.

2. Sample Description

All data were obtained on two 6 x 6 x 15-mm hot-pressed SiC test bars. Sample A-2 had been cracked by thermal shock; Sample A-1 was crack-free. The samples were identified by strips of tape at one end. Two faces of Sample A-2 were marked 90 and 0°, respectively. Acoustic micrographs were obtained by insonifying the sample at an incidence of ~10° to the normal in water. Within the bar, the sound propagates at approximately 60° to the normal, owing to its greater velocity (see Fig. 6).

Fig. 6

Angle of Refraction for Shear Wave in SiC Bars. ANL Neg. No. 306-78-827.

3. Documentation

All micrographs were labeled to give the following information:

- a. Slide number. Identification code for slide from which the print was made.
- b. Mode. I = interferogram; A = single-frequency acoustic-amplitude micrograph; Am = frequency-modulation acoustic-amplitude micrograph; O = optical micrograph.

Fig. 7. Orientation of SiC Test Bar on Acoustic-microscope Stage. Neg. No. MSD-66098.

For example, a sample orientation of 0° means that the sound field is detected at the 0° face, which is in direct contact with the coverslip. The micrographs shown an ~ 3 - by 2-mm area of the sample. The interference lines are $\sim 100\text{ }\mu\text{m}$ apart.

d. Micrograph location. This identifies the location of the micrograph along the bar. The numbers represent the distance, in mm, of the center of the micrograph from the reference edge of the bar (marked " 90° " and " 0° " in Fig. 7). For example, micrographs obtained at the right edge of the bar shown in Fig. 7 would have a location designation of 1.5 mm.

4. Acoustic Microstructure in Fracture-free Region

Typical acoustic microstructure of a fracture-free region of test bar A-2 is shown in Fig. 8. The fracture-free region exhibits homogeneous sound transmission and low-contrast acoustic microstructure. The horizontal streaking in micrograph AR-728-18 (Am) is attributed to the surface texture of the sample bar. The interferogram lines in AR-728-19 are straight, indicating homogeneous sonic velocity. The slight perturbations of the interferogram lines, like the horizontal streaking, are related to the surface texture. This type of structure is commonly observed in Sample A-1. However, in Sample A-2, it represents the exception; most regions of this sample show clear evidence of fractures.

5. Morphology of Surface-opening Cracks

a. Variation of amplitude and phase across the fracture boundary. The micrographs AR-8076-10 (I) and -11 (Am) (180° , 83 mm) in Fig. 9 show a fracture opening to the 180° surface of Sample A-2. The structure observed in the micrograph can be understood in the context of Fig. 10. Sound propagating through a sample will be attenuated at the fracture interface, resulting in a shadowed region to the right of the fracture. The micrographs are oriented such that the horizontal component of the sound field propagates from left to

c. Orientation. Samples were mounted on the microscope as indicated in Fig. 7. The x-y plane defines the plane of the stage. In Fig. 7, the sound is incident on the 180° face, which is designated as the bottom, and is detected at the 0° face, which is designated as the top. Because micrographs were obtained in a variety of orientations, we have adopted the convention of identifying only the top surface.

right across the field of view. The characteristics of the shadowed region are related to properties of the fracture interface such as interface texture, crack-opening width, and crack depth. The primary feature that distinguishes a surface-opening crack (SOC) is the abrupt and sharply defined onset of the shadowed region. A sharp boundary is seen separating the light and dark portions (to the left and right, respectively) of micrographs AR-8076-10 and -11 (Fig. 9). The attenuation of sound across the fracture surface in this micrograph pair is quite severe. This fracture is one of the most highly attenuating cracks observed in Sample A-2. However, some fractures showing no sound propagation across the interface were observed. The left-hand portions of micrographs AR-8076-10 and -11 show better acoustic transmission than the shadowed regions. However, compared with that in regions containing no fractures (e.g., AR-728-18 and -19, Fig. 8), the transmission is perturbed, indicating sonic propagation across a subsurface fracture interface.

AR-728-18

AR-728-19

Fig. 8. Acoustic Micrographs AR-728-18 (Am) and -19 (I) of a Fracture-free Region of Test Bar A-2. Neg. No. MSD-65748.

AR-8076-10

AR-8076-11

Fig. 9. Acoustic Micrographs AR-8076-10 (I) and -11 (Am) of a Fracture Opening to the 180° Surface of Sample A-2. Neg. No. MSD-66099.

Fig. 10

Sound Shadow Produced by Crack in
SiC Bar. ANL Neg. No. 306-78-825.

In Fig. 11, micrographs AR-8076-8 (A) and -7 (I) (270° , 105 mm) show the sharply defined interface of an SOC. Transmission through this crack interface is somewhat better than that seen in AR-8076-10/11, indicating a narrower crack opening. Partial coherency of the interferogram lines is maintained. The circled feature in the portion of the micrograph to the left of the shadowed region probably corresponds to a fracture oriented horizontally to the SOC.

AR-8076-8

Fig. 11

Acoustic Micrographs AR-8076-8 (A)
and -7 (I) Showing the Interface of a
Surface-opening Crack in Sample A-2.
Neg. No. MSD-66100.

AR-8076-7

In Fig. 12, micrographs AR-806-6 (Am) and -5 (I) (0° , 48 mm) show a sharply defined SOC. Although the level of transmission through the fracture interface appears to be comparable to that of AR-8076-8/7, the interferogram lines are less perturbed. This can be attributed to a more uniform opening of the crack gap or perhaps filling of the crack with water.

b. Effect of crack orientation. Figure 13 shows micrographs AR-807-27 and -28 (0° , 90 mm) of nearly horizontal fractures in Sample A-2. Here, the amount of shadowing depends on the orientation of the crack relative to the sound field. Cracks oriented vertically in the micrographs will cast the widest shadow, while horizontally oriented cracks will lead to interferogram disruption but will cast no appreciable shadow.

AR-806-5

AR-807-27

AR-806-6

AR-807-28

Fig. 12. Acoustic Micrographs AR-806-6 (Am) and -5 (I) Showing a Sharply Defined Surface-opening Crack in Sample A-2. Neg. No. MSD-66101.

Fig. 13. Acoustic Micrographs AR-807-27 and -28 of Nearly Horizontal Fractures in Sample A-2. Neg. No. MSD-66102.

c. Scattered waves at fracture surface. The scattering of sound at the SOC can lead to interference fringes as the scattered sound mixes with the incident acoustic wave. Evidence for this phenomenon appears in several micrographs already presented. Clear examples are visible in micrographs AR-728-9 (A) (0° face, top) in Fig. 14 and AR-806-14 (I) and -15 (Am) (90° , 2 mm) in Fig. 15.

d. Measurement of crack extension. Figure 16 shows micrographs AR-728-26 (I) and -27 (Am) (0° , location not documented) of an SOC in Sample A-2. By measuring the extent of the shadow, an estimate of crack length can be made. The extent of the shadow, marked by the arrow, measures 1.6 mm. Using the propagation angle of approximately 60° , the crack length is estimated as $1.6 \text{ mm} / \tan 60^\circ = 1 \text{ mm}$. Many of the fractures encountered in Sample A-2 produce shadows of spatial extent $>3 \text{ mm}$ (the dimension of the field of view) and thus must be analyzed with multiple continuous micrographs. Furthermore, the occurrence of multiple fractures often disturbs the sonic transmission sufficiently that the end of the shadow cannot be unambiguously defined. This often precludes crack-depth analysis on a sample with a crack density as high as this one. However, for samples containing only a few fractures, depth analysis is greatly simplified and can be easily carried out.

AR-728-9

Fig. 14. Acoustic Micrograph AR-728-9 (A) Showing Interference Fringes Produced by an SOC in Sample A-2. Neg. No. MSD-66103.

AR-806-14

AR-728-26

AR-806-15

AR-728-27

Fig. 15. Acoustic Micrographs AR-806-14 (I) and -15 (Am) Showing Interference Fringes Produced by an SOC in Sample A-2. Neg. No. MSD-66104.

Fig. 16. Acoustic Micrographs AR-728-26 (I) and -27 (Am) of an SOC in Sample A-2, with Extent of Shadow Indicated by Arrow. Neg. No. MSD-66105.

e. Structure caused by buried fractures. In the previous micrographs the primary emphasis was on the analysis of SOC's. These cracks are easily identified by the sharp definition of the shadowed region. However, in many of the micrographs the "background" structure is not as clean as anticipated, based on the analysis of the unfractured Sample A-1. Instead, the micrographs often contain regions showing high-contrast structure and interferogram deviations even though no sharply defined crack boundary can be seen. This structure can tentatively be attributed to the presence of buried fractures that do not intersect the top surface. A few micrographs are presented here to substantiate this hypothesis.

Fig. 17. Acoustic Micrographs AR-807c-23 (I) and -26 (I) of an SOC in Sample A-2. Neg. No. MSD-65751.

Figure 17 shows micrographs AR-807c-23 (I) (0° , 56 mm) and -26 (I) (180° face, 63 mm) of a region of Sample A-2 containing an SOC. Micrograph AR-807c-26 was obtained with the 180° face of the sample (the surface to which the fracture opens) near the coverslip. Micrograph AR-807c-23 was obtained with the coverslip in contact with the 0° face, in a region containing the projected image of the SOC. In this case, there is no clear definition of the crack edge. However, the existence of the crack clearly leads to disruption of the interference fringes.

f. Buried fracture oriented parallel to top surface. The micrographs shown in Figs. 18 and 19 were obtained at the edge of Sample A-2. Micrographs AR-807-7 and -8 were obtained with the 0° face at the top, while -33 and -34 were obtained with the 270° face nearest the coverslip. The 0° -

270° edge of the sample is visible in both micrographs, appearing at the bottom of AR-807-7 and -8 and at the top of -33 and -34 (see diagram in Fig. 18). Micrographs AR-807-7 and -8 show excellent acoustic transmission in the region to the left of the shadowed portion, except in the area indicated by the arrow. This dark region appears to be a fracture oriented parallel to the 90° face. In this orientation (0° face up), the sound is propagating parallel to the fracture. However, with the 270° face up, the sound must propagate across the fracture zone. This is shown in micrographs AR-807-33 and -34. The SOC, apparent in AR-807-7 and -8, is continuous around the edge. However, the point of interest here is the more highly attenuating zone to the left of the shadow. The structure and increased attenuation can be attributed to the fracture (indicated by the arrow in Fig. 8) oriented parallel to the 270° face.

The locations of six surface breaking cracks studied with the acoustic microscope are indicated in Fig. 20 along with dye penetrant (PT) indications on bar A2. The location of four of the six cracks coincide with the PT indications. One location is slightly off and the other is a crack in a region not carefully examined with the dye.

Fig. 18. Acoustic Micrographs AR-807-8 and -33 of an SOC in Sample A-2. Neg. No. MSD-36106.

Fig. 19. Acoustic Micrographs AR-807-7 and -34 of an SOC in Sample A-2. Neg. No. MSD-66107.

Fig. 20

The Locations of Six Cracks Detected with the Acoustic Microscope Are Compared with Dye Penetrant Indications. Asterisks indicate the cracks detected acoustically. The dotted lines show the shape of the cracks suggested by the acoustic micrographs; the other lines from penetrant testing. Neg. No. MSD-66073.

6. Acoustic Characteristics of Fractures: Summary of Results

- (a) Regions containing no fractures displayed excellent acoustic transmission.
- (b) The presence of fractures is detected by observing variations in sound propagation across the fracture interface (i.e., the shadow).
- (c) A variety of fractures were detected, with attenuation across the fracture interface ranging from total loss of signal to only a slight change in acoustic contrast. The amount of attenuation can presumably be correlated with the crack-opening width.
- (d) Sound propagation across crack interfaces is often but not always accompanied by perturbation of the interferogram lines. This may be related to the texture and orientation of the fracture interface. Refraction and scattering of sound by fractures are also observed.
- (e) SOC's are characterized by sharp onset of the shadowed region. Only the most highly attenuating fractures are observed optically at the surface.
- (f) Buried fractures lead to decreased total acoustic transmission and perturbed interferogram lines. The point of origin of the buried fracture is not always well defined.
- (g) The attenuation of sound propagating across fractures is generally not spatially uniform. The shadowed region often displays a textured acoustic-amplitude structure with high-contrast detail. This is important from the standpoint of interpreting data obtained from heat-exchanger tubes.

D. Analysis of Heat-exchanger Material

This study is divided into two parts: (1) microstructural characterization and (2) acoustic imaging of intact components. In part (1), slices of tubes were obtained in order to characterize the elastic microstructure of the tube material. The investigation included samples of carborundum sintered, NC430 and CVD SiC. The wall thickness of the sintered and NC430 tubes was approximately 3 mm. Two cross-sectional slices (1 mm and 3 mm thick, respectively) were obtained from each of these tubes. Characterization of the CVD material was performed with partially broken 1-mm-thick tube segments. Results obtained on these samples, which have a simple geometry, will be useful in interpreting structures found in the intact components. Photographs of specific areas which exhibit abnormal acoustic microstructures will be compared with results obtained by destructive analysis.

In part (2), the necessary fixtures were developed and fabricated to obtain images from the intact components.

1. Microstructural Characterization

a. Sintered tube. Micrographs AS2-9 (Am) and -11 (I) of a region of the 1-mm-thick section are shown in Fig. 21. The structure observed in this field of view is typical of that encountered in this sample. Note that many of the acoustic-structure and sonic-velocity variations are oriented horizontally in this field of view. This indicates that the density variations and laminar flaws are, in general, oriented circumferentially around the tube. Thus, we can anticipate that the propagation properties will be anisotropic. This is important because here the sound is propagating along the regions of discontinuity, while sound will propagate across the discontinuities when the intact tubes are tested. Micrographs AS3-25 (I) and -29 (O) in Fig. 22 show a large flaw in the same 1-mm-thick section. Figure 23 shows Am, I, and O micrographs of the same tube section. The ability to reproduce the slope of a surface flaw acoustically is clearly shown in the micrographs of Fig. 23. Figure 24 shows Am, I, and O micrographs of the tube section. This field shows one of the more uniform regions of the tube.

b. NC430 tube. Figure 25 shows Am and I micrographs of the 1-mm-thick section of NC430 material. The acoustic attenuation of this material is slightly higher than that encountered in the sintered tubing. Optical inspection of the NC430 sample revealed no laminar flaws similar to those encountered in the sintered material. The acoustic structure observed in both the Am and I micrographs of NC430 tubing (Fig. 25) is typical of that encountered in the latter material and is more complex than that of the sintered tubing. The variation in the acoustic properties between these samples may be related to variations in the amount of unreacted material.

AS2-9

AS2-11

Fig. 21. Acoustic Micrographs AS2-9 (Am) and -11 (I) of a Section of Sintered α -SiC Heat-exchanger Tubing. Neg. No. MSD-66109.

AS3-25

AS3-29

Fig. 22. Acoustic Micrographs AS3-25 (I) and -29 (O) Showing a Large Flaw in a Section of Sintered α -SiC Heat-exchanger Tubing. Neg. No. MSD-66110.

Fig. 23. Acoustic Micrographs AS3-10 (Am), -9 (I), and -13 (O) of a Section of Sintered α -SiC Heat-exchanger Tubing. Neg. No. MSD-66111.

Fig. 24. Acoustic Micrographs AS3-17 (Am), -16 (I), and -18 (O) of a Section of Sintered α -SiC Heat-exchanger Tubing. Neg. No. MSD-66112.

AS4-8

AS4-6

Fig. 25. Acoustic Micrographs AS4-6 (Am) and -8 (I) of a 1-mm-thick Section of NC430 Heat-exchanger Tubing. Neg. No. MSD-66113.

Figure 26 shows the Am, I, and O micrographs obtained on a region of the 1-mm-thick NC430 tube section. The inner-diameter edge of the sample appears at the top of the micrographs. Note that the acoustic transmission is more uniform at the inner edge and gets progressively poorer toward the outer diameter (i.e., toward the bottom of the micrographs). This is a general feature of this tube section, although it is more obvious in this field of view than in others.

Figure 27 shows an I micrograph of the same NC430 tube section, taken in a region of poor acoustic transmission. The most notable change is the decrease in the level of acoustic transmission, which is not well represented in the micrograph. The interferogram structure is only slightly more complex than that encountered typically.

c. CVD tube. An approximately one-inch-long broken portion of a CVD heat-exchanger tube was examined. The wall thickness is approximately 1 mm and the outside surface of the tube is textured with "blister-like" thickness variations on the order of several hundred μm . Acoustic microscopy was carried

out on the tube itself. No sectioning or special adaptation to the SLAM was required. The sample was mounted on the microscope stage as shown in Fig. 28. Although the acoustic transmission of these tubes is complex, anomalous regions, with acoustic transmission properties different from that normally encountered, can still be identified. Figure 29 shows typical micrographs of a CVD tube section. In Fig. 29b, poor transmission is evident in the right portion of the field of view. This can be attributed to the presence of a tight crack. The presence of the crack, which is not visible optically in this unprepared tube sample, was confirmed by microscopy.

AS4-23

AS4-19

AS4-28

Fig. 26. Acoustic Micrographs AS4-23 (Am), -19 (I), and -28 (O) of a 1-mm-thick Section of NC430 Heat-exchanger Tubing. Neg. No. MSD-66114.

AS4-35

Fig. 27. Acoustic Micrograph AS4-35 (I) of a 1-mm-thick Section of NC430 Heat-exchanger Tubing. Neg. No. MSD-66115.

Fig. 28

Configuration for Examining CVD SiC
Tube Section. Neg. No. MSD-66116.

AS4 33

AS4-26

AS4-34

AS4-25

(a)

(b)

Fig. 29. Acoustic Micrographs of a CVD Tube Section: (a) AS4-33 and -34; (b) AS4-25 and -26. Neg. Nos. MSD-66117 and -66108.

d. Summary of Results

Sintered Tube

(1) Penetration of the tube wall thickness (3 mm) is easily accomplished at 100 MHz.

(2) Optical inspection of the cut surface of tube sections revealed a number of laminar flaws.

(3) Acoustic images obtained from tube sections reveal a number of surface and buried flaws; e.g., laminar voids and density fluctuations.

(4) Most flaws were oriented circumferentially, indicating that the tubes are anisotropic.

(5) The microstructure and defect population within a given specimen are consistent across the tube wall thickness.

NC430 Tube

(1) Interrogation of 3-mm-thick sections of the tube at 100 MHz indicates that the wall thickness can be easily penetrated.

(2) Although optical inspection of the surface suggests a more uniform structure than that of the sintered tube, the acoustic structure of the NC430 material is, in fact, more complex and slightly more attenuating.

(3) The regions near the inner- and outer-diameter surfaces of NC430 tube sections tend to be more uniform in structure than the center of the tube wall. This may indicate a variation in the amount of unreacted material with depth.

(4) Although some degree of circumferential orientation of the acoustic structure is apparent, NC430 is more isotropic than sintered material. Also, CVD tube material can also be interrogated effectively.

2. Acoustic Microscopy of Intact Tubes

a. Introduction. Examination of intact tubes with the SLAM did not require the development of new electronic or optical hardware. However, to make the cylindrical tube geometry compatible with the SLAM, special fixtures for handling the tubes and insonification sources were needed; these were developed as part of the present study. Additionally, a reproducible scanning procedure had to be formulated. The primary goal of this prototype development was to obtain good-quality acoustic data on the components under the constraints imposed by the tube geometry. The rationale of this approach was:

(1) Establish the sensitivity and efficiency of acoustic microscopy for detecting critical flaws in fully processed material under the constraints imposed by the tube geometry.

(2) Realistically evaluate the difficulties that might be encountered in the development of a system dedicated to testing the tubes on a production-line basis.

(3) Experiment with a variety of insonification geometries to establish the optimum configuration and scanning procedure.

(4) Develop the data base required to establish adequate "accept and reject" criteria for the tubes.

b. Hardware development. This involved the following steps:

(1) Fabrication of a small transducer operating at a frequency of 100 MHz (see Fig. 30).

(2) Fabrication of a water bath sufficiently large to accommodate 6-inch tube sections.

(3) Modification of the SLAM to accommodate the water bath, transducer, and tube (see Fig. 31).

(4) Transmission imaging in tubes.

The diagram in Fig. 32 shows the insonification geometry used to obtain transmission acoustic micrographs of the sintered tubes. The tube may be scanned either circumferentially (by rotating it) or lengthwise.

Fig. 30. Transducer for Tube Inspection. Neg. No. MSD-65752.

Fig. 31. Modification of SLAM to Accommodate Water Bath, Transducer and Tube. Neg. No. MSD-66118.

Fig. 32

Insonification Geometry
for Tube Inspection. ANL
Neg. No. 306-78-828.

c. Documentation. Tube-section regions from which the micrographs were obtained were marked in ink. Figure 33 relates the marked regions to the micrograph identification codes. The words "CARBORUNDUM C514-47-14 SiC8," printed circumferentially around the outside portion of the tube section analyzed in this study, served as a reference for the ink marks.

Fig. 33

Labeling of Tube Section Inspected by Acoustic Microscopy.
Neg. No. MSD-66119.

d. Presentation of data. A six-inch-long section of sintered SiC tubing was used. The wall thickness is ~3 mm. Approximately 80% of the area around the circumference of the tube revealed regions of high-contrast amplitude structure and perturbed interferogram fringes. This structure is similar to that observed in the shadowed regions of cracked SiC test bar A-2 (see Section IV.C). This suggests the presence of numerous cracks or laminar flaws oriented circumferentially around the tube, a result that can be anticipated from the SLAM investigations of the tube sections. Two distinct regions around the tube exhibited transmission comparable to the fracture-free region of the SiC test bar. Although acoustic transmission at 100 MHz is sufficient to permit characterization of the tubes, the technique may be too

sensitive in that flaws which may not be detrimental to operation of the exchanger are easily visualized. A lowering of the acoustic frequency would reduce the sensitivity of the method.

Figure 34 shows A and I micrographs of the microstructure typically encountered in the tube. The field of view is asymmetric, with di-

SR2-29

SR2-28

Fig. 34. Acoustic Micrographs SR2-28 (A) and -29 (I) of a Region of Sintered α -SiC Tubing. Neg. No. MSD-66120.

mensions 1.0 by 5.5 mm. Within a 360° circumferential scan, approximately 80% of the area shows this type of elastic microstructure. The variation in the A micrograph and the perturbed nature of the I fringes suggest that the normal material contains a number of inclusions and density variations which give rise to considerable scattering.

Figure 35 shows A and I micrographs of the sintered tube taken in a region directly adjacent to that depicted in Fig. 34. This was one of two regions around the circumference of the tube that was found to exhibit rather uniform acoustic transmission. The dark zone on the left edge of micrograph SR2-27 is the edge of the sound field. The curvature of the interferogram lines in SR2-26 is attributed to the shape of the tube.

Figure 36 shows a boundary between an area of typical microstructure (right half) and a region showing good acoustic transmission. Figure 37 shows the region of the tube giving the most uniform acoustic transmission. The field of view measures 1 by 5.5 mm.

Figure 38 shows two micrographs obtained from different regions of the sintered tube. Micrograph AS1-12 (I) shows the type of acoustic structure encountered most often. Micrograph AS1-20 (I) shows a region of poor acoustic transmission. The complete disappearance of the interferogram lines indicates total attenuation of the acoustic energy. This suggests the presence of a laminar flaw in this zone. Figure 39 shows a photograph of a tube slice from the region where these micrographs were made. The extent of delaminations and their location in this and other photographs of sectioned tubes are consistent with the acoustic-microscopy results.

SR2-27

SR2-35

SR2-26

SR2-34

Fig. 35. Acoustic Micrographs SR2-27 (A) and -26 (I) of a Region Adjacent to That Shown in Fig. 34. Neg. No. MSD-66121.

Fig. 36. Acoustic Micrographs SR2-35 (A) and -34 (I) of a Region of Sintered α -SiC Tubing. Neg. No. MSD-66122.

SR2-5

AS1-12

SR2-6

AS1-20

Fig. 37. Acoustic Micrographs SR2-5 (A) and -6 (I) of a Region of Sintered α -SiC Tubing Showing Comparatively Uniform Acoustic Transmission. Neg. No. MSD-66123.

Fig. 38. Acoustic Micrograph AS1-12 and -20. Neg. No. MSD-66124.

Fig. 39

Photograph of Slice through
Region of Sintered Tube Pic-
tured in Figs. 34-39. Neg.
No. MSD-66125.

In summary, it appears that the acoustic microscope can be adapted to SiC tube inspection and is capable of detecting cracks and subsurface anomalies as well as variations in acoustic properties related to microstructure. Flaws on the order of $100\text{ }\mu\text{m}$ in size can be detected and characterized by acoustic microscopy techniques.

V. DYE-ENHANCED RADIOGRAPHY

The objective of the dye-enhanced radiographic method is to fill surface defects with a substance (doping agent) that will absorb penetrating radiation more effectively than the base material. Thus, although normal radiographs may not reveal surface defects such as cracks and pitting, these flaws may become detectable when dye is present. Dye enhancement has been successfully used with neutron radiography. In this case, gadolinium nitrate is mixed with acetone and a wetting agent to form the penetrant.¹⁶ The dye can reveal cracks even if the neutron beam is not parallel to the crack plane. Thus, a surface crack may be mapped from its shadow. Since gadolinium has a mass absorption coefficient for neutrons three orders of magnitude greater than iron, for example, the technique is particularly useful. Neutron radiography techniques can be used for ceramics; however, the main effort in the present study has been to find a doping agent with sufficient contrast so that conventional x rays could be used effectively. This seems feasible because of the low atomic number and low density of SiC.

The absorption of a parallel narrow beam of monochromatic x rays in a plane-parallel layer of homogeneous isotropic material can be described by the relationship

$$I = I_0 \exp(-\mu t), \quad (5.1)$$

where I is the emerging intensity, I_0 is the incident intensity, μ is the total linear absorption coefficient, and t is the material thickness in cm. The mass absorption coefficient (μ/ρ) is generally used in Eq. 5.1 because the intensity reduction is determined by the quantity of matter traversed. The quantity μ/ρ is essentially independent of the physical state of the material and is additive with respect to the elements that make up the material;

$$\mu/\rho = \sum_i g_i (\mu/\rho)_i, \quad (5.2)$$

where g_i is the mass fraction contributed by the element i with mass absorption $(\mu/\rho)_i$. Thus,

$$I = I_0 \exp\left(-\frac{\mu}{\rho} \cdot \rho t\right). \quad (5.3)$$

The mass absorption coefficient is dependent upon wavelength. For silver nitrate (AgNO_3) at $0.3 \text{ \AA}$,

$$\mu/\rho(0.3 \text{ \AA}) = 10.5 \text{ cm}^2/\text{g}.$$

For SiC,

$$\mu/\rho(0.3 \text{ \AA}) \approx 0.5 \text{ cm}^2/\text{g}.$$

The ratio of mass absorption coefficients is approximately the same even for shorter wavelengths. Thus, AgNO_3 absorbs x rays 20 times more strongly than SiC, and surface flaws not visible by ordinary x-ray methods may be observed by a dye-enhanced technique.

In previous radiography work with SiN ,⁸ many commercially available enhancing agents were tried. A dye consisting of lead oxide mixed with alcohol was also investigated as an agent.

The most successful doping agent, however, was an AgNO_3 solution formed from equal parts, by weight, of AgNO_3 and water plus a small amount of Photoflow to serve as a wetting agent. The procedure for generating the dye-enhanced radiographs is as follows:

- (a) Clean the component in an ultrasonic bath.
- (b) Place in hot (80-90°C) AgNO_3 solution.
- (c) Remove and x ray, using conventional techniques.
- (d) Clean the component in an ultrasonic bath.

An 80-kV x-ray machine with a film-to-object distance of 100 cm and Type SR Kodak film was used.

Some of the current work involves the selection of the x-ray energy that will maximize the observed contrast between the AgNO_3 dye (filling surface cracks) and the SiN or SiC base material. In Fig. 40, the ratio of mass absorption coefficients for AgNO_3 and SiN (data from Ref. 17) is plotted

Fig. 40
Ratio of Mass Absorption Coefficients
of Silver Nitrate and Silicon Nitride.
ANL Neg. No. 306-78-612.

as a function of x-ray energy and wavelength. The absorption edge for silver results in a peak in the ratio of mass absorption coefficients at an x-ray energy of about 25 keV. If most of the x-ray energy were concentrated at 25 keV, the contrast might be enhanced. Another factor is the penetration of the x rays at different energies. The lower energies result in longer exposure times and some loss of contrast for thicker parts of the component. Conventional x-ray machines produce a broad energy spectrum up to a set maximum. To determine the effects of energy variation, a cracked SiN sample was radiographed at four different x-ray voltages (25, 50, 80, and 140 keV) and the ability to detect the dye-filled cracks was qualitatively determined. The poorest result was at the 25-keV setting, as expected, since this is just at the absorption edge. Better results were obtained for 50 and 80 keV; exposures at 50 keV revealed, subjectively, a slight increase in clarity not present at 80 keV. With a 140-keV beam, the contrast was slightly inferior to that at 80 keV. As can be seen, some care is required in selecting the optimum x-ray energy for dye-enhanced radiography, since it will depend

critically on the character of the x-ray absorption of the specimen and the dye elements. The results obtained here are applicable to SiC as well as SiN components.

As an example to demonstrate the enhancement technique, a layer of AgNO_3 , nominally 0.05 mm thick, was placed on a 3.8-mm-thick flat plate of hot-pressed SiN. Although the AgNO_3 represents an increase in thickness of only 1.3%, the film density increased by almost 10%.

The advantages of the dye-enhanced radiography technique have been demonstrated using a piece of SiC tubing (MTC-CVD) with several inner-surface cracks (Fig. 41). In the sample of interest, only one crack could be observed with conventional x-radiography techniques. After treatment with AgNO_3 dye, five cracks were clearly revealed by dye-enhanced radiography. After sectioning, only these

Fig. 41. CVD SiC Tube Section Used for Dye-enhanced Radiography. Neg. No. MSD-66127.

five cracks could be observed microscopically (at ~100X magnification). These tight ($<20\text{ }\mu\text{m}$ wide) ID cracks penetrated approximately halfway through the wall (~25 mils deep), and several were slanted with respect to the tube axis. The tightness and the skew prevented detection by conventional radiography. Figure 42 shows a cross section of the piece of tube after sectioning, and Fig. 43 is a higher magnification of Fig. 42. The smaller crack in Fig. 43 was detected only via the dye-enhanced radiography technique. A scanning-electron micrograph (SEM) of the entire crack is shown in Fig. 44. X-ray fluorescent analysis showed silver close to the crack tip, indicating that some dye had penetrated almost to the maximum crack depth. The x-ray spectrum is shown in Fig. 45. The largest peak is the result of the penetration of silver at the crack tip. Other peaks are from silicon and gold (the gold was deposited as part of the procedure for SEM photography). Fluorescent dye-penetrant techniques have also been used on the specimen subjected to dye-enhanced radiography. With the fluorescent dye, the cracks could be seen. However, under normal dye-penetrant examination, cracks located on the inner surface of the tube would not be detected easily, if at all.

The cracked SiC bar designated A-2 was also checked by dye-enhanced radiography. A crack was observed by normal radiography. After the bar was doped with AgNO_3 , several additional cracks could be faintly observed. Since the ultrasonic and dye-penetrant examinations revealed more than 20 cracks in Sample A-2, the 6-mm thickness of this sample may well

Fig. 42. Section of SiC Tube After Dye-enhanced Radiography, Showing Several Cracks. ANL Neg. No. 306-78-607.

Fig. 43. Higher Magnification of SiC Sample Shown in Fig. 42. ANL Neg. No. 306-78-610.

Fig. 44. Electron Micrograph of the Crack Examined by X-ray Fluorescent Analysis. ANL Neg. No. 306-78-613.

Fig. 45. X-ray Fluorescent Analysis of One of the Crack Tips in the SiC Specimen Shown in Fig. 42. ANL Neg. No. 306-78-608

represent the practical limit for crack detection in SiC via dye-enhanced radiography. The sample bar was also doped in a partially evacuated chamber to assist the penetration of the dye into the cracks. After this vacuum doping technique was tried, a few more cracks were indicated in the radiographs while the previously noted cracks were revealed more clearly. A comparison of crack indications via radiography for doped and undoped Sample A-2 is shown in Fig. 46. A comparison with results of other techniques is presented in Section X.

Fig. 46

Comparison of Crack Indications in Sample A-2, as Revealed via Conventional Radiography (RT) and Dye-enhanced Radiography [(DE)RT]. Neg. No. MSD-66132.

VI. ULTRASONIC TESTING

The capabilities and limitations of conventional preservice ultrasonic testing of ceramic heat-exchanger tubing are being determined. One of the problems in evaluation of SiC tubing is the high velocities and correspondingly long wavelengths of both longitudinal and shear waves. The velocities for SiC are about twice those found for steel, for example, and thus the ultimate resolution is significantly reduced. Measurements of velocity from hot-pressed SiC and SiN samples are shown in Table III. These data were obtained by means of a pulse-echo technique, using 25-MHz longitudinal and 5-MHz shear waves on 6-mm-thick samples.

TABLE III. Velocity of Sound and Wavelength for SiC and SiN

	Velocity, 10^5cm/s		Wavelength μm			
			20 MHz		100 MHz	
	Longitudinal	Transverse	Longitudinal	Shear	Longitudinal	Shear
SiN	11.1	6.30	560	320	110	60
SiC	12.0	7.65	600	380	120	77

A. SiC Tubes

In order to obtain reasonable resolution for inspection of 25-mm-diam tubing with a 1- to 3-mm-thick wall, it has been necessary to use a 20-MHz, 13-mm-diam focussed immersion probe. With this probe, convenient inspections can be carried out in an immersion tank with the beam incident on the outer wall. Because of the sharp curvature of the tube wall, a considerable amount of acoustic energy is scattered or refracted from the outer surface. Refracted longitudinal rays can cause the unwanted generation of shear waves which may make data interpretation difficult. The focussed probe minimizes this problem.

In order to evaluate the ability of the 20-MHz focussed probe to detect inner-wall defects, a section of Norton NC430 tubing was removed as shown in Figs. 47 and 48, and a groove 0.5 mm wide with depth varying linearly from 0.25 to 0.8 mm was cut in the inner wall. This groove could be detected to a depth of ~ 0.3 mm by normal-incidence longitudinal waves with the 20-MHz focussed probe. The groove can be detected over its entire length when shear waves are employed (via mode conversion of the longitudinal waves). Figure 49 shows a radio-frequency trace of an ultrasonic echo from the 0.3- and 0.8-mm-deep regions of the groove; the S/N ratio is $>2:1$. In addition, normal-incidence longitudinal waves were able to generate an echo from a 0.5-mm-diam by 0.5-mm-deep hole at the tube inner wall with a signal-to-noise ratio of about 3.

Fig. 47. Schematic of Section Removed from Norton NC430 SiC Tube.
Neg. No. MSD 66133.

Fig. 48

Photographs of NC430 Tube and
Section Removed for Evaluation.
Neg. No. MSD-66134.

Fig. 49. Radio Frequency (rf) Traces of Ultrasonic Shear-wave Reflection from Notch. Neg. No. MSD-66135.

A short section of an intact NC430 tube was also interrogated with the 20-MHz focussed probe and C-scan tank. The tube had a 25-mm diam and 3-mm wall thickness. Both normal-incidence longitudinal and refracted shear waves were used to inspect the tube. An Aerotech model UTA-3 transducer analyzer was used for this investigation. Radiographs showed the presence of several internal planar defects. Seven echoes from within the tube were observed with normal-incidence longitudinal waves. Six of the seven echoes coincided with planar or circumferential cracks indicated by radiography. No echoes were observed with refracted (45°) shear-wave beams in a pulse-echo mode.

B. SiC Bars

The 6 x 6 x 150-mm hot-pressed SiC bars A-1 and A-2 were investigated using the 20-MHz focussed immersion probe. Figure 50 depicts the transducer configuration for crack detection in the bar. Two examples of echoes are seen in Fig. 51. The top trace is from a crack at the top of Sample A-2. The bottom trace shows a reflection from the crack at the bottom of the bar. (Figure 72 in Section XII shows the location of all crack indications in the thermally cracked bar A-2, as determined by ultrasonic means with the 20-MHz probe. These results are compared to those of other techniques in that section.)

Fig. 50. Schematic of SiC Bar and Transducer Arrangement for Crack Detection. Neg. No. MSD-66136.

Fig. 51. Two Echoes from Cracks in SiC Bar (Sample A-2). Neg. No. MSD-66137.

In summary, it appears that in SiC heat-exchanger tubing, flaws on the order of several hundred μm (the estimated critical flaw size) can be detected but not necessarily sized via conventional ultrasonic inspection techniques. Nevertheless, as discussed in Chapter XII, where ultrasonic testing is compared with dye-penetrant examination, it is evident that many cracks are missed by conventional ultrasonic testing. This may be due to a small crack size or a crack orientation not favorable for detection via 45° shear waves.

VII. HOLOGRAPHIC INTERFEROMETRY

A. Background

The overall objective of this effort is to assess the applicability of holographic interferometry to the detection of cracks in ceramic heat-exchanger tubing. The work presented below is the continuation of studies reported earlier.⁸ In Ref. 8, the basic principles of crack detection by means of holographic interferometry are discussed, as well as the theoretical limits of resolution of the method.

Fig. 52. Schematic Representation of the Optical System Used for Holographic Interferometry. ANL Neg. No. 306-78-826.

B. Optical System for the Observation of Cracks

In line with the experience obtained from earlier efforts in this program, a real-time image system was adopted. Figure 52 shows a schematic representation of the optical system. Lens holography is utilized to facilitate the application of double-beam holography and to allow for reconstruction with a less coherent source when a reduction of the speckle noise is needed.

As discussed in Ref. 8, one of the most troublesome aspects of holographic interferometry is the localization problem. The interference fringes are localized on surfaces that do not coincide with the object surface. This effect is particularly important when the image must be magnified.⁸ The problem of controlling the localization plane has now been analyzed and the necessary equations derived. These equations show that it is always possible to force

the localization plane to be near the object surface. To achieve this objective, a real-time plate holder with a universal-motion mounting has been designed and built (Fig. 53). The plate holder has micrometric motions in three perpendicular directions, and can be rotated to the desired angle.

Fig. 53

View of the Real-time Plate Holder with Controlled Motions. Neg. No. MSD-66139.

The real-time observation of the fringe-pattern formation allows the introduction of the necessary corrective motions to bring the localization surface very close to the object surface. This system has been proved to be particularly useful in the thermal test that will be described later. The dilatation produced by heating introduces translations that localize the fringes far away from the object surface. These translations can be compensated by equal and opposite motions of the plate holder.

C. Experimental Applications

A number of experimental setups were tried and several concepts were explored. Both mechanical loading (via internal pressure) and thermal loading were used. In the case of turbine blades, it was concluded⁸ that mechanical loads revealed flaws more effectively than thermal loads. In the case of tubes, since they are redundant structures, thermal gradients can produce very high stresses and thus lead to crack detection.

For these studies, cracks were produced both by cutting with diamond wafering blades and by thermal shock. The cracks produced by the wafering blades had a circular profile; the maximum depth depends on the penetration of the blade, and the thickness approximated the thickness of the blades (0.2 mm). Thermal shock produced arbitrary patterns of cracks, some visible by microscopic observation, others too tight to detect optically.

D. Internal Pressure Tests

1. Phase-difference Technique

An internal-pressure system was utilized to apply the phase-difference concept to enhance crack detection in heat-exchanger tubes. A device was built which was capable of generating pressures up to 2000 psi (internal leaks in the valve system of the oil pump prevented steady pressures higher than this). A saw-cut longitudinal crack 1500 μm deep and 9 mm long

was made in a heat-exchanger tube. The crack was focussed on the plate so that the axis of rotation of the plate coincided with the projection of the crack on the tube surface. It can be shown that for small rotations, the rotation of the plate⁸ is equivalent to a rotation of the object. Figure 54 illustrates the effect of the superposition produced by the displacement due to the internal pressure plus the fictitious rotation. Displacements added on one side of the crack are sub-

Fig. 54. Phase-difference Technique to Enhance Crack Detection. Neg. No. MSD-66140.

tracted from the other. As a result, the number of fringes on one side of the crack is different from the other side, making the crack clearly visible. The

result of this technique is illustrated in Fig. 55. The idea is extremely useful since it can be exploited to enhance extremely small phase differences. Mechanisms other than plate rotation can be employed to introduce the required phase differences.

Fig. 55

Phase-difference Image of Saw-cut Crack in a Heat-exchanger Tube. Neg. No. MSD-66141.

2. Analysis of the Crack Tip Field: Determination of the Crack Stress-intensity Factor

The possibility of not only detecting the presence of a crack but also finding the stress-intensity factor was explored in this series of tests. Since we could not introduce high enough pressures in the ceramic tubes to quantitatively measure a very shallow crack, a plastic pipe was utilized. The characteristics of this pipe are as follows: outside diameter $d_o = 4.85$ cm (1.91 in.); inside diameter $d_i = 4.06$ cm (1.6 in.); modulus of elasticity $E = 2800$ MPa (4×10^5 psi); Poisson's ratio $\nu = 0.37$. A circular saw-cut crack 185 mm long, with maximum depth $d = 500$ μ m, was made in the pipe.

The double-beam technique¹⁹ was utilized to observe the displacement field in the tube surface. The sensitivity of the technique depends on the angle of illumination subtended by the illuminating beam with the surface normal. The sensitivity is given by equation

$$\delta = \frac{\lambda}{2 \sin \alpha} \quad (7.1)$$

where λ is the wavelength of the laser light and α is the angle formed between the illuminating beam and the surface normal. A $\sin \alpha$ value of 0.3 was used. Then,

$$\delta = 10.5 \times 10^{-4} \text{ mm}$$

Fig. 56. Coordinate System for the Crack Displacement Field Analysis. Neg. No. MSD-66142.

3. Method for Obtaining the Stress-intensity Factor

Let us project the displacement field on a plane tangent to the cylinder (Fig. 56). The crack is a surface crack and is subjected to a 3-D state of stress. No rigorous solution exists for a surface crack on a cylinder. The v -displacement field, or field of displacement, parallel to the y -axis is given by

$$v = \frac{K_1}{G} \sqrt{\frac{r}{2\pi}} (2 - 2\nu) \sin \frac{\theta}{2}, \quad (7.2)$$

where r is distance from crack tip and G is the shear modulus.

From the holographic moiré pattern obtained by pressurizing the tube, we can obtain the v -field and by replacing the corresponding constant we can obtain K_1 .

A holographic moiré pattern was obtained for the pressure $p = 0.35$ MPa (50 psi). The pattern is shown in Fig. 57. The presence of the crack produces a perturbation. The crack tip is defined by the fringe configuration shown in Fig. 58. The fact that two moiré fringes seem to join indicates the presence of a singularity in the displacement field.

Fig. 57

θ Displacement Field for a Surface Crack on a Tube Subjected to Internal Pressure. Neg. No. MSD-66143.

Fig. 58

Configuration of Moiré Fringes in the Neighborhood of the Crack Tip. Neg. No. MSD-66144.

If the displacement field is represented by Eq. 7.2, the fringe orders in the y-direction should follow a $\sqrt{r}$ variation; consequently, in a log-log plot, the fringe orders should plot as straight lines with a slope of 0.5. The plots for the left and right sides of the crack tip are shown in Figs. 59 and 60, respectively. In both cases the slope is 0.49.

Fig. 59. Fringe Orders vs Distance to the Crack Tip (left side). Neg. No. MSD-66145.

Fig. 60. Fringe Orders vs Distance to the Crack Tip (right side). Neg. No. MSD-66146.

4. Computation of K_I

Inserting the appropriate values in Eq. 7.2, we obtain

$$v = 0.0243 \sqrt{r} K_I \times 10^{-4} \quad (7.3)$$

or

$$K_I = \frac{41.09 v}{\sqrt{r}} \times 10^4. \quad (7.4)$$

From the picture for the 2.5 order, the distance r is equal to 0.39 cm (0.154 in.). The displacement $v = 2.6 \times 10^{-4}$ cm; substituting into Eq. 7.4, we find

$$K_I = 0.12 \frac{N}{m^{3/2}} (107 \text{ lb/in.}^{3/2}).$$

An approximate value of K_I can be computed at the midpoint of the crack. The crack can be assumed to be in single-edged plate, since the crack is long and the crack depth is small relative to the wall thickness:

The value of K is then

$$K = 1.12 \sigma \sqrt{\pi a} \quad (7.5)$$

which leads to a value of $K_I = 0.1 \text{ MN/m}^{3/2}$.

The ratio between the values at the surface and the midpoint is 1.23. This result cannot be checked, since there is no known solution. The solution found by Tracey²⁰ for semicircular cracks also gives the ratio 1.23, with the maximum again at the surface.

Fig. 61. Heat-exchanger Tube on the Holographic Table. Neg. No. MSD-66147.

5. Temperature Tests

The introduction of a temperature gradient can produce very high stresses on a tube. Tests were conducted on heat-exchanger tubes by means of a linear heating element located in the center of the tube (Fig. 61). The outer-wall temperature was measured with a thermistor. Cracks produced in several tubes by thermal shock have been observed in real time and photographed. Some cracks which are visible to the eye cannot be effectively recorded by photography. Fig-

ures 62 to 65 show examples of cracks which can be photographed. Figure 65 is particularly interesting because although the saw-cut crack is very shallow, the discontinuity produced by the presence of the crack is clearly visible in the fringe system.

Fig. 62. Transversal Crack in SiC Tube Produced by Thermal Shock. ANL Neg. No. 306-78-831.

Fig. 63. Curved Crack in SiC Tube Produced by Thermal Shock. ANL Neg. No. 306-78-830.

Fig. 64

Vertical Crack in SiC Tube
Produced by Thermal Shock.
ANL Neg. No. 306-78-829.

Fig. 65

Vertical Saw-cut Crack in SiC Tube.
Maximum depth is $500\text{ }\mu\text{m}$. Discontinuity of the fringes is clearly visible.
ANL Neg. No. 306-78-824.

VIII. INTERNAL-FRICTION MEASUREMENTS

The objective of this task is to employ flexural resonance bar internal-friction measurements to determine the presence of flaws in SiC ceramic bars.

A. Procedure

The experimental system, shown schematically in Fig. 66, consists of the following elements:

1. A flexural fixture to suspend the sample at nodal points and position the ends of the sample at the tips of the magnetostrictive drive and pick-up transducers (Type 3055A transducers--Electro Products Laboratories, Inc., Chicago, IL 60648).
2. A General Radio Type 1162A frequency synthesizer to generate a continuous stable ac signal measurable to within 0.1 Hz.
3. A General Radio Type 1191 frequency counter to provide readout of the drive and detected frequency.
4. Hewlett-Packard VTVMs, Type 400D, to monitor drive and detected voltages.
5. A Kron-Hite Type DCA10 amplifier to control the drive voltage.

Fig. 66

Experimental Arrangement for
Internal-friction Measurements.
Neg. No. MSD-66149

In this system, square low-C steel plates (0.8 x 0.8 x 0.1 cm) are cemented to the ends of the ceramic rod (0.6 x 0.6 x 15 cm), using either Eastman 910 two-part adhesive or Duco cement. The rod, whose ends are now ferromagnetic, is placed into the fixture and the frequency f provided to the drive transducer at voltage V_g is slowly varied. At resonance frequency f_r , a maximum appears in V_g , the voltage on the detector or gage VTVM. Minute adjustments are made in the nodal positions and the spacings between the magnetic disks and the transducer tips so as to maximize V_g . Finding f_r and maximizing V_g is tedious and time-consuming when a rod of unknown f_r is first studied.

The damping δ is calculated from the resonance response curve (plot of V_g versus f in the vicinity of f_r) by the relation

$$\delta = \frac{\pi \Delta f}{f_r} \quad (8.1)$$

where Δf is the width of the curve at 0.707 V_g (max). Presumably, as flaws appear in the ceramic, δ should decrease. In addition, δ and f_r are coupled by way of the modulus-defect phenomenon, and as δ increases in a given sample, f_r should decrease. Thus each internal-friction measurement provides two indices of the quality of the component, δ and f_r .

An alternate means of measuring δ follows from the fact that as δ increases at a given input voltage V_d , the output signal V_g should decrease; hence,

$$\delta = \frac{V_d}{V_g} \quad (8.2)$$

By means of Eq. 8.2, δ may be determined simply by determining f_r and V_g at f_r for a given V_g . The disadvantage of this simpler technique is that the proportionality constant K must be obtained by independent measurement.

B. Results

1. Damping in Quenched Rods

SiC bars A1 and A2, described in Section II, were used. Low-C steel plates were cemented to both rods. Sample A-1 was used as a control, while Sample A-2 was sequentially quenched according to the following schedule:

7/9/78	500°F for 5 min, air quench
7/15/78	500°F for 5 min, ambient water quench
7/22/78	500°F for 5 min, ambient water quench

The damping and fr were measured immediately after the end pieces were reglued and again after seven days to allow the sample to dry and the glue to thoroughly set. Each time δ and fr were obtained on Sample A-2, they were also measured on Sample A-1 to ensure that no changes occurred in the electronic and mechanical measurement system. The results are summarized in Table IV.

TABLE IV. Summary of δ and fr Measurements

Date	Sample A-1	Control	Sample A-2	Control	Remarks
	fr, Hz	Δf , Hz	fr, Hz	Δf , Hz	
7/1/78	2540.6	1.0	2747.1	1.0	
			2797.0	1.0	Reglue Sample A-2
7/9/78	2540.2	1.1	2795.0	1.2	Air Quench Sample A-2
7/15/78	2540.3	1.1	2794.8	1.2	
			2545.3	8.4	Water Quench Sample A-2
7/22/78	2541.1	1.1	2536.2	11.6	
			2516.7	17.7	

It is evident that the system produces results which are extremely reproducible, since there is negligible change in the control Sample A-1. It also appears that the air quenching is insufficient to produce thermal cracking. The second quench produced visible cracks on Sample A-2, and the increase in Δf and decrease in fr are easily measured. The cracks appear to grow with time, perhaps because of stresses generated during handling, so that Δf and fr are changing although no additional quenching has been done.

Resonance-bar internal-friction measurements appear to be an effective method for flaw detection in SiC bars. However, Sample A-2 is extensively fractured, containing perhaps 20 or more cracks. It is uncertain whether or not this technique is sensitive enough to detect the presence of a single small crack. Initial results on a 6 x 6 x 150 mm SiC bar with a small dent (100 x 50 μ deep) shows the dent cannot be detected with internal friction measurements. Further efforts will be required to thoroughly assess the capability of internal-friction measurements to detect flaws in SiC heat-exchanger tubing and to determine whether this technique would be cost effective.

2. Comparison of fr Results with Theoretical Predictions

Since rods with square cross sections are employed, it is difficult to compare the measured values of fr with those predicted by elasticity theory. However, a rough comparison may be made by calculating E from velocity data and then calculating fr by means of a relation valid for circular rods, as shown in the following.

For SiC,

$$v_t = 7.65 \times 10^5 \text{ cm/sec}$$

$$v_\ell = 12.0 \times 10^5 \text{ cm/sec}$$

$$\rho = 3.18 \text{ g/cm}^3$$

From elasticity theory,

$$G = \rho v_t^2 = 1.86 \times 10^{12} \text{ dynes/cm}^2$$

$$C_{11} = \rho v_\ell^2 = 4.58 \times 10^{12}$$

$$\lambda = C_{11} - 2G = 0.86 \times 10^{12}$$

$$E = (2G^2 + 3\lambda G)/(G + \lambda)$$

$$E = 4.3 \times 10^{12} \text{ dynes/cm}^2$$

From elasticity theory for circular rods,

$$fr \text{ (fundamental)} = f_1 = (0.890) \frac{d}{\ell^2} \left(\frac{E}{\rho} \right)^{1/2}$$

$$d = 2.67 \times 10^{-3} \text{ cm}$$

$$f_1 = 2757 \text{ Hz.}$$

This compares favorably with a measured value of fr for SiC of about 2600 Hz. The validity of the flexural system is thus confirmed.

Both δ and fr appear to provide measures of flaws in SiC.

IX. INFRARED SCANNING

Investigations have been carried out to assess the heat-transport properties of ceramic tubing by infrared scanning techniques. An AGA model 750 camera was used for this investigation. Initial efforts were directed toward flaw detection in ceramic tubes. Flaws may distort transient heat-flow patterns, and consequently show up as temperature distributions in infrared images. However, in our effort it was not possible to observe small ceramic-tube anomalies except in severely cracked tubing. Nevertheless, infrared images can be used to rapidly indicate variations in thermal conductivity (or thermal diffusivity) between similar types of tubing or to compare heat-transport properties in tubes from different manufacturers.

Thermal transients were first generated in 200-mm lengths of 25-mm-diam SiC tubing manufactured by Norton (NC430) and Carborundum (SKT). To assure uniform emissivity among the various tubes, they were sprayed with graphite. In the first effort, thermal gradients were generated by changing the temperature of water flowing through the tubes. It was assumed that variations in thermal properties, or delaminations, would show up as hot or cold spots on the infrared image of the tube. Reproducible results were difficult to obtain and a second approach was tried. The ends of several 200-mm-long, 25-mm-diam tubes, initially at room temperature, were simultaneously lowered into a water bath. The axial heat-flow patterns were then observed as a function of time. In this case reproducible differences in temperature distributions could be observed. The relationship between temperature gradients along the tubing and variations in thermal conductivity can be approximated by a model which assumes that the tube is a rod. Thus, in principle, the observed temperature gradients indicated by the thermogram can be related to variations in thermal conductivity. A 10% variation in thermal conductivity should result in a variation in ΔT of about 1°C .

Figure 67 shows a schematic of the equipment used. Figure 68 is a thermogram (initially in color) of seven tubes. The different colors (or different shades of gray) indicated can be related to surface temperature. The tubes in Fig. 68, identified by letters, are nominally of the same dimensions (250 mm long, 25 mm in diam, 3-mm wall thickness). Tubes A, E and G are Norton NC430. Tubes B, D, F, and J are Carborundum super KT. All three NC430 tubes conducted the heat better than any of the super KT tubes. The variation in temperature along the tubes was about 2°C . Of the four super KT tubes, tube F was severely cracked by thermal shock. This tube conducts heat less effectively than the other super KT tubes. The Norton tubes show generally uniform heat-flow patterns.

Fig. 67. Schematic of Equipment Used to Obtain Infrared Images of Sections of Heat-exchanger Tubing. MSD Neg. No. 65343.

Fig. 68. Thermogram of Seven SiC Heat-exchanger Tubes. MSD Neg. No. 65344.

Efforts are continuing to carry out effective quantitative analyses of the thermal images and confirm by other techniques the results obtained by infrared imaging.

The temperature of the rod may be described as a function of position x and time t by the following equation:²¹

$$T(x, t) - T_{\text{amb}} = \frac{2}{L} \sum_0^{\infty} \frac{K(\beta_n)(-1)^n T_0}{K\beta_n^2 + \nu} \cos(\beta_n x) [1 - e^{-\nu t - K\beta_n^2 t}] \quad (9.1)$$

$$K = k/\rho c, \nu = HP/\rho c\omega \text{ and } \beta_n = (2n + 1)\pi/2L$$

where

T_{amb} = ambient temperature of air

T_0 = bath temperature -- ambient temperature

k = thermal conductivity = 120 watts/M°C

ρ = density = 3100 kg/M³

H = coefficient of heat transfer = 20 watts/M²°C (Ref. 22)

p = circumference of rod = 8 cm

ω = cross-sectional area = 2.2 cm

L = tube length = 15 cm

c = specific heat = 627 J/Kg°C

The values of K , ρ and c are for NC430.

Figure 69 shows a graph of the theoretically predicted temperature versus time for a point midway between the water-bath surface and the end of the tube. Also shown is the curve of temperature versus time for a thermocouple placed at this position and monitored during a heating cycle. The experimental and calculated values are in reasonable agreement. The variation in temperature resulting from a change in thermal conductivity can be estimated from Eq. 9.1. For example, a change in thermal conductivity of 10% results in a temperature change at the middle of the tube, 100 sec after immersion in the water bath, of ~1°C. This can be detected by the infrared camera as it is capable of resolving temperature differences of 0.2°C.

Fig. 69

Temperature vs Time at Middle
of SiC Tube Immersed in Water
Bath. Neg. No. MSD-66150.

To ascertain the adequacy of the infrared camera for measuring temperature gradients, the temperature difference between the middle of the tube and its end, as determined with the infrared camera, was compared with the data obtained from two thermocouples. The results from the thermocouples are shown in Fig. 70. The value of ΔT at 120 sec is 3.1° . Using the AGA camera with a full-scale reading of 10° , the observed temperature difference was 2.2°C . While not in close agreement with the thermocouple values, this result is good enough to suggest that thermal-conductivity differences of only 5-10% between tubes can be observed by infrared techniques.

Fig. 70

Temperature Difference between Top
and Middle of SiC Tube as a Function
of Time After Immersion in Water Bath.
Neg. No. MSD-66151.

These tests suggest a rapid means of assessing thermal heat transport properties of ceramic tubing is available.

X. COMPARISON OF TECHNIQUES

A. SiC Test Bars

Various NDE techniques were used to examine the cracked hot-pressed SiC bar (Sample A-2) described above. Except for the cracks, induced by thermal shock, the bar was relatively free of defects; thus, crack-detection techniques could be effectively compared. All the cracks were assumed to intersect at least one of the four surfaces of the bar. Thus, conventional dye-penetrant examination was used, as well as conventional and dye-enhanced radiography, conventional ultrasonic testing, acoustic microscopy, internal-friction measurement and infrared scanning. It appears that no single technique is adequate for detecting all of these surface cracks.

1. Radiography

Figure 71 compares the results obtained by dye-penetrant testing, conventional radiography, and dye-enhanced x-radiography. Numerous cracks

Fig. 71. Comparison of Dye-penetrant Testing (PT), Conventional Radiography (RT), and Dye-enhanced Radiography [(DE)RT] for Crack Detection in Sample A-2. Neg. No. MSD-66152.

were revealed by the dye-penetrant examination. (Sample A-2 is particularly suitable for inspection by this technique as it has very smooth and clean surfaces.) With conventional radiography, only one crack and a branch were detected. With dye-enhanced radiography many more cracks could be seen, including some missed in the dye-penetrant examination. Nevertheless, a large number of cracks were still missed. The tight cracks ($<20 \mu\text{m}$ wide) present in this specimen permitted only a limited accumulation of doping

agent (silver nitrate), even when the sample was placed in a partial vacuum to assist in the flow of penetrant. Furthermore, the sample is relatively thick (6-mm); this contributes to the low resolution observed with this technique.

2. Conventional Ultrasonic Testing

Figure 72 compares the dye-penetrant results with those obtained using a 20-MHz, 45° shear-wave beam. The circles in Fig. 72 represent regions of the bar where ultrasonic flaw signals were obtained. These signals exceeded (by 6 dB) the background signal level typically found in a crack-free bar Sample A-1. The areas where flaws were detected are superimposed on the

Fig. 72. Comparison of Conventional Ultrasonic and Dye-penetrant Techniques for Crack Detection in Sample A-2. Circles are regions where ultrasonic echo signals were detected. Neg. No. MSD-66153.

dye-penetrant results. Not all cracks were readily detected by ultrasonic testing, and some defects were revealed by the ultrasonic but not by the dye-penetrant examination. Thus, the ultrasonic technique was more effective than radiography for crack detection (but not characterization).

3. Acoustic Microscopy

The results obtained for SiC Sample A-2 using acoustic microscopy are discussed in Section IV. Many defects were observed (more than with conventional ultrasonics) and the ability to characterize the cracks, including their depth, was demonstrated. Acoustic microscopy can, in principle, detect and characterize cracks (as well as other subsurface defects) more effectively than any other technique evaluated in this study. The difficulty is that when the sample thickness approaches 6 mm, the acoustic beam is diffracted or scattered from certain types of cracks. This can make data interpretation very difficult. With thinner samples, such as ceramic tubing (1-3 mm thick), the problem is less severe. The acoustic microscope is, of course, a rather sophisticated instrument and not employed for routine examination of ceramic components. Some development would have to be carried out for industrial applications.

4. Internal-friction Measurements

Internal-friction measurements were discussed in Section VIII. This technique successfully detected cracking in Sample A-2. However, the technique is difficult to use and it is questionable whether or not internal-friction measurements would adequately reveal the presence of a single crack in a long (>10 cm) specimen. The method is also limited in its ability to indicate the size and location of defects, and has little applicability to systems with complex geometry.

5. Infrared Scanning

Attempts to observe differences in heat-flow patterns between Samples A-1 (crack-free) and A-2 (severely cracked) with an infrared camera were not successful. However, this method was successfully applied to cracked tubing, as discussed in Section IX. The difference is that the SiC bars are thicker and contain shallower cracks than the tubing. The depth of the cracks in the SiC bar may equal as little as 10% of the sample thickness. Thus, the cracks did not interrupt the heat flow as effectively in the bar as in the

SiC tubing, where their depth was estimated to be up to 75% of the wall thickness. As a result, the heat-flow variation between cracked and crack-free bars was beyond the detection capability of the infrared camera.

6. Fluorescent Dye-penetrant Testing

Fluorescent dye-penetrant testing has been shown to be an inexpensive and effective means for detection of surface cracks in the SiC bars prepared for this study. However, the usefulness of this technique is limited when cracks are particularly tight, short, or filled with a foreign substance, or the surfaces (e.g., tubing inner walls) not readily accessible. In the case of tight cracks, ultrasonic and some dye-enhanced radiography indications (surface flaws) have been observed where dye-penetrant examinations have not shown flawed areas.

B. SiC Tubing

Various NDE techniques were evaluated for their effectiveness in detecting flaws in SiC heat-exchanger tubing samples. The tubes were not of high quality; that is, they were filled with previously uncharacterized defects. The discussion below compares the effectiveness of the various methods which were described in detail in earlier sections. While a comparative study using SiC bars was relatively easy, the comparison of techniques for the tubes was difficult.

1. Radiography

Conventional radiography led to the detection of a large number of void-like and crack-like defects in several of the tubes examined. Sectioning of the tube in several places indeed showed the presence of numerous subsurface void-like and planar defects, up to 2 mm long, in addition some flaws were found not revealed by radiography. In one specimen (a 1-mm-thick CVD SiC tube section), four of five inner-surface radial cracks were missed by conventional radiography. All five, however, were detected by the dye-enhanced radiographic technique described in Section V. It appears that normal radiographic methods are not adequate to detect all critically-sized subsurface or surface flaws in SiC tubing and that dye-enhanced radiographic techniques are useful for crack detection, particularly for areas where dye-penetrant testing cannot be carried out efficiently (such as tubing inner walls).

2. Conventional Ultrasonic Testing

Two tubes (NC430) were examined by the ultrasonic techniques described in Section VI. One tube had an area in which six flaws had been indicated by radiography. All of these were detected as subsurface pseudoplanar circumferential defects by normal-incidence longitudinal waves at 20 MHz. Other subsurface reflectors were also indicated by ultrasonic testing, but

could not be confirmed by radiography. With a notched sample cut from an NC430 tube, it was demonstrated that defects on the order of several hundred μm in size could be detected in SiC tubes, but not necessarily characterized, by 20-MHz shear waves. Thus, ultrasonic testing appeared to be more useful than radiography for detecting the numerous circumferential defects and cracks found in the SiC tubes examined.

3. Acoustic Microscopy

The SiC tubes were difficult to examine by acoustic-microscopy techniques because of the presence of many flaws and large variations in the acoustic properties of the tubes. Nevertheless, it has been demonstrated that acoustic microscopy can be adapted for the examination of tubing. Variations in acoustic properties, as well as subsurface defects and surface-breaking cracks, can be readily detected for flaws of sizes comparable to or less than the estimated critical flaw size for SiC tubing (100-200 μm). The examination of the tubing can be carried out at a rate comparable to visual examination with a microscope. Furthermore, the acoustic-microscope system could, in principle, be adapted for industrial applications of SiC-tubing inspection.

4. Holographic Interferometry

Holographic interferometry techniques have been demonstrated to be effective for detection of surface-breaking cracks. This technique is relatively insensitive to surface conditions, as are dye-penetrant methods and acoustic techniques. The minimum flaw depth detected to date in this effort is only 500 μm , which suggests that ultrasonic techniques are more sensitive; however, the use of optical magnification schemes may make optical techniques superior to acoustic ones. Detection of cracks <500 μm deep would probably require considerably more development than has been carried out in the current effort. The holographic system may be adaptable to inspection of the tubing inner wall, in which case the technique could be very useful. It will be possible to analyze holographic-interferometry images to obtain information on crack characteristics not attainable by conventional ultrasonic or other techniques discussed in the present report. If holographic interferometry were adapted for inspecting the bore side of a tube, it could be a reliable method for inner-wall crack detection and characterization. It would be better than acoustic-microscopy in those situations where the acoustic properties of the tube are poor enough to make interpretation of acoustic micrographs very difficult. Holographic interferometry could provide the basis for a rapid inspection technique in conjunction with a semiautomatic system to apply the required stress and produce and analyze the holographic interferograms. Such a system can, in principle, be developed.

5. Overload Proof Testing

Experimental efforts on overload proof testing have not been carried out in this program. However, the potential application of this method

to flaw detection in ceramic heat-exchanger tubing was discussed in Section III. Overload proof testing could be a useful method for finding defective tubes. However, because of the potential for generating large thermal stresses while the tubes are in service, a system would have to be developed to pressurize the tubes to levels exceeding 35 MPa (5 ksi). This could be impractical and would not necessarily simulate the service-induced stresses. Furthermore, this testing procedure could lead to crack-growth instabilities during unloading.

6. Strategy for Preservice Testing of SiC Heat-exchanger Tubing

No single technique among those discussed in the present report is capable of detecting all flaws of critical size in SiC tubing. A procedure should be established which is cost effective yet gives reasonable assurance that critically sized defects will be detected. Since cost is a major factor in developing such a strategy, relatively simple techniques should be employed to screen grossly flawed components. Visual and fluorescent dye-penetrant examinations should be used as the initial NDT techniques, along with conventional radiography and possibly overload proof testing. Further examination, preferably by means of dye-enhanced radiography and/or acoustic microscopy and holography, will be required for components passing initial inspections. For tubing, an alternative to development of acoustic-microscopy techniques would be examination with conventional ultrasonic techniques at ~20 MHz. Additionally, infrared-scanning techniques applied to SiC tubing could provide a rapid method for detection of anomalous heat-transport characteristics. Holographic-interferometry techniques could also be developed for surface flaw detection if other techniques are found inadequate to assure reliable tubing. Experience acquired with components examined by the techniques described, and failed by simulated or actual field testing, will ultimately lead to identification of the most cost-effective preservice examination method for SiC heat-exchanger tubing.

XI. IN-SERVICE EXAMINATION OF CERAMIC HEAT-EXCHANGER TUBING

All of the techniques discussed in this study were evaluated for pre-service inspection of ceramic heat-exchanger tubing. The important area of in-service examinations must also be considered. Many of the techniques readily employed for preservice examination cannot be used effectively here because of the physical constraints encountered when inspecting a completed heat exchanger. We assume that the tubes are accessible only from the bore side during system shutdown. Of the techniques discussed above, conventional ultrasonic testing should be the most practical choice for in-service inspection. An inspection would be carried out from the bore side with a device similar to that used for metallic heat exchangers,²³ except that higher frequencies would have to be used. A small probe operating at 20 MHz could be moved through the bore of the tube to carry out an examination of the tube wall. Both longitudinal and shear waves could be generated in radial and circumferential

patterns to scan the entire tube. With the system interfaced to a computer, rapid scanning and data reduction could be carried out with commercially available equipment. Results from in-service tests would be compared with results from preservice testing. Flaws with characteristic dimensions on the order of several hundred microns should be detectable.

Infrared scanning could be adapted to bore-side examinations. The information obtained would indicate both hot spots (wall thinning; erosion) and areas with poor thermal-transport properties, thus identifying anomalous areas of the tube. This examination could even be carried out at elevated temperatures.

Radiographic techniques would be difficult to implement because the radiation source and film would have to be in different tubes. Acoustic microscopy may be feasible for short tube lengths, but the requirements of this technique suggest that it would be impractical for in-service inspection of long tubes (<25 cm).

Holographic-interferometry techniques employing pulsed lasers may be applicable to detection of surface cracks in a bore-side inspection of straight tubes, but a considerable amount of development work would be required. Conventional visual examination using flexible borescopes could be effective if coordinated with an ultrasonic examination. One other technique not discussed here but discussed in Ref. 5, surface-wave generation, may be adaptable to bore-side inspections and could be effective for detection of inner-wall cracks developed during the heat-exchanger service life. The most critical aspect of in-service inspection will be acquiring a reproducible set of data from a preservice test which can be compared with future in-service examination data.

XII. SUMMARY

This report discusses the adequacy of several NDE techniques for the detection of flaws in SiC heat-exchanger tubing. Experimental results regarding adaptability to ceramic tubing and minimum detectable flaw size have been obtained for conventional ultrasonic testing, acoustic microscopy, conventional and dye-enhanced radiography, holographic interferometry, infrared scanning and internal-friction measurements. The advantages and disadvantages of overload proof testing are also discussed. NDE techniques applicable to ceramic heat-exchanger tubing but not evaluated in this study include microfocus x ray,³ microwave NDE,⁴ acoustical surface waves,⁵ and photoacoustic spectroscopy.⁶

No single NDE technique will be sufficient to detect all types of critically sized flaws. Preservice inspection should include traditional visual and dye-penetrant examinations for outer-wall surface defects, and possibly overload proof testing to screen weak ceramic tubing. Further examination should employ dye-enhanced radiography for inner-wall surface defects and acoustic microscopy or conventional ultrasonic testing at 20 MHz (and possibly holographic interferometry) for other defects. Anomalous heat-transport characteristics may be detected by infrared techniques. Holographic interferometry could be useful if the other techniques mentioned are found to be deficient.

For in-service examinations, where it is assumed that access to the tube is from the bore side only, inspection employing 20-MHz longitudinal and transverse waves appears to be the most practical technique. Infrared scanning through the bore could possibly provide data with regard to hot spots, wall thinning and anomalous heat-transport characteristics. Holographic interferometry, while requiring extensive development, may be capable of providing inner-wall crack-detection data not generated by other techniques. Table V summarizes the attributes of the various techniques evaluated in this report.

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TABLE V. Comparison of Several NDE Techniques for Flaw Detection in SiC Heat-exchanger Tubing^a

Method	Relative Effectiveness for Flaw Detection							Effectiveness for Characterization of Detected Flaws	Adaptability to Tube Geometry	Adaptability to Preservice Examination	Adaptability to In-service Examination	Reliability of Technique	Extent of Development Required for Commercialization	Relative Rate at Which Tubes Could Be Inspected
	Surface				Subsurface									
	Cracks		Pitting											
	ID	OD	ID	CD	Inclusions	Voids								
Acoustic Microscopy	G ^b	E	G	E	G/E	E	E	F	G	P	G/E	high	slow	
Dye-enhanced Radiography	G	G	G	G			G	G	E	P	G	some	slow	
Conventional Radiography	F	F	G	G	G	G	G	G	E	P	G	none	slow	
Conventional Ultrasonic Testing	G	G	G	G	G	G	F	F/G	E	G	G	some	fast	
Overload Proof Testing	G	G	?	?	?	?	P	P	P		F	some	fast	
Holographic Interferometry	?	G/E	P/F	F/P			E	F/G	G	F	E	very high	fast	
Infrared Scanning	P	P	P	P	P	P	P	E	E	G	G	some	fast	
	effective measure of heat transport properties													
Internal-friction Measurement	F	F	?	?	?	?	P	P	P	F	P	very high	slow	
Fluorescent Dye Penetrant Testing		G		G			F	E	E	F	G/F	none	slow	

^aFor tubes with wall thickness ≤ 3 mm.^bE = excellent; G = good; F = fair; P = poor; . = not applicable.

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