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LOCATING TUBE BLOCKAGE THAT X-RAY CANNOT DETECT

By

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This paper presents the results of an NDT program established to detect and define foreign-material inclusions in closed metal tube assemblies (when the foreign material is of a composition that cannot be detected by X-ray). These results may well be useful in resolving many inspection problems.

INTRODUCTION

In 1967 a temporary nondestructive-testing enigma arose in the inspection of the propulsion nozzle being fabricated for the nuclear-reactor-powered rocket engine.* Some of the 320 coolant tubes lining the inside surface of the nozzle were found, by flow tests, to be blocked by accidental foreign-material inclusions. Optimizing of X-ray technique revealed some thick pieces of rubber O-ring caught in narrow portions of the tubes, but one known blockage still could not be detected. Immediately available nondestructive tests other than X-ray were not applicable to location of the blockage and correction of the problem; the standard NDTs, such as penetrant, magnetic-particle, ultrasonic, and eddy-current techniques, were of no help.

Cutting of the tubes, probing for the unlocated blockage, and welding repair did result in removal of the blockage and subsequent acceptance. The foreign material was found to be a stiff piece of plastic of very low density. But, there still remained the problem of the failure of nondestructive testing to perform its primary tasks, these being, first, to prove that the product is without functional defects, or, second, to locate and define the defects if they do exist.

With interplanetary manned space flight as the goal of the nuclear rocket program, this deficiency in NDT required correction. Even though removal of the coolant-tube blockage and welding repair readily effected in this case, there remained the possibility that something like a plastic chip could lie in a tube in a position that would permit an adequate flow test but would later move to a position that would cause blockage of the liquid-hydrogen coolant. The result could be tube burnout and consequent nozzle malfunction. Also, other types of delayed-blockage possibilities exist. An example would be the presence of scattered fibrous contamination such as threads that later could tangle into a ball at a narrow place in the tubes or at a turn.

* The Nuclear Engine for Rocket Vehicle Application (NERVA) program is administered by the Space Nuclear Propulsion Office, a joint agency of the U. S. Atomic Energy Commission and the National Aeronautics and Space Administration. Aerojet-General Corporation as prime contractor for the engine system and Westinghouse Electric Corporation as principle subcontractor responsible for the nuclear subsystem, are developing a nuclear propulsion system.

A study was conducted to determine the limits of X-ray capability to detect foreign materials within the coolant tubes. Because the high-nickel alloy of the nozzle wall was generally greater than 1/2 in. in thickness, the energy level of the X-ray needed to penetrate the metal was too high to permit absorption and resulting X-ray shadows of thin low-density materials. Wood chips, bits of cloth, plastic chips, thin pieces of rubber, and similar low-density materials were found to be capable of causing blockage and escaping detection by standard X-ray procedures.

Four NDT methods were selected for trial to resolve the problem. Three selected in order of usefulness and reliability were determined to be first, negative radiography; second, neutron radiography, and third, liquid crystal. The fourth, ultrasonics, was eliminated as inadequate. No single test was found to be totally reliable by itself, but combinations of two or more tests did provide adequate assurance of freedom from foreign materials of significant size in the nuclear-rocket nozzle system.

The results of the separate investigations follow.

I. NEGATIVE RADIOGRAPHY

DEFINITION; NEGATIVE RADIOGRAPHY

Negative radiography employs a heavy liquid that will absorb X-rays. The liquid creates a radiographic shadow of itself on film. When a low-density material such as plastic or rubber is surrounded by a heavy liquid, the light material prevents part of this X-ray absorption; and the material's image appears on the film as an apparent void within the radiographic shadow of the liquid. The location and shape of the low-density blocking object can be determined by this method with standard X-ray equipment, when the tube system or individual tube is filled with Freon 114-B2.(1)

TEST RESULTS (See Figures 1 and 2)

Figure 1 is a copy of the X-ray made of the final test specimen. Note that this X-ray is made through a 1/2 in. nickel alloy wall. Comparison of the X-ray with Figure 2, the section view of the specimen, will provide an understanding of the image. In the fluid-filled tubes, the thicker samples appear as darker images. This is the negative radiography effect. The image effect is the reverse of that for the faint image visible in the air-filled tube.

Cellulose-acetate plastic, 0.065 in. thick, was clearly imaged on film when the tube was filled with Freon 114-B2. Because 0.065 in. is the smallest thickness of low-density material used in this series of tests, this thickness represents the maximum sensitivity that can be assured to date when X-rayed through the 1/2 in. wall. However, greater sensitivity appears to be possible.

Either rubber or plastic materials greater than 0.065 in. thick are seen with proportionately better contrast. The thicker the specimen, the darker the image of the void it appears to create within the fluid column.

Figure 1 - Negative Radiograph of rubber and plastic step-wedges inside coolant tube sample

Figure 2 - Sketch of test piece showing placement of rubber and plastic step-wedges in coolant tubes

Greatest sensitivity with this method was obtained with Freon. An aqueous solution of cadmium chloride gave good results but, even as a saturated solution, does not offer the radiographic density of Freon 114-B2, which has a specific gravity of 2.2 and contains the heavy element bromine. Other heavy-metal-salt aqueous solutions were tried but were disregarded because of inferior results or contamination problems. ASL⁽²⁾, although sufficiently dense, has a chemistry that is incompatible with the materials of the nuclear rocket.

A special Freon formulation was selected as the most suitable high-density fluid for negative radiography of coolant-channel systems. Radiographic results and previous Aerojet-General Corporation research on its metallurgical compatibility supported this decision.⁽³⁾ Additionally, this solvent tends to clean rather than contaminate a system and leaves no residual solids.

METHOD USED

Tube Blockage Samples: Three calibrated step wedges were made. Two consisted of three 0.100 in. thick layers of rubber, and one of five 0.065 in. thick layers of cellulose-acetate plastic.

Simulated Nozzle Wall Sample: A sample was constructed containing five service-weight tubes of Hastelloy X attached to 0.500-in. Hastelloy X plate.

Tube Filling: Rubber and plastic samples were inserted in the tubes as illustrated in Figure 2. One end of the tubes was sealed with rubber and Duco cement. The tubes were filled with Freon 114-B2, cadmium-chloride aqueous solution, or air, as shown in Figure 2, and then sealed to enclose the fluids.

X-ray employed Eastman Type-M film with lead intensifying screens, 36-in. focal film distance, 250 kv, 8 ma, 1-1/2-min exposure.

METALLURGICAL CONSIDERATIONS

Corrosion due to fluid filling of dehydrated Hastelloy X or 347 stainless-steel tubes is not a problem if Freon 114-B2 is used. This fluorocarbon is dibromotetrafluoroethane with a formula of $\text{CBrF}_2 - \text{CBrF}_2$. It is used by Aerojet in quantity for rocket fluid-injection vector-control systems and is chemically controlled for cleanliness and metallurgical compatibility⁽⁴⁾ as follows:

Chemical and physical properties - The material must be in accordance with the chemical and physical properties listed in Tables I and II.

TABLE I

<u>Property</u>	<u>Requirement</u>	<u>AQL, %</u>	
		<u>LSL</u>	<u>USL</u>
(a) Material purity	99.8 weight percent (min) symmetrical dibromotetrafluoroethane	2.5	--
(b) Free bromine	None allowed (not detectable)	--	4.0
(c) Water content	20 parts per million (max) by weight	--	1.5
(d) Particulate contamination	Table II	--	6.5
(e) Acidity	0.1 part per million (max) by weight when expressed as hydrochloric acid	--	4.0
(f) Halides	No visual evidence when tested with silver-nitrate and thorium-nitrate solutions	--	4.0
(g) High-boiling	0.01 percent by volume (max)	--	4.0

TABLE II

Particle Contamination Limits per 100 ± Milliliters

<u>Particle Size</u> <u>(Microns)</u>	<u>Maximum Number of Particles</u> <u>- plus Fibers</u>
(a) 5 through 25	47,500
(b) 25 through 50	2,500
(c) 50 through 100	125
(d) 100 through 350	15
(e) Over 350	Less than 1

The above controls were established on the basis of a study conducted to determine the compatibility of various materials of construction with Freon 114-B2. Its use in dehydrated Hastelloy X and 347 stainless-steel is free from corrosion danger. Specimens of 347 stainless-steel, after 90 days of exposure in Freon 114-B2 at room temperature, showed no visible corrosion and no weight loss.(3) Maximum exposure to the fluid in the event of its use for this X-ray procedure would not exceed two days and would be at room temperature.

It is advisable to remove all water from tubes before filling with Freon because excess water can cause the Freon to react with metals. Dehydration of nozzles prior to X-ray is standard procedure, and this requirement does not create an additional operation.

APPLICABILITY CONSIDERATIONS FOR NEGATIVE RADIOGRAPHY

Negative radiography can be applied in the average X-ray facility, with the requisitioning of Freon being the only additional need. It can be performed on an entire channel system or on individual tubes. The Freon can be reused. Negative radiography is simpler and cheaper to use than neutron radiography. In addition, it obviates neutron radiography in those cases where the blockage is sufficient in size and low enough in density so that the detail resolution of neutron radiography is not called for. It can also reveal materials such as Teflon that can be overlooked with both X-ray and neutron radiography because fluorocarbons do not readily absorb either X-rays or neutrons.

II. NEUTRON RADIOGRAPHY

DEFINITION; NEUTRON RADIOGRAPHY

Neutron radiography is a means of detecting materials inside solid objects in a manner similar to that used in X-ray. While X-ray provides shadowgraphs of dense materials, neutrons penetrate the majority of dense materials and make shadowgraphs of most low-density substances. The contrast and resolution of hydrocarbon-structured materials with this technique are effectively the equivalent of those obtained for metallic inclusions in metallic structures by X-ray techniques. Thorough exposition of this method is presented by H. Berger in "Neutron Radiography".⁽⁵⁾

TEST RESULTS

Figure 3 shows the materials used and comparisons of film images by both neutron and X-ray methods. Figure 4 shows the degree of resolution obtainable; the image of 0.009-in. nylon dental floss is clearly shown in the original film through 1/2-in. Hastelloy X alloy.

General test conclusions, based on the demonstrated results, are as follows:

- (1) Neutron radiography shows small pieces of hydrogen-containing materials, such as plastic and paper, that cannot be detected by neutron radiography,
- (2) X-ray shows small metallic materials that cannot be detected by neutron radiography,
- (3) A combination of the two methods can show all of the materials listed if the total amount of the material is large enough to cause tube blockage, and
- (4) small pieces of some materials, such as Teflon and Kel-F, may be missed by both methods.

Figure 3 - Positive Prints of Neutron Radiograph (left) and X-ray (right) for comparison

Figure 4 - Positive print of sharp image of Neutron Radiograph showing 0.009-in. thick nylon dental floss through 1/2-in. thick nozzle wall (Hastelloy-X)

METHOD USED

Neutron Sources: Two separate reactors were used, stationary installations that emitted thermal neutrons at approximately 10^7 n/cm²/sec on the subject.

1. 30-kw General Electric Vallecitos Neutron Test Reactor, used small port and 20-ft distance with collimating tube, direct to core.(6)
2. 250-kw Aerojet-General Nucleonics Industrial Reactor, Pool Type, used 6-in. air filled tube through water to side of reactor. This reflects neutrons upward, thermalizes them, and collimates the beam in a 23-ft distance.(7)

Film Imaging: Kodak hard-cassette standard X-ray film was used, containing gadolinium metal screens that contact film to absorb neutrons and convert beam to gamma and beta emission, which exposes film. Also, indium screens were used, which, with subsequent film contact, transfers the image by residual radioactivity decay in the indium.

Film Processing: Standard X-ray film processing method.

Time: Setup time equivalent to that for X-ray; exposure was 45 and 30 min. Slight addition of time involved to bring reactor up to full power and to monitor radioactivity of part after exposure.

Residual Radioactivity: Because of nuclear activation resulting from neutron bombardment, a count just after exposure indicated a surface gamma radiation of less than 2 mr/hr. A count 22 hours later showed a barely detectable residual effect. The maximum level permitted for radiographic personnel is 2 mr/hr. This amount of nuclear activity is not usual, but in this case an uncommon alloy element caused residual radioactivity that approached a level requiring temporary control.

APPLICABILITY CONSIDERATIONS FOR NEUTRON RADIOGRAPHY

When a tube flow test shows blockage and neither X-ray nor negative radiography can provide an image of the foreign material, the neutron radiograph is valuable. Aerojet has a neutron-radiographic capability and has the General Electric facility nearby. Such facilities are not common, and geographic availability for industrial use presently is limited. Those laboratories currently doing neutron radiography are soliciting business and generally have some open time. Because of the cost, usually upward of \$150 for individual 14 by 17 films, neutron radiography is used only in those cases where it is specifically needed.

III. LIQUID-CRYSTAL INSPECTION

DEFINITION; LIQUID-CRYSTAL INSPECTION

Liquid-crystal nondestructive testing is a means of detecting small temperature changes on the surface of a material. This technique can reveal the location of hot spots in assemblies, a lag in temperature change due to blockage in coolant tubes, (8) or the movement of changing temperature fronts.

Liquid crystals now available are cloudy, white, grease-like cholesterol materials. When thinly coated on a blackened surface, this material exhibits vivid color reflections from incident white light as the surface passes through a predetermined narrow temperature range. These cholesterols exhibit the optical properties of a crystal by scattering light selectively in birefringent color patterns.⁽⁹⁾

The color patterns that result from this optical phenomenon are specific to certain temperatures. The phenomenon therefore, provides a means of detecting dynamic or progressive temperature changes taking place within objects or assemblies that are coated with liquid crystal. The process is reversible and repeatable. The color pattern can be held when temperature does not change or can be repeated dynamically for study of the change as many times as the observer wishes to cycle the temperature.

METHOD USED

Liquid Crystal: Figure 5 illustrates the test piece. The simple test procedure is as follows:

1. Coat tube outer surface black with India ink.
2. Brush or spray even coat of liquid crystal on black surface.
3. Illuminate surface with white light.
4. Open hot-water flow and observe surface as color changes, noting areas of time lag in color change for correlation with tube blockage.

The U-tube sample panel is one of the test pieces used for neutron-radiographic, negative-radiographic, and ultrasonics tests. Comparisons between these methods are therefore valid in terms of equivalency of material conditions.

Temperature was controlled by connecting the manifold, attached to the test piece, to the hot and cold water lines in a sink in the NDT laboratory. The faucet valves of the sink were opened and closed to cycle the temperature and to cause the color change in the liquid crystal on the tube surfaces. This setup permitted quick repeatability of the test, and the part was cycled many times under each of the various blockage conditions to establish the validity of the conclusions.

The test was recorded with 16-mm color motion pictures at 24 frames per second with 3200 K light. The film has been retained for projection to show the dynamic color change in relation to the various blockage conditions.

APPLICABILITY CONSIDERATIONS FOR LIQUID CRYSTAL

Liquid-crystal inspection is only of minor value for finding tube blockages in the case of large rocket nozzles. It should be considered as an inexpensive alternative technique if negative radiography and neutron radiography are not available. Also, if the other methods are ineffective for some reason, the liquid-crystal technique provides a backup method.

Absence of tube blockage should be determined first by individual tube flow tests. This testing is considered a functional nondestructive proof test.

Figure 5 - Liquid crystal test piece showing placement of partial tube blockage, water flow system to change temperature, and area of blockage indication observed as color change delay

If blockage is found to exist, the liquid-crystal may be applied to the specific tube to locate the trouble by temperature-cycling the water and observing the color-change delay; but this approach will not be effective if the blockage near the tube surface is not large. Standard X-ray is still considered of first importance in defining blockages discovered by flow test. If the blockage is too low in density to be detected by X-ray, negative radiography should be the next backup technique. No problem should exist after exploiting these inspection methods, but neutron-radiography and liquid-crystal techniques are available to support the other procedures if an unanticipated blockage problem arises. As an inspection method, the liquid-crystal technique offers great simplicity. It is a means of obtaining infrared effects without complicated equipment and, in some cases, provides additional information. By choice of the cholesterol compound, these effects can be studied at temperatures as low as -20°C and up to 350°C . The narrowness of the temperature band through which the color change occurs can be 3°C to 50°C .⁽⁹⁾ This range makes the method sensitive to minute heat-transfer effects and provides a means of studying such effects over an entire surface.

When contrasted with the spotty information obtained by thermocouples, liquid crystals offer a broad and promising potential to design and stress engineers. If a given total surface is first studied with liquid crystal at near-ambient temperatures to determine the pattern of heat transfer within the working range of the liquid crystals, spot thermocouple information, obtained at functional temperatures, can then be knowledgeably related to other points on the surface. Also, because heat transfer can be observed in motion and relative to time, it is possible to observe points of heat concentration relative to thermal shock. As a reliability tool, the liquid-crystal technique suggests a means of proving efficiency of design as related to thermal change.

IV. ULTRASONIC C-SCAN

DEFINITION; ULTRASONIC C-SCAN

As applied to this investigation, a focused high-frequency sound beam is generated by an ultrasonic transducer in water. The beam passes through the tube wall and through the water with which the tubes are filled, reflects from the nozzle wall at the back side of the tube, and is partially returned to the transducer. That portion of the sound beam returning from the nozzle wall is selected by electronic gating for X-Y recording on paper. When tube blockage prevents transmission of the beam to the nozzle wall, no return signal is recorded. In theory, this should provide a map of any included blockage.

TEST RESULTS

Figure 6 shows one of several recordings made by this method. Although the theoretical result is obtained, the quality is very inferior compared with that for neutron and negative radiography.

Better recordings could be obtained by careful refinement of equipment and procedure. However, this poor result was considered sufficient evidence that further refinement was not justified for this investigation.

1/4"
RUBBER
ROD

1/4"
BRASS
ROD

Figure 6 - Ultrasonic Pulse-Echo C-Scan Recording

METHOD USED

The five-tube test panel was leveled in the immersion tank and scanned at frequencies ranging from 2-1/4 to 10 MHz. A 3-1/2-in. focused-point transducer was used for the recording shown in Figure 6. The ultrasonic assembly used was the Immerscope 424D, an automation tank with bridge-mounted search unit, and an Alden recorder.

APPLICABILITY CONSIDERATIONS FOR ULTRASONICS

Ultrasonics should not be used to find small inclusions in this type of tubing which can have irregularities in the tube assembly. Scattering of the sound beam due to small irregularities results in irrelevant signals, and tube curvature causes failure of the beam to scan the entire internal space with equal power. This decision applies to ultrasonics for this use only and is not intended to depreciate ultrasonic inspection, which is often the ideal NDT method under more favorable circumstances.

CONCLUSIONS

The coolant-tube system of a rocket-engine nozzle must be free of any actual or potential blockage.

Enclosed systems such as those for coolant tubes can be effectively inspected for absence of all foreign material large enough to be seen on film as follows. Functional flow tests should be performed first, and then X-ray. If flow tests show that blockage exists and the responsible foreign materials are too low in density to be detected by X-ray through a heavy metal nozzle wall, negative radiography is a highly effective, easy, and low-cost alternative. If a flow test still indicates undetected blockage, neutron radiography is advised, if economics permit. High radiographic sensitivity is obtainable with neutron radiography, and this type of service is now commercially available. The above methods provide thorough inspection for blockage-size foreign materials.

The liquid-crystal inspection method can also be applied, to provide a functional test to determine the cooling efficiency of accessible coolant-system surfaces. Ultrasonic techniques are generally ineffective and unreliable, for the particular application discussed herein.

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