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Nondestructive Testing of Materials

Involved in the B-77 Roll Control Program:

June 1, 1976 - December 15, 1976

Walter A. Dudley

February 28, 1977

Monsanto

MOUND LABORATORY

Miamisburg, Ohio

operated by

MONSANTO RESEARCH CORPORATION

a subsidiary of Monsanto Company

for the

**UNITED STATES ENERGY RESEARCH
AND DEVELOPMENT ADMINISTRATION**

U. S. Government Contract No. EY-76-C-04-0053

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I. INTRODUCTION

This report covers all nondestructive development effort on the Roll Control Program for the period June 1, 1976 through December 15, 1976. Information previously reported will be re-presented here and appropriately updated.

II. AGING AND SHELF LIFE PREDICTION SAMPLES

A. Effort and Results to Date

Initial radiographic and visual inspections of the Thiokol-fabricated tensile, butt tensile, and chemical reactivity samples were completed on schedule. The samples consist of solid propellant and composites of propellant and inhibitor that are currently being examined in the accelerated aging and shelf life prediction studies of materials involved in the B-77 Roll Control Program. The initial NDT performed on these samples shows them to be free of defects. No fabricated radiographic standards and/or penetrameters are yet available for these samples. However, light-section-microscopy measurements of nonuniform saw cuts, which had been witnessed visually and radiographically for several tensile samples, indicate the present radiographic technique to have a film contrast sensitivity equivalent to or better than a 2T (2%) penetrameter. Because the physical sizes of the remaining aging study samples are similar to those of the tensile samples, their respective film sensitivities are also considered to be similar.

B. Planned Future Effort

Aging study samples will again be inspected after aging but prior to any destructive stress-strain analysis for comparison. Radiographic inspections of aging study samples will eventually incorporate the use of radiographic standards. These standards will be used to quantify radiographic sensitivities for

detection of specific flaws relative to aging study sample type. The standards are to be made from mock propellant* and will contain flaws simulating voids, surface cracks, and disbonding. Sketches of the standards are shown in Figures 1 through 3. Fabrication of these standards, at present, is still being negotiated.¹ Currently, Mound is waiting for a fabrication cost response from Thiokol. Sizes of the simulated flaws shown in Figures 1 through 3 were chosen relative to Thiokol's recent response concerning its best estimate of the critical sizes of flaws permissible with a product item.² The reason for using standards rather than penetrameters in future radiographic inspections is that the standards for this application are easier to fabricate and should provide more meaningful information about flaw detection. Repeat future visual inspections for the aging study samples will again be with no optical magnification unless considered warranted or specified.

III. LITERATURE SURVEY

A. Effort and Results to Date

A computerized literature survey was completed on the NDT of solid propellants and related materials. Results of this survey indicate that ultrasonic inspection is potentially the best NDT method for detecting aging cracks in bulk propellant and delaminations in adhesive bonds. In addition, Mr. Dahlke, SLL, supplied some NDT report references not acquired via the computerized survey. Some of these additionally supplied references were reviewed for applicability and will be commented upon later in this report.

B. Planned Future Effort

No further extensive literature surveys are planned at this time. However, the current literature will be read for recent developments.

*Mock (inert) propellant uses the same fuel as live propellant. However, with the mock material, ammonium sulfate is substituted for ammonium-perchlorate (AP) oxidizer to effect a material which is nonpyrophoric. The nearly equivalent densities plus particulate sizes well below 1/16 in. for the two formulations

permit the mock material to be used as a substitute material for fabrication of radiographic standards. In addition to the handling benefits of its nonpyrophoric nature, the inert material (according to Thiokol) should cure and age similarly to live propellant.

1 Unit Required

FIGURE 1 - Dog bone tensile specimen dimension equivalent to items previously received by Mound.

1 Assembly Required

FIGURE 2 - "Butt Tensile Specimen." Specimen criteria same as for Figure 1.

1 Assembly Required

FIGURE 3 - Mound "Chemical Reactivity Testing Sample."

IV. ULTRASONIC EVALUATION

A. Effort and Results to Date

Ultrasonic parameters of immediate interest for live (Thiokol Type HP-3314) propellant and related Roll Control material were determined by early November, 1976. The pertinent ultrasonic data obtained for the initial investigative effort are shown in Tables 1 and 2 and in Figures 4 and 5. The technique used to obtain the data in Figures 4 and 5 was previously presented³ and is discussed elsewhere.⁴ The data of Figures 4 and 5 show that the propellant material is too attentive to transmission of acoustic energy to permit commercial ultrasonics to be employed at the more commonly used test frequencies of 1 MHz and above. Thus, ultrasonic inspection of propellant must be performed at test frequencies below 1 MHz, and as a result of the lower test frequency, the inspection will be limited to a flaw detection sensitivity nearly equivalent to the test frequency. The data presented in Table 1 were obtained from literature sources or determined at Mound by other commonly used ultrasonic techniques. Data and conclusions listed in Table 2 were determined by a spectrum analyzer and/or time-domain measurements of the resultant dominant frequency of ultrasonic pulses after being transmitted through a bonded composite of 1.8 in. of propellant and 1/8 in. of plastic. The

ultrasonic pulses were generated by a borrowed commercial ultrasonic analyzer and the transducers used previously in the attenuation measurements. A video outlet on the analyzer permitted external connection of the analyzer to the auxiliary measuring equipment. Measurements were made for both Pulse-Echo (PE) and Through-Transmission (T-T) modes of permissible ultrasonic operating technique. An illustration of basic PE and T-T measurement technique is shown in Figure 6. Demonstration of the estimated flaw size detectability listed in Table 2 is impossible at this time since it would require permanent availability of an appropriate ultrasonic analyzer, focused transducers, and flaw standards. The only commercial ultrasonic analyzers known to have operating parameters suitable for inspection of Roll Control Propellant* are:

1. Automation Industries, Model 775 Reflectoscope video display or equivalent, featuring a Model 5N-UR pulser receiver module.

*Mr. Dahlke inquired about the possibility of SLL loaning Mound one of the listed ultrasonic systems. The outcome of Mr. Dahlke's effort was that SLL does not have any of the systems listed.

Table 1
ACOUSTIC PROPERTIES OF GAS GENERATOR MATERIALS

<u>MATERIAL</u>	<u>DENSITY</u> <u>(g/cm³)</u>	<u>V₁</u> <u>[(cm/sec)x10⁵]</u>	<u>IMPEDANCE</u> <u>[g/(cm · sec)x10⁵]</u>	<u>REFLECTION</u> <u>COEFFICIENT (%)</u>
Titanium	4.50	6.07	27.3	89.6%
Propellant	1.51	1.745 (20.4°C)	2.63	27.7%
ABS	1.016	1.905 (25°C)	1.93	11.9%
Water (21.6°C)	1.0	1.487	1.49	--

Table 2
ULTRASONIC APPLICABILITY TO INTERROGATION OF PROPELLANT

	<u>APPROACH</u>	<u>INTEG. FREQ.</u>	<u>MIN. EST. FLAW</u> <u>SIZE DETECTABILITY</u>
Propellant	T-T (Yes)	≈.7 MHz	0.1
	P-E (Possibly)	≈.7 MHz	0.1
ABS/Propellant	T-T (Yes)	≈.7 MHz	0.1
	P-E (Yes)	0.4 to .5 MHz	0.175
Case/ABS/Propellant	T-T (No)	--	--
	P-E (No)	--	--

FIGURE 4 - Relative attenuation of sound energy in TP-H-3314B propellant.

FIGURE 5 - Attenuation coefficient as a function of frequency for TP-H-3314B propellant.

Through Transmission
Transmitting and Receiving
Transducers using Longi-
tudinal Wave Mode

Pulse Echo
Transmit-Receiving Trans-
ducer using Longitudinal
Wave Mode

FIGURE 6 - Illustration of P. E. and T-T ultrasonic technique.

2. Tektran Immerscope II video display, featuring a Model PR-17 pulser receiver module.
3. Zenotec components, featuring selected ultrasonic modules adaptable to series TM-500 Tektronix oscilloscopes.
4. Panametric Ultrasonic Modules, featuring a Model 5050AE-160B pre-amplifier, either of the Model 5000 series pulser receivers, and an appropriate auxiliary oscilloscope.

The potential of the first two analyzers has been demonstrated through vendor-loaned equipment. Potential of the latter two has been verified verbally by investigators of similar materials. The first three would require an investment on the average of \$8,000. The cost of the fourth, Panametric, is the same except that Mound presently has all the system except the preamplifier. Acquisition of the Panametric preamplifier component would be the least expensive way to develop ultrasonic inspection techniques for propellant. It should be emphasized that Mound presently has the ultrasonics necessary to develop an inspection

technique for examination of the ABS-to-propellant bonds. From preliminary investigation of these bonds at a test frequency of 5 MHz and higher, it is conjectured that bond separation will not be differentiable from a condition of poor bond. Samples used for the preliminary examination of bonds consisted of disks of live propellant bonded on one face with nominal 0.090 in. of plastic.

To accommodate planned future ultrasonic development and radiographic and visual technique development as well, a single standard containing simulated flaw types of current interest has been designed. The standard's design, a half-length propellant cartridge, is shown in Figures 7 through 11. As with the aging study standards, fabrication and cost of the standard shown in Figure 7 are still in the negotiating stage. Recently obtained ultrasonic data for inert propellant (Figure 12) show that the inert material is acoustically comparable to live propellant. The immediate benefit of this comparability is that all phases of future NDT development can be pursued without initial consideration of providing a necessary safe environment for handling pyrophoric

FIGURE 7 - Disassembled view of "half" length RC propellant cartridge showing induced flaw locations.

FIGURE 8 - Specifications (Figure 7).

material. Thiokol, therefore, will be informed that the half-length standards also are to be fabricated using inert material and to reflect this directive in its cost quotation. Applying developed NDT techniques to aged product at Mound requires compliance to DOD and ERDA safety requirements for handling Class B type explosive materials. At this time, proposed NDT on aged product is still being reviewed by the Mound Safety Section; a decision is anticipated by mid-January, 1977.

Concurrent with the ultrasonic development, a method to permit practical ultrasonic inspection is being pursued. Direct immersion of the propellant is impossible since all commonly used liquid couplants would definitely leach the propellant's AP oxidizer and would probably cause the fuel itself to swell. One solution is to find a compatible, possibly strippable,

material that could be applied to the propellant as a thin hermetic seal. A candidate material is high-purity wax. Two samples of low melting, high-purity wax were sent to Dr. Brundage of Thiokol for DTA analysis. Thiokol's data for the performed DTA analysis of the submitted waxes are shown in Table 3. The data indicate that both waxes are chemically compatible with Roll Control propellant. Thiokol will, for \$3000, determine the effect of ignition of a wax coating on Roll Control propellant by multiple comparative testing of coated and noncoated propellant samples by Arc Image analysis. However, Mound will proceed with other simpler analyses such as controlled dip coating of a sample piece of inert propellant followed by destructive examination for penetration of the wax on both cast and machined surfaces of the sample.

FIGURE 9 - Segment A (Figure 7).

B. Planned Future Effort

Planned future ultrasonic effort is to develop techniques to interrogate aged product items for detection of bond separation between ABS and propellant and propellant inner-bore radial cracks. Goals of the planned effort are to demonstrate developed techniques by July 1, 1977. Anticipated sensitivities of the ultrasonic techniques considered for development are by estimation expected to be 0.1 in.² or better. However, initial development effort will be to successfully demonstrate detection of the flaw sizes specified for the half-length scale standard. Sketches

of the ultrasonic approaches presently considered for development are illustrated in Figures 13 and 14. The approach in Figure 14 is similar to an acoustical-holographic approach described in Reference 5. Development of either ultrasonic technique, however, will necessitate concurrent development of a practical method to seal the propellant from the required liquid couplant. Possible sealing methods to be investigated including the previously discussed wax approach are listed below. The methods are listed in order of resultant inspection fixturing complexity.

1. Continued investigation of wax as a suitable hermetic sealant.

FIGURE 10 - Segment B (Figure).

2. Development of an ultrasonic inspection fixture which permits end-sealing of the propellant cartridge.
3. Procurement of transducers which would house a couplant and track under applied tension along the outside diameter surface of the cartridge.

Of the three methods presented, the first would not necessarily be the least costly to pursue. For the second, a fixture design has been proposed. Fabrication of a workable prototype fixture for the second method is estimated at minimum to cost \$7,500. For the third method, no cost inquiry from commercial transducer manufacturers has been sought. However, such units as described would definitely not be off-the-shelf items. For adoption of approaches 1 or 2, the cost of transducers would fall within conventional

price ranges. Another ultrasonic approach called Acoustical Profilography⁶ was suggested by Mr. Dahlke, SLL. This approach will be reviewed for possible consideration as a better method for detection of propellant inner bore radial cracks.

V. RADIOGRAPHIC DEVELOPMENT

A. Effort and Results to Date

The radiation field of the 150 keV x-ray machine was determined. The measured radiation field strength at a distance of 48 in. from the x-ray tube is shown in Table 4. Also given in Table 4 is the value of the radiation strength of the B-77 device as known to date. It is determined from Table 4 that the total radiation dose that a propellant grain would be exposed to during its anticipated lifetime from both the B-77 device and

Flaw	Length	Width	Depth
A	0.100"	0.010"	0.100"
B	0.100"	0.010"	0.100"
C	0.100"	0.010"	0.100"
D	0.050"	0.010"	0.050"

FIGURE 11 - Segment C (Figure 7).

NDT radiography will be much lower than 1 MRad. The latter value is reported in the literature as an upper limit for neglecting radiation damage in propellants.⁷ In addition to the radiation field measurements, capability of the 150 keV x-ray device to adequately perform on product grains was demonstrated as follows: A 2T (2%) penetrameter relative to cross-sectional thickness (3.6 in.) of a product grain was machined from a flat disk sample of live propellant. The penetrameter was then placed on top of one of two pieces of rectangular live propellant both cut to grain wall

thickness, and separated from each other by a distance equivalent to product grain inner-bore diameter of 2.2 in. This assembly was then radiographed. The resultant film contrast and resolution were physically demonstrated at the November 9, 1976, meeting at Mound. The demonstration definitely shows that radiography has a proven capability to easily detect a 1/16 in. diameter void.

B. Planned Future Effort

Future radiographic effort will be to develop techniques to interrogate

FIGURE 12 - Acoustical data for inert propellant.

Table 3

COMPATABILITY^a OF PENTOLITE WAXES WITH TP-H-3314B
 PROPELLANT (THIOL DATA GENERATED AT REQUEST OF MOUND)

	INITIAL EXOTHERM (°C)	AP ENDOTHERM (°C)	SELF- HEAT (°C)	IGNITION (°C)
TP-H-3314B	209	234	240	249
TP-H-3314B + "Ultraflex" ^b	209	237	243	257
TP-H-3314B + "Victory" ^b	211	236	241	252

^a Compatibility tests were run on a 1:1 basis with TP-H-3314B and each of the two Pentolite waxes "Ultraflex" and "Victory", using differential thermal analysis (DTA) at a 10°C/min scan rate. The results indicate that there is no readily apparent compatibility problem.

^b Melting point of waxes as given in Petrolite Corporation's Product Data Release No. 400 are as follows: "Victory", 79.4°C; "Ultraflex", 62.2°C.

FIGURE 13 - Ultrasonic scan detection of ABS to propellant bond separation.

FIGURE 14 - Ultrasonic scan detection of inner-bore radial cracks.

Table 4

RADIATION^a DOSE RATE MEASUREMENTS FOR
MOUND NORELCO MODEL MG-150 X-RAY UNIT (S/N 302)

<u>kV SETTING</u>	<u>CURRENT (mA)</u>	<u>MEASURED RADIATION AT 48 IN. FROM X-RAY TUBE^b (rem/min)</u>
25	10	10.0
30	10	15.0
50	10	32.0
65	10	42.0
70	10	45.0
75	10	47.0
150	10	70.0

^a Value of radiation field of B-77 device known to date is as follows: Gamma plus neutron at 1 m from the device over a 25-yr period is 25.7 Rads.

^b Victorian Condenser R-Meter (Chamber #131) used for measurements.

production and aged product for detection of bond separation between ABS and propellant, and for voids and cracks in the propellant grain. Goals of planned future effort are to demonstrate defect detection sensitivity relative to using the half-scale length standard and conventional x-ray methods by July 1, 1977.

VI. VISUAL INSPECTION

A. Effort and Results to Date

No development effort has been pursued during the present reporting period. Visual examination to date has strictly been "by eye" and employed only on the aging study samples as discussed in Section II of this report.

B. Planned Future Effort

Future visual inspection development effort is to employ a Boroscope to aid in detection of microcracking at the inner bore of production and aged product. Goals are to determine and demonstrate sensitivity of this technique

relative to detecting the simulated inner-bore cracks requested for the half-length scale standard and for natural aging cracks if occurring.

VII. OTHER NDT METHODS

A. Effort and Results to Date

Two other NDT methods were investigated in the literature. The first method is that of microwaves for which several source references are listed.^{8,9,10} This technique is particularly applicable to nonconductors, such as propellant, and in addition requires no couplant as does the ultrasonic approach. An approach method of the technique described in Reference 10 for detection of surface cracks could possibly be made adaptable to the Roll Control product grain. At present, Mound has no microwave capability. The second method, suggested as a possibility by Mr. Dahlke, SLL, is that of "Electrified Particles" and is fully described elsewhere.¹¹ This NDT method is very sensitive to detecting cracks of very small width (below light reflection

detectability; e.g., cracks with a width of 4 millionths of an inch or less). However, employment of this technique to propellant would require using a liquid penetrant which is out of the question.

B. Future Effort

No future effort is intended except keeping current on microwave capability as presented in the available literature.

VIII. COMMENTS

The actual need for planned ultrasonic inspection of aged product is at best marginal. This statement reflects Thiokol's recent response to Mound's technical request to be supplied with its current best estimate of the critical sizes of flaws associated with Roll Control product.² For example, as concluded from the Thiokol response, bond separation could exist at a delamination tightness below radiographic detectability but not cause any problem with product performance. If this conclusion is true, why then develop an ultrasonic technique to detect bond separation when the

ultrasonic approach in this application is known to be more sensitive than radiography in detecting delamination tightness? Thiokol does not state in its response the minimum area and delamination tightness it can detect radiographically for bond separation defects. The latter detectability is assumed to be not much better than 5 mils. In addition, it is expected that, at anticipated test frequency of 5 MHz or higher, the ultrasonic area detectability of bond separation will be equivalent to or better than that achieved with radiography. A similarly concluded situation exists for planned ultrasonic development to detect inner-bore radial cracks. However, the forced ultrasonic test frequency of 1 MHz or lower in this particular inspection application reverses the order of technique sensitivities in terms of area detectability. At present, ultrasonic area sensitivity in this application is estimated to be 0.1 in.² or better. It is not expected that minimum ultrasonic sensitivity will be equivalent to Thiokol's indicated radiographic area detectability of 0.05 in.². However, the ultrasonic technique in this application would detect an inner-bore crack of much less width than 10 mils.

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