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SUBJECT: INSPECTING AND
TESTING LEAD LININGS REPRINT

Inspecting and testing lead linings, particularly for chemical construction, is a subject that has been attracting much interest. The attached reprint of a paper presented before the Society for Nondestructive Testing at their annual convention in Philadelphia, October 1955, was published in the January-February 1956 issue of NONDESTRUCTIVE TESTING. Specific methods of testing and inspecting sheet lead linings and bonded or lead clad linings are detailed. Procedures to be followed in the acid wash, fluorescent penetrant and dye penetrant methods are presented. The application of ultrasonic testing is discussed, along with the special possibilities of radiography for brick lead lined vessels.

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Yours very truly,

Robert L. Ziegfeld

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Secretary

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Inspecting And Testing Lead Linings*

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Methods of testing and inspecting sheet-lead linings and bonded or lead-clad linings used in the chemical-process industries are described. The procedures to be followed in the fluorescent-penetrant and the dye-penetrant methods are presented. The application of ultrasonics is discussed. The special possibilities of radiography for brick-lined vessels are noted.

More rigorous operating conditions, higher pressures and temperatures, and higher corrosive concentrations are acknowledged trends in the chemical-process industries. All are exerting their influence on the choice of materials, as well as methods of construction, and have helped focus attention on the need for more exacting inspection and testing techniques for processing equipment.

Lead is a common material of chemical construction and has been no exception to these trends. As a result, a number of methods have been developed for inspecting and testing sheet-lead linings and bonded or lead-clad linings.

The methods presented here are actually being used, in some cases by the fabricators of the equipment and in others by the users.

Lead-protected chemical-processing vessels are normally inspected for the presence of pinholes or porosity in welded seams on sheet-lead linings, hair-line cracks, either in the weld or the sheet, resulting from fatigue or stress corrosion, and pinholes, inclusions, or bond discontinuities in the case of clad vessels.

Defects falling into any of the above classifications, although they may not penetrate the lining completely, do form a potential trouble spot that may shorten the lining's expected life—a problem that can be minimized through the use of proper inspection and testing.

TESTING PROCEDURES FOR SHEET-LEAD LININGS

Sheet-lead-lined vessels generally consist of a steel, wood, or concrete vessel or shell lined internally with sheet lead, the thickness depending on the type and conditions of service. Lead less than 1/8 in. (8-lb) is rarely used in handling corrosives. The lead is supported at the top by lapping over a flange and on the inside by sheet-lead-covered steel straps bolted through the lining to the outside, the presence and number of straps depending on the size of the vessel. Joints in the lead are welded by a lead *burner* using a fine-flamed oxy-hydrogen or -acetylene torch, no flux, and no dissimilar metals. The lead is merely melted and flowed together, with any additional metal supplied by the *burner* from a bar of lead of the same composition.

After the completion of a new installation or extensive repairs on an old lining, the seams are carefully checked visually by the lead *burner* and all obvious defects corrected. If it is necessary to check the soundness of a lead weld, such as on butt joints, in order to determine the depth of fusion, a section of the seam can be trepanned with a trepanning drill to obtain a cross section. On a bonded lining, this technique has also been used to check the nature of the bond and the thickness of the lining. In either case, however, the test is destructive, whereas radiographic techniques might provide a more effective and, at the same time, nondestructive test for weld soundness. More will be said about this later.

For closer and more exacting examination of lead welds, a fluorescent or dye penetrant may be used. This method enables the inspector to examine large areas more rapidly and accurately than is otherwise possible.

The penetrant method is based on the fact that tiny cracks or pores will absorb certain chemicals by capillary action. After the excess is cleaned off, the chemical remaining in the imperfections can be *developed* or brought to the surface, where it may readily be identified, either by a bright glow under ultraviolet light, in the case of the fluorescent penetrant, or by a distinctive color, in the case of the dye penetrant.

Interpretation of each indication is all important. Practically all procedures involving the fabrication of lead result in the possibility of confusion between a defect and a simple surface marking; therefore, it must be realized that a high degree of discernment must exist to differentiate between a true defect and a harmless surface irregularity.

Fluorescent-Penetrant Method

PREPARATION OF SURFACE. The preparation of lead for inspection using fluorescent penetrants is of prime importance. One successful method is to sandblast the surface to remove surface films and ragged edges. The sandblasting nozzle should be held at a constant angle to the lead in order to prevent calking action by the blast. This may close certain defects that would otherwise appear during inspection. It is not necessary to sandblast a new lead lining, but thorough cleaning is still necessary. Oil and grease films must be removed, usually by washing with a grease solvent, followed by a warm soapy-water wash. The last cleaning step is thorough rinsing with clean water. The reason for such particular care is that oils, greases, soaps, etc., are inherently fluorescent and, when removal is incomplete, false indications may show up during the later stages of inspection.

APPLICATION OF PENETRANT AND SOAKING. A generous amount of penetrant should be applied to the lead lining, either by brush or by spray. More penetrant is needed with lead than with other metals, however, the soaking time is much shorter. At

*Presented before the Society's 15th Annual Convention, Hotel Sylvania, Philadelphia, October 17-20, 1955

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normal temperatures this will be from 12 to 20 min

The removal of the penetrant from the surface is the most critical step. It should be removed quickly, since it will become hardened and more difficult to remove if allowed to soak beyond the 20 min. The two methods of removal most used are sandblasting and water washing with either cold or hot water, the latter being preferred, especially in the case of large vessels. The problem with either method is that just enough sand or water to remove the excess penetrant is needed, and any amount beyond that may reduce the sensitivity of the final inspection, especially if hot water or steam is used. For example, it is very likely that all of the penetrant may be removed from larger defects when washing is done in excess.

DRYING AND DEVELOPING. If sandblasting is used, a developing powder is not required, since sand leaves a fine powdery film on the lead. When water washing is used, the lead must be allowed to air dry before application of a developing powder. The powder acts as a blotting material, drawing the penetrating oil from the defect to the surface, where it can be observed.

INTERPRETATION. The interpretation of results is largely dependent on the experience and judgment of the inspector and is the most important single criterion of good inspection. Training material, schools, and on-the-job training are available. Some defects are readily distinguished, whereas other indications are difficult to interpret. These usually require the assistance of a lead burner, who probes the areas with a torch. This will quickly determine whether the defect is a true flaw or not. It must be understood that all indications revealed by this inspection method are not cracks or holes completely through the lead lining. In fact, by far the majority penetrate only partially, but, nevertheless, are important as potential trouble spots.

A limitation of the fluorescent method of inspection is that the operator must exclude as much light as possible in order to observe the fluorescence.

Dye-Penetrant Method

A nondestructive method of testing lead linings and welds requiring no electrical equipment involves the use of a dye penetrant. Imperfections such as cracks, laps, cold shuts, and porosity are indicated by lines, dots, or blotches of a distinctive color on a white background. The process involves the use of three liquids: penetrant, cleaner, and developer, which may be sprayed or brushed on the surface to be inspected. First, the surface is cleaned, and then covered with the penetrant, which is left on for 5 or 6 min. One gallon of penetrant will cover an area of 1000 to 3000 sq ft. After the penetrant is removed with the cleaner, the developer is applied. The colored penetrant will remain in any crevices until it is drawn to the surface by the action of the developer. A colored line or blotch indicates a crack or cold shut; a series of dots in a line indicates a tight crack, cold shut, or partially welded lap. Figure 1 shows the appearance of an imperfect weld in the dye-penetrant test.

The dye-penetrant method has some limitations, compared with the fluorescent-penetrant method, in that the actual surface of the lead is obscured by the developer. The test area cannot be studied alternatively under either black or white light, as is the case with the fluorescent method. Also, it is perhaps less sensitive than the fluorescent penetrant in not showing up very minute flaws and in over-emphasizing others.

Other Tests for Sheet-Lead Linings

Several other inexpensive yet practical methods have been devised for the inspection and testing of sheet-lead-lined equipment in the field, particularly for the detection of perforations. One of these involves the use of kerosene containing dye. The dyed kerosene is pumped or poured between the sheet lead and the vessel shell, after weep holes or other areas that might allow the liquid to escape have been sealed off. The inside of the vessel can be filled with water to counteract the pressure of the kerosene and prevent bulging of the lining. After the water is removed and the kerosene drained through a weep hole, the surface of the lead inside the vessel is inspected for the presence of dyed kerosene, indicating a perforation. Repairs can then readily be made.

Another technique that is still simpler is to pump Freon-loaded air into the area between the shell and the lead lining. By using a halide torch, the presence of the gas escaping through perforations can be readily detected and repairs made. The flange area, of course, must be sealed to prevent gas escape.

Carbon tetrachloride can be used in the same manner by injecting it between the lining and testing with a halide torch. It effectively cuts sludge that might be present and penetrates better than kerosene.

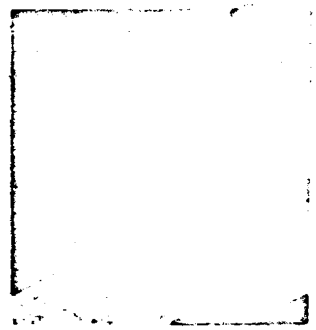

Figure 1—Dye-Penetrant Test on Welded Seam. The colored blotch indicates a defect in the weld. Magnaflux Corp photograph.

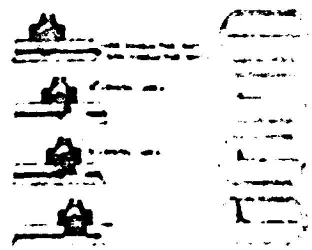

Figure 2—Type of Reading Obtained with SONIZON From Bond Discontinuity in Lead Clad Steel. Magnaflux Corp. photograph.

Figure 3—Radiograph of Brush and Lead Lined Steel. The dark center area is lead on steel, the lighter areas on the right are lead on a double thickness of steel and on the left side is brush lead, and steel. Esso Bayway Refinery Photograph.

[illegible] Procedures For [illegible] Bonded Linings

A bonded lead lining or lead cladding is simply a layer of lead anywhere from 1/8 in. to 1 in. thick (usually 1/4 in.) bonded to steel or copper or even aluminum so as to form a homogeneous metallic structure. This type of construction is used where heat transfer is important, where high temperature, thermal shock, or vibration is encountered, or when the vessel operates under vacuum.

Most lead-clad steel equipment today is produced by variations of the pouring process. In this technique, the steel is prepared by first coating it with a thin film of lead or lead alloy. Molten lead is then poured on to a greater thickness than that specified for the completed vessel. This results in pouring such a large quantity that the lead covering the steel remains completely molten for some time after being applied.

At this point, it is the practice to employ steel rakes to push the lead back from the surface of the coated steel so that the presence of a full bond may be easily and visibly detected. Whenever the first coating has been burned from the steel, which would result in a lack of bond, the condition is easily seen and remedied. The *flame washing* method of removing excess lead, described later, also serves as a practical means for detecting lack of bond during manufacture.

Slight surface irregularities normally present on lead claddings due to the finishing methods (scraping or *flame washing*) that are used to assure uniform thickness and freedom from dross make the use of the penetrant tests described above more difficult. However, as mentioned earlier, light sandblasting can be employed as an effective means of preparing the surface for testing by penetrants. It exposes surface imperfections that might have been concealed by the finishing operation. Furthermore, the light sand residue remaining on the surface after this treatment forms an excellent base for penetrants and makes the *developing* step unnecessary.

ACID WASH TEST. The acid-wash test has been accepted as a standard inspection technique on bonded-lead lined vessels for the detection of pinhole penetration, surface imperfections, and porosity. It will not detect subsurface flaws. First, the entire lead surface must be cleaned free of any oil or grease films. Then, a dilute solution of hydrochloric acid (5 to 15 per cent acid) is freely applied to the entire surface by brushing or swabbing and is allowed to remain for 12 to 24 hr. Before the acid is cleaned off, the lead surface may be examined by an inspector for wet spots or discolorations, or, better still, the acid washed off with water, and the surface allowed to dry and then examined. A wet spot indicates either surface porosity or a pinhole that has not penetrated the lead. Rusty discoloration indicates a pinhole that has penetrated through to the steel. Repairs are usually made on the wet spots by touching up with a lead *burner's* torch. Rust-spot areas are removed and the steel cleaned so that new lead can be bonded. After all rust-spot indications have been rebonded, the acid-wash test is repeated to give a final check.

Potassium ferricyanide solution may be used as a subsequent wash after the hydrochloric acid. Potassium ferricyanide reacts with iron salts to produce a distinctive Prussian blue color that can be readily observed by the inspector.

BOND DISCONTINUITIES. Tests for bond discontinuities or unbonded areas include the two methods described below.

- The completely bonded vessel is sealed and heated internally by means of an appropriate hot-air blower system to raise the temperature of the shell to between 400 and 500 F. If any unbonded areas are present, the thin film of air or gas trapped in this area expands and raises a conspicuous bubble in the lead. It is a simple matter, after cooling, to inspect the vessel for the presence of bubbles and rebond these areas by hand.

- A second method is the same test in principle, but is more effective and is restricted to lead-clad pressure vessels. The vessel is heated by internal steam pressure to about 500 F (680 psi absolute) until thermal equilibrium is reached. At this point, the steam is turned off and pressure rapidly released from the vessel. Air is then admitted and the vessel is cooled and inspected for bubbles. If none are found, the test is repeated. After the second cycle, if none are found, the lining is accepted as 100 per cent bonded. Any bubbles that do occur are removed and the areas rebonded by hand.

This principle is also utilized in the manufacture of bonded linings when *flame washing* is used. This method, employing a *broom* of flame to melt off excess lead, heats the lead sufficiently to raise bubbles in any unbonded areas and also tends to remove those surface areas where dross inclusions would most likely be encountered.

The question of the degree of bond is a thorny one. There are any number of applications where 100 per cent bond is by no means necessary, just as there are others where discontinuities could not be tolerated without courting premature failure. For a consumer to specify arbitrarily a 90 per cent bond, for example, would have little if any practical meaning to the fabricator of the vessel and would have little bearing on anticipated performance, inasmuch as the allowable 10 per cent bond discontinuity could be distributed over the entire surface of the vessel in the form of minute unbonded areas or it could be concentrated in one area where, even though it represented only 10 per cent of the total, it could easily be responsible for failure of the vessel. Thus, it is imperative that the vendor and his customer agree beforehand, not only on how much discontinuity can be tolerated, but also on its location and area concentration.

ULTRASONIC TESTS. Ultrasonic tests involving the transmission and reflection of ultrasonic waves lend themselves well to the location of unbonded areas and subsurface inclusions, and can also provide a simple nondestructive means for determining the lead thickness in bonded linings. Two basic methods of ultrasonic inspection have been used successfully on bonded lead linings: the *Resonance* method, employed by the *AUDIGAGE,** *SONIZON,** and *SONIGAGE,** and the *Pulse* or *Echo* method, employed by the *REFLECTOSCOPE.**

In the *Resonance* method, the crystal head on the instrument transmits an ultrasonic wave into the metal and the waves reflected from the opposite surface are detected audibly in headphones or visibly on a calibrated cathode ray tube or a dial reading.

**AUDIGAGE manufactured by Branson Instruments, Inc., the SONIZON and SONIGAGE manufactured by the Magnaflux Corp., and the REFLECTOSCOPE manufactured by Sperry Products, Inc.*

When lead is well bonded to steel, the waves are not reflected from the steel-lead interface, and consequently no signal is obtained. Small unbonded areas, one half of the crystal-head size or smaller, or semibonded areas, may result in partial reflection of the waves from the interface, indicated by weak signals. Strong signals reveal a complete lack of bond, larger in area than the crystal head and whose outline can be easily traced with the instrument.

In the *Pulse* or *Echo* method, vibrations are sent out through a similar crystal head. The pulse travels through the metal until it strikes a flaw and is reflected, whereas the rest of the waves travel on until they hit the opposite surface and are reflected. Although the vibrations are alternately off, the crystal detects the reflected pulses and the time difference is converted electronically to readable indications of depth on a cathode-ray tube.

Inspection by ultrasonics in most cases is made from the steel side. The steel surface must be free of weld spatter, heavy mill scale, deep gouges, etc., although light rusting, as found on the average plates in fabricator shops, is not objectionable. The criterion is that the crystal head must make good contact with the surface. This may be accomplished by means of a water-soluble oil added to water for work to be inspected in a downhand position or, for vertical and overhead surfaces, a heavy oil, SAE 40, or light grease (vaseline).

The surface to be inspected may be laid off in crosshatched lines at approximately 6-in. centers and checked at the intersections of the lines, or, if necessary, may be tested over every square inch of surface area.

When ultrasonic sound transmission is used, the acoustic properties of the materials involved have a direct bearing on the effectiveness of the technique. This is an advantage in the case of lead bonded to steel because lead has poor sound-transmission properties and therefore enhances the indication of bonded areas. *Figure 2* shows the type of reading that could be expected from a bond discontinuity in lead-clad steel[6] when using the *Resonance* method. When the crystal of the detector passes over an unbonded area, a pip will be obtained whose height is proportional to the defect area.

With the *Pulse* or *Echo* method, the pips at or near both ends of the horizontal line on the face of the cathode-ray tube are back reflections from the far and near surfaces. Defects show up as pips of lesser height between the end reflections and are located by their distance from the end pips.

When a reading is observed that would indicate lack of bond, the area is checked and the instrument rechecked to determine whether there is a lack of bond, the instrument is out of calibration, or foreign matter is present between the vessel and the crystal. If a definite area larger in diameter than, say, three times that of the crystal is established, the lining is sampled at this point to determine the degree of bond by *burning* or chipping away the lead. The area is then rebonded by hand.

Although the sensitivity of ultrasonic testing is high, it is not a panacea for production testing. It is most sensitive to voids normal to the beam, such as a poor bond, whereas a thin seam or crack having a direction parallel to the beam may be missed entirely. The width and length of a defect can be measured but its depth cannot, unless three-dimensional probing is used. For this reason, the selection of the proper wavelength, and the technique used, should be based on the size and type of flaw that harms the operation of the vessel.

The use of a reference plate with known unbonded areas and other flaws is recommended. It aids in adjusting the sensitivity of the instruments and also permits exact duplication at any future time.

Since the presence of other metals such as antimony or tin substantially affects the sound transmission of lead —pure lead is almost *dead*, whereas 4 per cent antimonial lead approaches steel as a sound transmitter—it is conceivable that ultrasonic devices would also be practical, though not as a primary function to detect nondestructively and quickly the presence and perhaps approximate percentage of alloying constituents in lead equipment.

Peening an unbonded lead area, it had been suggested, might alter the ultrasonic characteristics of the metal so as to give a false reading. To test this theory, a plate was pr[illegible] large user of bonded-lead e[illegible] with deliberately unbonded ar[illegible]. The plate was tested with a *REFLECTOSCOPE* (*Pulse* and *Echo system*) and the unbonded areas located and marked. The lead was then heated to about 100 F and sledged with an 8-lb hammer. Re-examination disclosed no change in the ultrasonic pattern whatsoever.

RADIOGRAPHIC TESTING

Although, because of its high density, lead is normally used as a material for shielding against radiation, it is possible with the higher radiation energies commercially available today to obtain practical radiographs of the lead in lead-lined steel vessels. Pioneering in this work, Skiba and Bracken[7], of the Esso Standard Oil Co., Bayway Refinery, and Detler of Esso Research and Engineering Co., found that the radiographic inspection of lead linings is definitely feasible for locating entrapped gases and subsurface foreign inclusions not detectable by acid-wash methods. They obtained radiographs that were useful in locating the cause of failure in a lead- and brick-lined steel vessel consisting of 3/16-in. lead bonded to 7/8-in. steel and covered with 4 1/2-in. carbon brick. A 500-millicurie cobalt-60 source was employed at 36 in. with a 12-hr exposure over an entire 24-in. manhole area. Figure 3 is a radiograph of the brick- and lead-lined steel.

Radiographic techniques are especially useful when testing brick-lined lead-clad vessels as a means of avoiding the excessive expense and time loss involved in removing the brick lining. However, proper and careful interpretation is especially important in this test because radiographs are extremely sensitive and can easily lead to false indications of a faulty installation. In this respect, the use of stereo techniques to show depth of inclusions would aid considerably in interpreting the seriousness of flaws. Lack of bond, incidentally, is practically undetectable by radiographic methods.

Radiographic techniques might also provide a practical means for nondestructively testing lead welds for soundness, much as welds in steel are commonly checked.

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[6] [illegible], "Nondestructive Testing of Bonded Materials With Ultrasonics: a Survey of Theory and Applications", paper presented before ASTM meeting June 1956.

[7] Skiba W. and Bracken V. P., "Radiographic Inspection of Lead Linings", Paper No. 55-PET-13, ASME Conference, New Orleans, September 25-28, 1955.

CONCLUSIONS

All of the foregoing inspecting and testing techniques—penetrants, acid washes, ultrasonics, and radiography—reflect an increasing concern with means of eliminating service failures and downtime in processing equipment. It is a concern that is by no means restricted to lead linings. In fact, all of the above techniques saw their beginning with other materials and owe their present stage of development to this past experience. In the case of lead-lined equipment, the conditions of the past were such that conventional fabricating techniques were sufficient to assure long and uninterrupted service life. However, these conditions are changing today.

The demand for operation at higher temperatures and pressures and under vacuum has thrown more emphasis on the need for effective and practical pre-service tests. In addition, interest is now developing in lead claddings for radiation protection, where uniform density and continuous bond are all important.

Most lead *burning* contractors and lead-equipment fabricators have recognized this need and have already begun using some of the tests described herein. At the same time, the users of lead equipment are themselves developing, applying, and specifying tests, usually working hand in hand with the fabricator. The methods described perhaps do not represent the last word or even reflect the general practice, but they are proof of the attention that is now being given to testing lead linings.

The problem, however, is not so much that of finding defects as of deciding on practical standards of acceptance or rejection. We know, for example, that 100 per cent bond in a bonded lead lining is not absolutely necessary for most applications, but how much bond discontinuity can be tolerated is a question still to be resolved. The Lead Industries Association has attempted to compile as much information as is available and presentable within the limits of this paper on the subject of testing and inspecting lead linings. It is hoped, through this, that the lead fabricators, the contractors, and their customers will be better able to coordinate their efforts and to decide on criteria, to the ultimate advantage of all concerned.

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