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LEAD INDUSTRIES ASSOCIATION

420 LEXINGTON AVENUE

NEW YORK 17, N. Y.

METALLIC LEAD PRODUCTS DIVISION

See Lead Chemical Construction Bull. No. 9

January 11, 1954

CHEMICAL BULLETIN NO. 55

SUBJECT: MATERIALS OF CHEMICAL CONSTRUCTION ANNUAL REVIEW

To Members of the Metallic Lead Products Division:

Attached is a reprint of the Lead and Lead Alloys Section of the Materials of Chemical Construction Annual Review which appeared in the October, 1953, issue of "Industrial & Engineering Chemistry."

Included in "Industrial & Engineering Chemistry's" Annual Review for the first time in four years, this is an up-to-date report on new developments in the uses of lead and lead alloys in chemical construction since 1949. The report covers research progress, developments in engineering applications and such technological advancements as methods of testing linings, automatic lead welding and air pollution control.

Additional copies up to twenty-five may be had free of charge upon request. Larger quantities may be had at our cost of four cents per copy.

Yours very truly,

[signature]

Secretary

Seventh Annual Materials of Construction Review (1953)

LEAD AND LEAD ALLOYS

Distributed by
LEAD INDUSTRIES ASSOCIATION
420 Lexington Ave.
New York 17, N. Y.

Reprinted from
INDUSTRIAL and ENGINEERING CHEMISTRY
Vol. 45, Page 2210, October 1953

Lead and Lead Alloys

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Lead is now in a free market and is readily available; furthermore, a continuing plentiful supply is assured. During the 4 years since the last "Lead and Lead Alloys Review" bonded lead has been accepted as a practical means of combining the corrosion resistance of lead with the strength of steel. Lead covered copper similarly couples the electrical conductivity and greater heat transfer of copper with the superior corrosion characteristics of lead. New and more reliable methods of testing sheet lead linings and lead welds for imperfections have been put into practice along with a nondestructive method for detecting bond discontinuities in bonded lead linings. Interest has been reawakened in the use of cage-type sheet lead construction and in the combination of brick and lead for high temperature, erosion-corrosion service. Installation of strapped sheet lead lining has been speeded up by the perfection of automatic stud welding. The invention of an automatic lead welding machine has contributed to further automation in the lead fabricating industry. Lead and lead alloys are playing an important role in the control of corrosive pollutants, including acids that would normally waste into streams and sulfurous gases and mists wasting into the atmosphere.

FOUR years ago in the "Third Annual Chemical Engineering Materials of Construction Review" (1949), George O. Hiers of the National Lead Co. Research Laboratories presented the last review devoted to lead and lead alloys. This article will bring us up to date.

Market conditions are such that the users of lead are assured of a continuing plentiful supply available free of government restrictions at a price competitive with other materials of chemical engineering construction. Technical improvements have been made in methods of installing lead for sheet linings and for bonding lead to steel or copper, thus combining in effect the corrosion resistance of lead with the strength of steel or the conductivity of copper. Recognition of the merits of using brick linings and lead linings in combination has been renewed. Reliable and nondestructive methods for testing lead linings and welds and a unique method for locating possible discontinuities in bonded lead linings have been developed, field-tested, and accepted.

NEW BOOKS

Several books involving lead and its alloys were published, and revised editions of others were released since 1949.

One of the first was by Lee (19) who succeeded in bringing together and correlating information dealing with more than 200 chemicals and combinations of chemicals. Each chapter is devoted to a specific chemical or process and opens with a brief introduction followed by sections on production, handling, and packaging. In most cases this is followed by a description of the process in which the materials of construction possible for each piece of equipment is brought out. Each chapter also covers the corrosiveness of the chemical when it is being used as a finished product or as a raw or intermediate material in the production of some other chemical or product. By making good use of his considerable experience in this field, as former managing editor of *Chemical Engineering*, and the data from the "Corrosion Forum," a regular feature in *Chemical Engineering*, Lee succeeded in providing much useful and, for the most part, up-to-date information about applications of lead in the chemical process industries.

Also in 1950, the fifth edition of Riegel's "Industrial Chemistry" (22) was released. The author brought up to date a book which, since its first printing in 1928, has become a standard reference for chemical engineers and others interested in current practices and materials of construction for practically all important industrial processes. It is in this book that a statement, familiar to many, first appeared: "It may be said that it is to lead that the chemical industries owe their success."

An English language edition of Rabald's excellent guide on the selection of materials of construction for use with corrosives was published in 1951 (21). Beginning with some of the fundamental aspects of corrosion, the author devoted the bulk of the book to tables of corroding agents with the behavior of various materials of construction including chemical lead and hard lead. Because of the broad scope of coverage it was difficult for the author to provide enough information, at least as far as lead and lead alloys are concerned, to explain some apparent anomalies of behavior. Living up to its title, the book, however, does serve as an excellent corrosion guide.

Zimmerman and Lavine, having published their book on "Chemical Engineering Costs," proceeded to issue supplements in the form of a quarterly devoted to costs of specific materials used in chemical construction (30). The April 1952 issue of *Chemical*

Engineering Costs Quarterly was devoted to lead—the types of equipment available, methods of construction, and costs. Unfortunately their numerous cost tables do not indicate, except in one instance in the text, the date on which these costs were in effect, a factor particularly significant in view of the comparatively rapid fluctuation in the price of lead since World War II.

There were no books still in print devoted entirely to lead until the Lead Industries Association published in the Fall of 1962 a 330-page book entitled "Lead in Modern Industry" (21). Covering in technical detail the manufacture, applications, and properties of lead, lead alloys, and lead compounds, the book devoted two of its twenty-eight chapters to the functions of lead in modern chemical construction. One chapter is devoted to the composition of lead and its alloys recommended for chemical construction—methods of joining, methods of construction and applications for sheet lead linings, bonded lead linings, and brick and lead linings—and concludes with a description of lead pipe used for heating and cooling coils. The latter section gives a formula for calculating the length of lead pipe needed for heating solutions and a chart indicating the maximum allowable fiber stress in chemical and 6% antimonial lead at various temperatures. The other chapter deals with the corrosion resistance of lead and lead alloys including a section on how to interpret corrosion rates; fatigue and stress corrosion; galvanic corrosion; atmospheric and sea water exposure; soil corrosion; soil, waste, and vent exposure; and corrosion data for 136 specific chemical agents.

RESEARCH PROGRESS

Advancements in research, though often in apparently unrelated fields, have often proved to be of direct interest to the manufacturers and users of lead. For example, in a study of some interest to corrosion engineers although prepared specifically for the storage battery industry, Burbank and Simon (1) determined the relationship of the microstructure of lead and lead alloys to anodic corrosion occurring in sulfuric acid. The authors found that pure lead and the lower alloys corroded by intercrystalline attack and that the higher alloys corroded by penetration of the antimony-rich constituents. Alloys containing from 0.5 to 6% antimony corrode by a combination of intergranular and interdendritic penetration.

Lander (11) anodized pure lead at varied constant potentials in sulfuric acid solutions. At potentials below those of lead dioxide (PbO_2) formation a layer of tetragonal lead monoxide (PbO) is formed nearest the lead. Lead sulfate is formed as an outer layer. The rate of corrosion of lead was found to increase with increasing temperature and decreasing acid concentration. At constant time it was found that weight loss is related to potential. At higher potentials a protective film, presumably lead dioxide, is formed.

Electrochemistry contributed to the theoretical understanding of the corrosion of lead according to Delahay, Pourbaix, and Van Rysselberghe, who established the reactions and equilibrium equations of the system lead-water and used them in constructing a potential-pH diagram (5). A modified form of the diagram showed corrosion, passivity, and passivation domains. They also presented a modified diagram which took into account the presence of sulfate in solution and reported on the behavior of lead storage batteries studied on the basis of the modified diagrams.

Experiments by Hopkin and Thwaites (9) showed that little or no age hardening occurred in a lead-antimony alloy (0.25% antimony) of extreme purity although alloys of commercial purity are known to age harden extensively. The authors also showed that alloys containing 0.01% arsenic or silver will age harden rapidly and that the rate of age hardening of ternary alloys containing arsenic was independent of the arsenic content. In fact, 0.001% arsenic had almost as much effect as 0.02%.

Investigations conducted by Nearath and Koehler (20) at Carnegie Institute of Technology, under the sponsorship of the National Advisory Committee for Aeronautics, on single crystals of lead resulted in the establishment of creep properties for lead over a wide range of temperatures. The creep apparatus used was constructed around tensile specimens on which two Tuckerman strain gages were applied.

CORROSION

While the corrosion characteristics of lead enter into most of the developments selected for this review, some papers were devoted principally to this subject.

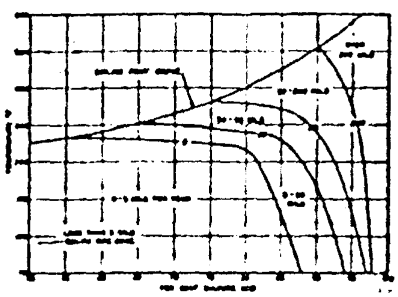

Figure 1. Corrosion of Lead by Sulfuric Acid as a Function of Temperature

One of the most recent of such presentations prepared by Rall (22) gave performance data for lead and lead alloys against 135 specific chemical agents. The corrosion data, presented in semigraphical form showing concentration and temperature limits, are a compilation of information that had appeared in part in a variety of publications, including the books of Rabald and Lee (both reviewed above), The Corrosion Data Survey of the Shell Development Co., and prior publications of the author. In the same article the author described in some detail how lead and lead alloys are currently being used for guarding against corrosive attack, the forms in which lead is used, and unit processes in which lead might be found.

Fontana (8) summarized the corrosion performance of lead in sulfuric acid by means of a chart showing the boiling point curve of the acid on which is marked the range of lead corrosion. Figure 1 neatly indicates where lead can and cannot be recommended for use with sulfuric acid. In the text of his presentation, Fontana describes in some detail the conditions under which lead is used with sulfuric acid, and briefly mentions some advantages of the different forms of lead. He points out that the correct method for testing the corrosion rate of lead should take into account that its corrosion resistance is due to a film of lead salts which form on the surface of the metal. Formation of this film is responsible for an initial weight gain instead of loss after corrosive attack, and it is the adherence and chemical resistance of this film which actually determines the corrosion resistance of lead—not the initial rate of attack during which the films are formed.

ENGINEERING APPLICATIONS

Concern for air and stream pollution and methods and devices for eliminating both have increased with the growth of the chemical process industries. Lead has joined in the suppression of air pollution where sulfuric acid mist and sulfur dioxide, two of the

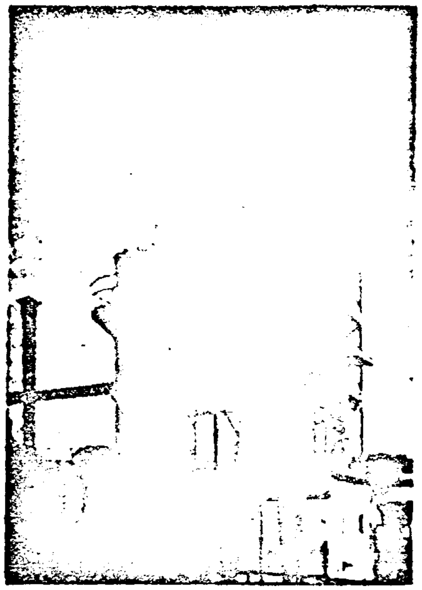

Figure 2. Electrostatic Precipitator Constructed Entirely of Lead Supported in a Steel Strap and Angle Framework

chief offenders, are concerned. Hall (*21*) drew attention to methods and equipment for the control of corrosive air pollutants. Equipment specifically designed for air pollution control including electrostatic precipitators, sonic collectors, and other mist collectors are described with details on their operation and construction. Other methods of controlling air pollution are given including the recovery of acid mists with vacuum-type and drum-type acid concentrators, both of which utilize either lead alone or lead and brick in combination. Figure 2 shows an electrostatic precipitator constructed entirely of lead supported in a steel strap and angle framework. The precipitator treats sulfuric acid mist from a drum-type sludge acid concentrator. The role of lead in the conversion and collection of other harmful gaseous pollutants is also discussed. Chief among these was the absorption of waste sulfur dioxide either for conversion to sulfuric acid or for recovery as purified gas.

The most recent contribution to the latter field appeared in April 1954, in *Chemical Engineering* where the production of high purity liquid sulfur dioxide from roaster gases was presented in the form of a pictured flow sheet (*5*). Practically all the equipment used in this operation is constructed of lead, principally because of its excellent resistance to corrosion by dimethylaniline plus sulfur dioxide. It is interesting to note that this particular system resulted in the development of a unique countercurrent lead heat exchanger that was first publicly described in *Lead* (*14, 18*).

The heat exchanger coils consist of one lead pipe within another with the exterior of the inner pipe extruded in the shape of a six-pointed star to provide support and also to retain concentricity (*14*). The pipes were designed so that when inserted one within the other, the cross-sectional area between the two was approximately equal to that of the inner pipe. Saturated dimethylaniline containing 15% by weight of sulfur dioxide enters three such heat exchangers in series at a temperature of 86° F. and leaves at about 175° F. while the stripped dimethylaniline flowing countercurrently enters at a temperature of 196° F and leaves at about 110° F. Other materials that were used in the initial construction of the coils failed in service and were replaced by lead. The inner pipe is chemical grade lead and the outer 6% antimonial lead.

Details of the complete process for manufacturing sulfur dioxide from waste roaster gases, of which these coils were a part, were published (*16*). Absorption of the dilute gas takes place in a bubble cap tower whose shell is made of 12-pound chemical lead erected in sections and supported by steel columns and ring angles. Inside the tower the trays are made of 8.5% antimonial lead, $^{3}/_{8}$ inch thick, supported on bonded lead covered steel spiders. The bubble caps are 4-inch-diameter antimonial lead extrusions. Stripping of the sulfur dioxide-laden dimethylaniline and recovery of the absorbent are accomplished in bubble cap towers of similar size. Drying is accomplished in a steel bubble cap tower, the lower section of which is lined with 16-pound lead to serve as a sulfuric acid storage vessel. The first tray, subject to weaker acid and wet gas, is constructed of lead as in the other towers while the upper trays and caps exposed to 98% sulfuric acid are of steel.

An article with engineering drawings was devoted to a complete description of a sewage solvent trap and a neutralizer constructed of 6% antimonial lead (*18*). The device designed and used for preventing stream pollution consists of two lead chambers, one filled with broken limestone and the other with a steam line constructed in such a manner that laboratory wastes, usually acidic and containing small amounts of immiscible solvents, enter the steam traced vessel where the solvents are volatilized and leave through a vapor vent. Then the solvent-free acid solution passes through the limestone-filled chamber where the acid is neutralized and passed on out to the sewer system.

Phosphoric acid pickling with a built-in system for regenerating the phosphoric acid was described in *Lead* (*17*). Regeneration of the spent phosphoric acid is accomplished by means of a hydrogen zeolite cation exchange resin. The resin adsorbs iron in the pickling solution in the form of iron phosphate and converts it back to phosphoric acid. To reclaim the resin the flow of the pickling solution is stopped and the tank drained, back-washed with water, and flushed with dilute sulfuric acid. Lead pipe and sheet linings are used for storage and handling of the sulfuric acid.

The Technical Association of the Pulp and Paper Industry reviewed in detail how and where lead is being used in the pulp and paper industry, pointing out that its use is geared chiefly to the sulfite process (*26*). Chapters on stock handling, bleaching, and acid pulping all include sections on the use of lead in these processes. Edwardes (*7*) also described the use of lead in the pulp and paper industry in *Paper Trade Journal*.

The handling and use of tank truck delivered liquid alum by the pulp and paper industry was featured in *Lead* (*15*). Here lead is used because of its resistance to attack by liquid alum (aluminum sulfate, $Al_2(SO_4)_3 \cdot 14.8\ H_2O$) and because existing storage vessels can easily and inexpensively be converted to alum storage by lining with sheet lead. This applies to brick, concrete, wood, or steel vessels as long as they are structurally sound. Lead was used as 4% hard lead sheet, pipe, or valves, in storage vessels, conveying lines, feed tanks, and measuring boxes.

Cole (*4*) drew attention to problems of corrosion in the plants of the Consolidated Mining and Smelting Co. of Canada, which produce ammonia, sulfuric, nitric, and phosphoric acids and ammonium phosphate, sulfate, and nitrate. Considerable lead equipment is utilized in this plant, and the author has given details of the manner in which lead is used and its performance.

Details of a new sulfuric acid dilution and cooling unit were given in *Lead* (18). The equipment constructed entirely of lead consists of a mixing knot connected to one leg of an H-shaped dilution chamber, both submerged in a water bath for primary cooling. The water for diluting and the air for agitating the acid are introduced into the mixing chamber through a removable cover, the rate of dilution is controlled automatically by a specific gravity indicator. After dilution the acid is pumped through a secondary cooling unit consisting of an open rectangular steel tank through which cold water passes in a lead coil manifold. Starting with 66 Bé. acid at room temperature, after leaving the unit, the acid reaches a concentration between 50 and 56 Bé. and a temperature of 115° to 135° F.

Card (8) showed that an almost endless variety of designs of lead equipment is possible with a few simple welding procedures and described, with the aid of drawings and photographs, a variety of types of lead and lead-lined equipment for the electroplating and chemical process industries.

The construction details of new continuous type rotary drum vacuum filters were presented in *Lead* (16). Such filters make extensive use of bonded lead for the filter drum and rolls and the interior of the sludge tank; cast lead for the drainage members, trunnions, and valves; and lead pipe for the drain and feed lines.

How lead-clad steel and copper increase applications of lead was described by Knapp (10) who showed that by combining strength, electrical properties, and excellent corrosion resistance, clad metals comprising lead homogeneously bonded to steel or copper have succeeded in overcoming many of the disadvantages of lead when used alone.

In February 1950, the Lead Industries Association initiated a new series of bulletins for use by engineering students and practicing chemical engineers concerned with the use of lead as a chemical engineering material of construction. Designated "Lead Chemical Construction Bulletins," seven have been issued to date as follows:

1. Corrosives versus Lead: Sulfuric acid, chromic acid, copper sulfate, nitric acid, hydrocarbon solvents, phenol, caustic soda, fluorine, hydrofluoric acid, hydrochloric acid, sodium chloride, sulfur dioxide, wet and dry chlorine, acetic acid, and phosphoric acid
2. Use of Lead in the Pulp and Paper Industry
3. New Developments Expand Use of Lead Construction
4. Lead versus Sulfuric Acid
5. Lead in Modern Chemical Construction
6. Manufacture of Liquid Sulfur Dioxide
7. Lead and Lead Alloys: Corrosion resistance, forms available, fabrication, and applications

All the above, with the exception of No. 6, comprise reprints of articles published elsewhere. Lead Chemical Construction Bulletins are available to readers upon request. Many of the articles cited in *Lead* are also still available on request from Lead Industries Association, 420 Lexington Ave., New York 17, N. Y.

TECHNOLOGICAL ADVANCEMENTS

Despite lead's antiquity—it was probably the first metal to be used as a material of chemical engineering—new technological advancements are currently being made to cope with changing design and operating trends in chemical engineering today. The proper use of lead as a material of modern chemical construction was detailed in the January 1950 issue of *Chemical Engineering* by Roll (24). He explained what should be known about the various types of lead construction, what forms of lead are commercially available, and where these should and should not be used. For example, the article covered in technical detail the construction of sheet lead-lined equipment, pointing out the advantages and disadvantages of cage construction and loose and strapped lead-lined construction. Figure 3 shows typical sheet-lead cage construction in various stages of erection, lead pipe cooling coils, and (lower left) lead-lined valves and pipe. Although not a new technique, the author tried to emphasize that the use of a brick lining in conjunction with a lead lining offered many advantages which are being recognized by more and more chemical design engineers. Such a combination is a natural solution to many problems encountered in chemical construction—wherever high temperature, abrasion, or other conditions exist that tax the physical strength of lead but still require its corrosion resistance.

The use of bonded lead linings involving lead on steel or copper, methods of manufacture, and proper application were described. Also described were lead piping and coils and bonded lead pipe and lead-covered pipe. Formulas for calculating the theoretical over-all coefficient of heat transfer and the area of heat transfer surfaces for bonded lead-covered copper pipe were given.

The need has always existed for accurate, reliable, and inexpensive methods for nondestructively testing lead linings, before putting vessels in operation, in order to locate leaks in sheet linings and discontinuity of bond in bonded linings. Several methods have been evolved to meet this need, and they are discussed for the first time in this article (24).

One of these methods permits detection of tiny openings or discontinuities in lead linings or welds. The process uses three liquids—penetrant, developer, and cleaner—which may be sprayed, brushed, or dipped on the surface under test. The penetrant after developing reveals faults in the weld or lining as a red line, blotch, or series of dots depending upon the magnitude of the opening. Another system utilizes a fluorescent penetrant which is examined under a "black" light.

Roll's article also presented for the first time a new method successfully adopted for detecting unbonded areas in bonded lead-lined vessels. The lined vessel is sealed and heated internally with hot air to raise the temperature of the shell to about 400° F. If any areas are not bonded or only partially bonded the thin film of air trapped in this area expands and raises a conspicuous bubble

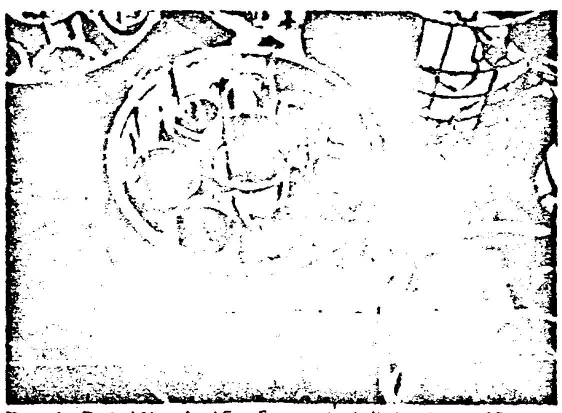

Figure 3. Typical Sheet Lead Cage Construction in Various Stages of Erection

in the lead. It is a simple matter, after cooling, to inspect the vessel for the presence of such bubbles and to rebond these areas by hand.

In another paper, Hall (27) revealed some new developments in lead construction methods, including sheet lead construction, brick and lead linings, bonded lead linings, lead piping and coils, and bonded lead-covered pipe. One of the improvements cited was a simplified method for attaching steel supporting straps to sheet lead-lined vessels by means of automatic stud welding. A threaded stud may be butt-welded, by means of a special gun, directly to the inside of a steel shell, either through precut holes in the lead or between two lead sheets where they butt together. Half-oval steel strapping is slipped over the threaded stud and bolted down on top of the lead.

Though still in a more or less experimental stage, the use of an explosive-powered stud to drive a hardened steel nail through a steel strap and lead lining and into a steel or concrete vessel will succeed in providing faster, less expensive, and more effective erection of strapped lead vessels and x-ray room linings than was heretofore possible with bolts.

The latest innovation in lead welding or "burning" is covered in a U. S. Patent (6) granted for the invention of an automatic lead welding machine. In operation the machine moves a conventional "burning" pipe arrangement over the work to be welded. The pipe is manipulated by cams and gears to simulate the movement of the flame employed in lead welding by hand. The machine has been refined to incorporate multiple burning pipes, thereby enabling it to lay down on prepared steel a strip of bonded lead up to 12 inches wide by 1/4 inch thick.

The operator can control the width and thickness of the bonded lead or "cladding" by regulating the speed of the machine and by spreading or narrowing the two bonding heads with which it is equipped. An important advance in design over earlier models makes it unnecessary for the machine to carry its own lead supply. A strip of lead is laid down under the machine; the bonding heads melt the lead and chemically bond it to the steel as the machine automatically moves across the plate. While the machine may run free, directly on the steel, it will also operate on a track. By taking off the bottom carriage, reversing it, and mounting it on the top of the machine, the unit will run on a lightweight aluminum or magnesium beam so that it can operate inside a tank shell as well.

A hand operated model has also been designed. This hand tool may be used to burn lead to lead as in welding sheet lead pipe. The hand unit, weighing less than 10 pounds, operates without a carriage on a single wheel or tripod arrangement with a grip for the operator.

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Printed in U. S. A.