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LEAD INDUSTRIES ASSOCIATION

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METALLIC LEAD PRODUCTS DIVISION

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SUBJECT: ANNUAL MATERIALS OF
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(1957)

Attached is a reprint of the section on Lead and Its Alloys, prepared by the Lead Industries Association and published in the September, 1957 issue of "Industrial and Engineering Chemistry," official publication of the American Chemical Society. The section is part of the Annual Materials of Construction Review.

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Secretary.

AN [illegible] REPRINT

Lead and Its Alloys

EDWARD J. MULLARKEY
Lead Industries Association, New York 17, N. Y.

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I&EC CHEMICAL ENGINEERING REVIEWS

MATERIALS OF CONSTRUCTION

Lead and Its Alloys

LEAD technology has gained new vigor in such fields as atomic power, particularly mobile power or package reactors; in ceramics with porcelain enameled aluminum; in compounds, inorganic and organic, as exemplified by ballistic modifiers, and in electronic devices for such applications as missile guidance. This interest and activity have been paralleled by several investigative programs. The lead industries, governmental agencies, the armed services, and various manufacturers are all taking part in lead research. Expectation is that such efforts will result in still greater uses. In fields of old-line uses lead continues to show customary dependability. On the economic scene, the lead supply picture is good. Increased demands will be met and the supply maintained at reasonable prices.

New Publications

New publications covering lead are diverse in scope. The Lead Industries Association published quarterly the magazine *Lead* (8), which covered all noteworthy uses and those of current interest. Annual review articles by the secretary were published in *Engineering and Mining Journal* (19), *Iron Age* (20), *Steel* (21), and in the "Year Book of the American Bureau of Metal Statistics" (12). Included in the material on economics was a chapter on lead for a text, "Economic Geography of Industrial Materials," edited by A. S. Carlson of Dartmouth College (2).

Reprints of Roll's paper, "Latest Methods for Inspecting Lead Linings," as it appeared in the November 1956 issue of *Chemical Engineering* (13) were distributed. It gave impetus to recognition by industry that the service life of lead-lined vessels for handling corrosives may be extended through proper inspection and testing. Reprints were distributed of a similar article appearing in *Petroleum Refiner* (15). "Corrosion. A Compilation," by Mars G. Fontana (4), collects informative monthly columns by the author which appeared in INDUSTRIAL AND ENGINEERING CHEMISTRY during the past 10 years. Material devoted to lead's use in chemical construction is collectively excellent. However, the lead corrosion chart for sulfuric acid solutions as it appeared in the September 1951 issue is in error with regard to corrosion rates at concentrations below 50%. It is stated that the acid corrosion rate is less than 5 per year, but there is evidence now that at concentrations approaching the lower extremes, 0 to 5%, the corrosion rate increases precipitously. The rate at 2% acid is roughly twice that at 5% and estimates failure conditions in terms of designs based on previous data.

"How Much Does Lead Change Steel?," an article by A. P. Weaver, was reprinted from *Steel* (17) and distributed as of current interest. According to Inland Steel Co., lead does much to improve metallurgical properties.

Lead pigmented paints were referenced in a reprint of "Paint Galvanized Surfaces," from *Factory Management and Maintenance* (10), and in Red Lead Technical Letter No. 11 (11). The latter referred specifically to surface preparation and its importance in achieving best performance from red lead paints.

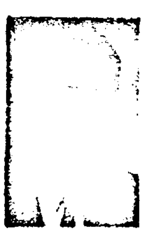
A new book, "Lead in the Ceramic Industries" (9), was published and distributed by the Lead Industries Association as a permanent record of properties and applications of lead compounds that might be helpful to a ceramist in whatever field of ceramics he may be engaged. It is also intended that the book be supplemented from time to time, as ceramic technology is constantly in a state of flux. An article on the use of lead compounds in glass used as insulation for AC spark plugs, which had appeared in *Lead* (7) and was reprinted in *Ceramic News*, indicated the importance of allowing glass to be fired at low temperatures while retaining desired quality and chemical durability. Beneficial aspects of lead were earmarked in this regard.

The handbook, "Radiological Health" (6), distributed by the Office of Technical Services, U. S. Department of Commerce, contains data pertinent to training in connection with radiological health at the Robert A. Taft Sanitary Engineering Center. This issue meets the demand of other federal agencies, state and local authorities, universities, and industry and relates in part to lead physicals and shielding concepts. A handbook, "Nuclear Radiation Shielding," by E. P. Blizard (1) of the Oak Ridge National Laboratory, prepared for the McGraw-Hill "Nuclear Reactor Handbook," was reproduced for use at the Oak Ridge School of Reactor Technology. This handbook is noted for its clarity and simplicity in dealing with the features of reactor shielding with references to lead.

The second edition of the "Trilinear Chart of Nuclides," by William H. Sullivan (16), sponsored and supported by the Oak Ridge National Laboratory and the Technical Information Service of the Atomic Energy Commission, presents the systematics of and physical constants data for all experimentally identified lead nuclides known from information available by approximately July 1956: lead-197 to lead-212 inclusive and lead-214.

Reprints of INDUSTRIAL AND ENGINEERING CHEMISTRY'S Materials of Construction Review for 1956, "Lead and Its Alloys," were distributed by the Lead Industries Association (14). Published proceedings of the Symposium on Atmospheric Corrosion of Nonferrous Metals conducted by the American Society for Testing Materials at its 1955 meeting were made available. The proceedings are based on a report of 20-year tests, including lead, which shows consistent durability in practically all types of exposure (5).

The "Encyclopedia of Chemistry" (3) contains a section on the Lead Industries Association contributed by the



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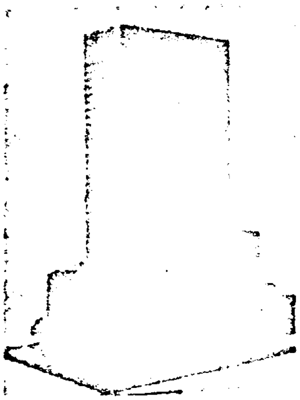
EDWARD J. MULLARKEY, technical director, Lead Industries Association, formerly with Arma Division, American Bosch Arma Corp., has had, in addition to his lead interests, experience in design, development, and manufacture of inertial guidance and control systems for the missile and aircraft industries, areas requiring the utmost from materials of construction. He attended New York University, received the degree of bachelor of Metallurgical Engineering and holds membership in AIME, ASM, AIChE, ASTM, SAE, NACE, AWS, and the Electrochemical Society.

The Doyle scrubber, a new device on the scrubbing scene, uses lead-lined equipment throughout



A model of a 38-story aluminum sheathed skyscraper now under construction in New York City. White porcelain enameled aluminum strips to set off the aluminum panels have increased resistance to chipping provided by addition of lead to porcelain enamel compositions. Lead also provides the low fusion temperature necessary, gives greater smoothness and brilliance to the enamel, improved elasticity, and increased corrosion resistance

secretary. The volume is designed for chemists, engineers, and scientists and refers to atomic power, automation, electron tubes, radioactivity, new elements, plastics, and other topics.

"Developments in Storage Batteries," by H. D. Watson (*1B*), was reproduced and sent to members of the Lead Industries Association. This paper was particularly informative and contemporary and covered all aspects of lead-acid cell development and use.

Engineering Advances

Possible applications and a theoretical and practical description of a lead sulfide photoconductive cell were given by Bradfield and Jenkins (*8A*).

Theory of criteria and efficiency of the extrusion process for metals, relations between extrusion force, ram velocity, friction at billet container interface, and high temperature and rheological properties of metals were established by Feltham (*3A*) and used to evaluate extrusion processes for lead.

Improvement of bearings by electrodeposition of films of lead, lead-tin, or lead-indium on the bearing surfaces of cast-iron, bronze, lead-bronze, silver, and silver alloy bearings was shown by Roggendorf (*5A*) to enhance their effectiveness considerably.

Enamels, developed for zirconium by Schultz and coworkers (*6A*), were adherent, free of crazes and other defects, and had low thermal-neutron absorption cross sections. Frit compositions were based on glasses in the lead monoxide-silica system and successfully protected the metal from oxidation when tested in air for 1000 hours at 600° C.

New developments in smoke control were made at Cominco as outlined by Bainbridge (*1A*). Recovery and collection of dust and fume containing metal values is achieved by means of electrostatic precipitation unit, baghouse, and wet scrubber. The Doyle scrubber, a new device on the scrubbing scene, employs lead-lined equipment throughout for handling corrosive gas mixtures.

Harkort (*4A*) reports a new process for the manufacture of lead glass. The possibilities of substantial savings in the manufacture of lead frits and glazes are presented by use of suitable intermediary products from sorting of lead minerals to metallic lead.

A previously unknown compound, $2PbO\cdot Fe_2O_3$, which may have significance in magnet technology has been identified (*2A*). Results of crystallographic and magnetic investigations so far include the identity, magnetic energy value, remanence, coercive force, and magnetization.

Low temperature ceramic dielectric bodies were fluxed with lead frits (*7A*). A number of highly interesting bodies were developed maturing in the range of 1600° to 2100° F. Some of the unique properties include low or zero firing shrinkage, high impact resistance, and/or ultra-low dielectric loss.

Research Progress

Physical Metallurgy. Considerable interest has been generated in Russia recently and to some extent in the United States and Canada in lead-tellurium alloys which have implications in semiconductor work. The effects of various admixtures on the type of conductivity exhibited by lead telluride, which is considered to be a typical instance of a bicomponent substance, were studied by Kovalchik and Maslakovets (*6B*). They attempt to define the conditions under which *p*- and *n*-conductivity may appear as has been done in the case of the monocomponent germanium and silicon.

Diffusion of lead in lead telluride was studied by Boltaks and Mokhov (*2B*). Experiments demonstrate that introduction of lead into lead telluride changes the conductivity of the latter from the hole type to the electron type. The coefficient of diffusion varies from 35 at 775° K. to 0.7 at 532° K., with the diffusion process being similar to that of copper in germanium.

Flow of heat through lead telluride, lead selenide, and their mutual solid solutions was measured by Joffe (*5B*). The thermal conductivity, *K*, was determined with an accuracy of 2 to 5%, and comparisons were made with electrical conductivity.

Occurrence of <100> glide in lead telluride was verified by Rachinger (*11B*) by means of prismatic punching. At low binding energies, as in lead telluride (61% lead, 39% tellurium), slip occurs by movement of coupled partial dislocations of type $^1/_{4}$ [001].

The ternary system, copper-lead-tellurium was studied in a paper describing the results of original research by Gravemann and Wallbaum (*3B*). Study was over the range copper-copper (I) telluride-lead telluride-lead by thermal analysis and microscopy.

The crystal structures of intermetallic compounds between lithium and lead were the subject of research by Zalkin and coworkers (*19B*). Single crystal patterns about the *a*-axis were photographed with the Weissenberg camera.

Sutton and Jones (*15B*) described experiments which confirm the previously reported inverse segregation of antimony in lead-antimony castings. Supercooling of antimony at eutectic temperatures and a preferred orientation and growth direction for the antimony crystals were cited as responsible for the excessively large coverage by antimony in the cast alloy surface. This information is particularly important in connection with battery grids.

Diffusion of lead in gold has been investigated by Ascoli and coworkers (*1B*) by a radioactive tracer method in temperature range 190° to 300° C.

Diffusion of lead and of trace amounts of bismuth in liquid lead were investigated by the capillary method using radium D (lead-210) and radium E (bismuth-210) as tracers. Rothman and Hall (*12B, 13B*) compared results with existing theories of diffusion in liquids. Agreement was fair, and the heat of activation for self-diffusion in lead was found to be close to the corresponding activation energy obtained from viscosity data. However, later studies (*14B*) of diffusion of bismuth and lead in molten bismuth using the same technique gave activation energies of more than twice the values calculated from the viscosities.

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Japanese research in this area by Niwa and coworkers (8B) has shown that the diffusion coefficient, D, of bismuth in molten lead-bismuth as measured at 450° to 600° C. in dilute solution confirms that diffusion takes place in the manner of metal ions. The influence of concentration was studied at 500° C. with a maximum value for D being found at 60 atomic % bismuth. This is in agreement with the thermodynamic properties of the solution, which shows strong negative deviation from Raoult's law. Similar studies of the diffusion of antimony in molten lead-antimony (9B) show that it is the antimony ions that diffuse and, assuming that the diffusing particles are ions, theoretical values agree with experimental results.

Studies of kinetics of dissolution of solid copper in liquid lead and bismuth under static isothermal conditions at temperatures in the range 360° to 510° C. were performed by Ward and Taylor (15B). Experimental data appear consistent with dissolution being a complex process involving diffusion of solute through laminar boundary layers, combined with convection effects introduced through density changes accompanying dissolution.

An improved capillary method for measurement of surface tension of molten metals up to 1000° C. has been devised by Melford and Hoar (7B).

The limits of solubility of three metals, tellurium, selenium, and manganese in molten lead (10B), show decreasing concentrations with decreasing temperature in all three cases.

Valenti (17B) used the electromotive method to determine the sections of immiscibility at the isotherms of 440° and 520° C. for the zinc-bismuth-lead system.

Good diffraction patterns on liquid lead have been obtained by Smith and Sharrah (16B) and analyzed to obtain the atomic distribution curve. The results differ somewhat from those obtained by x-ray diffraction methods.

The results of a study by Hoffman (4B) on the effects of low temperature on soft solder have just been released and indicate that lead is practically unaffected by low temperature. Tin, an alloying constituent, in its pure form was proved to be unreliable when exposed to long-term, low-temperature storage conditions. However, no conclusive information is available concerning what effect there may be on soft solder. Small additions of antimony and/or bismuth appear to inhibit the transformation of tin, and recent modification of government solder specifications reflect this thinking.

Mechanical Metallurgy. Tests to determine how pulses of plastic deformation disperse during propagation along a lead bar were conducted (1C).

The effect of fatigue stresses on recovery properties of lead was the subject of research by Kennedy (3C) who measured creep of lead with a sinusoidal fatigue stress superimposed on a static stress. He investigated the recovery of the transient creep properties during the unstressed periods of an interrupted creep test. An index of recovery as defined by Kennedy was determined, and an explanation of increase and decrease in rate of recovery is given in terms of dislocations.

A rapid method of estimating fatigue limit or endurance limit of lead in reverse bending is given by McKeown (5C). Progressive loading technique is used, and tests are conducted on Wohler-type rotating bending machines. The method is considered promising and might find its most important use in determining the effect of changes in design in improving or detracting from fatigue performance.

Cold-impact extrusion forces on lead were measured by Gokyu and coworkers (2C) oscillographically using strain gages. Force-time curves were obtained whose course was similar to that of curves for static extrusion. Reduction was a maximum at greater than 95%. The impact extrusion force might be equal to or less than the static force if reduction and rate of extrusion were increased.

The effects of frequency and amplitude of alternating cyclic torsion on the maximum plasticity of lead have been studied by Rybalko (6C). Maximum plasticity decreases with the amplitude of the deformation while increasing frequency increases plasticity.

The elastic and plastic limits of lead at the temperature of liquid helium have been determined by Khodcrvich (4C). Lead was shown to be still plastic in this area of cryogenic research.

Nuclear Physics and Nucleonics

Inelasticity in collisions between pions and lead nuclei was determined (3D) from direct measurements of the energy of charged pions interacting with lead nuclei and of energy of the neutral pions produced in these interactions.

Differential elastic scattering of 14-m.e.v. neutrons in lead was determined by measurement of differential cross sections for the elastic scattering in a ring geometry. A biased detector with a spherical liquid scintillator was used. Results are in qualitative agreement with theoretical predictions (8D).

High energy nuclear interactions in lead by cosmic ray protons were studied by Giacconi and coworkers (4D) in a multiple cloud chamber, triggered by Geiger-Muller counters. A study is made of the development of the nuclear cascade in lead.

Cross sections of (μ,n) reactions in the isotopes of lead have been made by Bell and Skargard (2D) by the activation method in lead-206, lead-207, and lead-208.

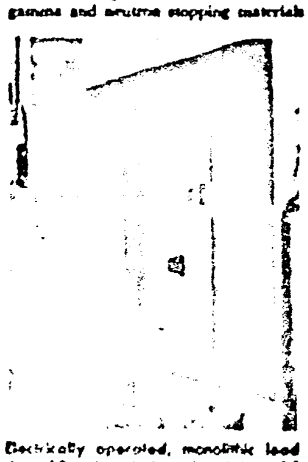
A value has been obtained for the absolute pair production cross section of lead at 2.76-m.e.v. γ rays of sodium-24. The value obtained according to Standil and Moore (10D) for the cross section is 2.38 ± 0.62 barns.

The lifetime of positrons in superconducting lead was investigated by Green and Madansky (6D) at 4.2° and 10° K. Comparisons were indicated.

Giera (5D) states that lead is an excellent γ shield especially where space is at a premium. Major design requirements are discussed for effective biological shielding of hot caves and cells and nuclear reactors, including a comprehensive review of the properties, characteristics, costs, and other considerations for several types of metallic, organic, and concrete shields.

Correction for build-up factor for lead shielding has been calculated by Arders (1D). Gamma shielding values for two common energy levels, 1 and 1.3 m.e.v., have been determined and plotted graphically together with values uncorrected for buildup.

Race (9D) described the composite shield materials designed to provide a combination barrier to stop γ-radiation with lead pellets and to stop neutrons with hydrogenous liquid filling the interstices between the pellets. By using single-sized pellets to obtain a specific packing factor, a range of ratios between the gamma and neutron stopping materials



Electrically operated, monolithic lead door 12 inches thick and weighing 15 tons permits access to concrete-lined cave containing 3 pounds of cobalt-60. Cities Service will use this installation to study the effect of nuclear radiation on refinery processes and products may be obtained by changing pellet core size and core filling material.

Attenuation of 84- and 176-m.e.v. synchrotron x-rays in lead was determined by Miller and Kennedy (7D).

Attack of metals by lead at 1000° C. was studied by Wilkinson (12D). Of the several metals studied, cast iron and uranium were the only metals to undergo complete dissolution.

Attack of metals by 52 bismuth-32 lead-16 tin alloy at elevated temperatures (11D), involved no attack on molybdenum at 800° C. or beryllium at 500° C. Aluminum, titanium, and zirconium were damaged. Iron is insoluble but subject to intergranular attack.

Corrosion

Oxidation. Researches on the oxidation of metals and alloys in the fused state by Spinedi (9E) referred in part to lead and the systems tin-lead and lead-cadmium. The behavior in the solid or liquid state was studied by thermogravimetric methods.

The effect of steam on metal corrosion by chlorine was the subject of studies by Tseitlin (11E). In addition, the combined effect of air and steam on corrosion of lead with a protective oxide coating by chlorine is included with effects of temperature.

Anodic. Anodic coprecipitation of trace amounts of manganese and silver with lead dioxide has been accomplished (3E). This was demonstrated by carrying of silver and manganese on anodically formed lead dioxide. Neither of these elements could be anodically deposited under the experimental conditions in the absence of lead.

According to Burbank (2E), the anodic corrosion product formed on lead in sulfuric acid solution depends on the potential of the metal surface. In the potential range between lead-lead sulfate and lead sulfate-lead dioxide, monobasic lead sulfate, lead hydroxide, and lead monoxide were identified by electron diffraction.

An outstanding development in this area in what constitutes a refutation, in part, of Burbank's findings is the report by Rüetschi and Cahan (7E) of the Electric Storage Battery Co. Their paper was well received and indicated that anodized lead and lead-antimony alloys exhibited the following corrosion products: α-PbO_2, β-PbO_2, and $PbSO_4$. Lead monoxide was not in evidence particularly and these findings were further enhanced by the independent discovery by Cahan of α-PbO_2, a crystalline modification of lead dioxide, which was also the principal anodization product. This work has been termed significant to the general theory of lead corrosion processes under anodic conditions and is of pertinent interest to lead-acid cell manufacturers.

In further studies on anodic corrosion of lead in sulfuric acid solution, Lander (6E) interprets the thermodynamics and kinetics of the corrosion process in order to indicate means of preventing or slowing down corrosion, thus allowing longer positive battery grid life.

Briggs and Wynne-Jones (1E) have studied the anodic behavior of lead in halide solutions by anodic polarization of freshly cut lead surfaces at constant current density.

Chemical. Corrosion of lead in sulfuric acid as influenced by the structure of the metal was described (4E) in terms of the mechanism of action of various anticorrosion additives in lead.

According to Trillat (10E) a new x-ray spectrograph (with a rotary tangent cylinder) has made it possible to study the kinetics of the formation of soaps resulting from the attack on lead by fatty acids as well as the structure modifications as a function of the temperature.

Hess (5E) has concluded that biological decomposition of jute and not phenol is producing the so-called "phenol corrosion" of lead cable.

Galvanic. Corrosion properties of titanium and zirconium coupled with lead and other metals in various inorganic solutions was the subject of research by Schlain and coworkers (8E). Titanium and zirconium usually resisted corrosion whether alone or in contact with a dissimilar metal.

Testing and Inspection. Of corollary importance to corrosion studies and mitigation, the methods used in maintaining and inspecting lead structures for handling corrosives are paramount.

Roll (2F, 3F) indicates that resistance of lead to chemical attack depends on protective films formed when lead corrodes and accordingly outlines proper inspection and maintenance of protective films on lead linings.

In a second look at radiographic inspection of lead linings during fabrication, Skiba (4F) has shown by experience that, with gamma-ray inspection defect-free, homogeneously bonded lead linings can be produced.

Lowrison and Heppenstall (1F) review the methods and materials of construction used in the manufacture of sulfuric acid, from the handling and storage of raw materials through the acid concentration stage. Lead is used extensively in this area.

Tait (5F) discusses corrosion of heat exchange equipment, cracking units, and equipment for the handling of acid and caustic liquids in petroleum refining. The main sources of corrosion and methods of prevention and inspection are reviewed with reference to lead.

Ceramics

"Lead in the Ceramic Industries" (9) affords a permanent record of the properties and applications of lead compounds that may be helpful to the ceramist. Phase diagrams are included.

Gallup and Dingwall (3G) have determined the viscosity, thermal expansion, density, and index of refraction for lead borosilicate glasses containing more than 70% lead monoxide.

Weight loss, dye absorption, crystalline phase or phases, dielectric constant, dissipation factor, and temperature variation of dielectric constant were determined for various bodies fired in an air atmosphere at temperatures from 800° to 1100° C. by McQuarrie (8G) in his study of the system (barium, calcium, lead) titanium trioxide.

Structure and phase transitions of ferroelectric sodium-lead niobates and of other sodium niobate-type ceramics have been studied by Francombe and Lewis (2G). Of importance to synthetic asbestos investigations, the synthesis and properties of fibrous potassium-lead silicate have been studied by Shell and coworkers (6G).

A patent covering an internal glass-to-metal seal has been granted to Dalton and Voss (1G). The seal has a metallic electric conductor passing through and hermetically united with the glass body. An especially suited glass was cited as of approximately 75% lead monoxide, 11% alumina, 11% boric oxide, and 3% silica composition. It has a working point of about 560° C. and an expansion coefficient of 84×10^{-7} per ° C.

Giles (4G) has discussed the general characteristics and processing methods of low temperature porcelain enamels. Low temperature vitreous glasses maturing in the 1000° to 1100° F. range are principally lead-bearing and fuse down to produce very thin, glassy coatings having a texture preferred for a number of uses.

Chemistry

A review of lead compounds in industry was given by Stewart and Reid (1H). Six areas were outlined in which new lead products will supplement older lead products now in use.

A method of particle size estimation has been developed by Thomas and Yoder (2H) which is dependent upon aerosol penetration through a lead-shot column.

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