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LEAD INDUSTRIES ASSOCIATION

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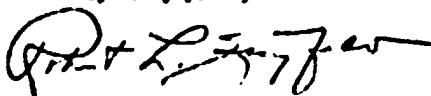
To Members of the Lead Industries Association:

Attached is a reprint of the section on Lead and Its Alloys prepared by E. J. Mullerkey, Technical Director of Lead Industries Association, and published in the September, 1958 issue of "Industrial and Engineering Chemistry," official publication of the American Chemical Society. The section is part of the Annual Materials of Construction Review.

This year's review marks a significant departure from past issues in extent of coverage as well as technical involvement, in keeping with present day trends and L.I.A.'s over-all program in research and development.

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Lead and Its Alloys

EDWARD J. MALLARKEY

Lead Industries Association, New York 17, N. Y.

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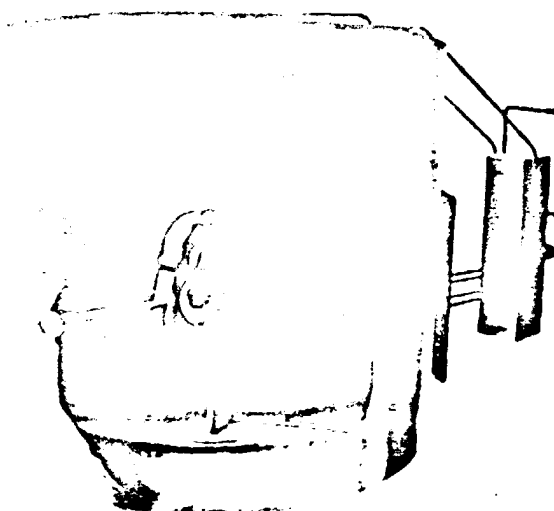
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MATERIALS OF CONSTRUCTION



Cutaway of miniaturized atomic energy research reactor. This unit is only 8 feet high and 8 feet in diameter. The 3500-pound pear-shaped lead reflector shield can be seen in center of reactor.

engineered for use by American firms, Ebasco in particular, for ship hull, steel pier, and pipeline protection and offers excellent working characteristics. Lead has always been important in general chemical, water, and soil corrosion and its use as an impressed current anode rounds out the applications picture.

In the field of high temperature, an uncommon application area for lead normally, two developments appear significant, both of which involve the beneficial addition of lead to a structural material. In the first case, it has developed that titanium benefits by as much as 2 weight % lead at high temperatures, in the area of 1000° F. Because of diffusion and solubility characteristics, lead has the ability to move with imperfections or dislocations and thereby maintain the properties of the bulk material, which may persist under similar uses. In the second case, ceramic-coated magnet wire for high temperature applications has been made possible by 1% additions to the coating material. Some measure of this effect can be obtained from a comparison between the volume resistivities for leaded and unleaded coatings at 550° F.—130,000 and 335 megohms, respectively. Such coatings include a number of different ceramic compositions that can withstand continuous operation at temperatures to 1000° F. They can also be used to bond electronic

components such as transistors, diodes, and relays under similar circumstances (7).

The Russians may have taken another jump in the field of alloying. They claim to have accomplished what long has been considered impossible—the true alloying of lead and iron (8). Experiments carried out at the Central Research Institute of Ferrous Metallurgy have shown that the properties of crystallizing metals can be altered substantially by subjecting it to ultrasound treatment (this is not new) and that such treatment also opens up far-reaching prospects for forming alloys unobtainable by ordinary methods. Little is known about the merits of such an alloy commercially and the proof of both remains to be seen in this country.

Lastly, Minneapolis-Henrywell Regulator Co.'s new Inertial Guidance Center is using the sound-attenuation properties of lead. Concomitant with the demands of the space age, lead has moved into the "white noise" room at this installation where 25 tons balanced on a knife-edge support vibration equipment. This use of lead is unique, as it forms part of a sandwich construction, the purpose of which is twofold: to isolate transmission of vibration to adjacent ground and structure and to assist in preventing undesired planes of vibration in missile component test equipment, so that the vibratory motion will be confined to a single plane.

Research Progress

It seems fitting that the extraordinary features of lead, lead alloys, intermetallics, metalbonds, and the like should in treatment provide the more rudimentary though important advances in lead technology. PbS, PbTe, and PbSe have become increasingly popular as semiconductors, particularly as infrared detectors and as working materials for thermoelectric refrigeration and power. With respect to detectors, the merits of lead compounds are well-challenged, whereas for refrigeration and power, certain efficiency breakthroughs remain to be made before such developments can become commercial. As heat engines, lead compounds are receiving careful consideration on both a fundamental and an applied basis.

Facts brought to light in connection with lead compound semiconductors would include derivative (5.1) and the interstitial diffusion of sulfur (4.1) and copper (3.1) in lead sulfide single crystals. The microstructure of photoconductive lead sulfide layers seems to consist generally of crystals 200 and 300 Å in size and, for vapor deposited layers, correspond to a regular fcc lattice of the sodium chloride type (5.1). Photoconductivity in lead selenide, as it relates to the theory of the dependence of sensitivity on film thickness and absorption coefficient, also received attention (3.1). In a more applied vein several papers presented at the Electrochemical Society's spring meeting are representative of the attention being given to lead compounds. Scandon (14.1) helped clarify many concepts as to the properties of PbS, PbTe, and PbSe and the understanding of polar semiconductors in general. For this class of compounds, the solid phase cannot be considered separately from its vapor phase, particularly at elevated temperatures.

In compound electroluminescence, lead as an additive appears beneficial. One study indicates that lead enhances the plaque brightness of a 3000° K. calcium halophosphate phosphor (16.4); in another, it activates the ultraviolet fluorescence of some urinary silicates (17.4); and in another, the wave length of the peak fluorescent emission of tungstate is shown to be dependent on its lead content (10.1). The implications are intriguing in terms of new products in the fields of electronics and lighting.

Ceramic materials, ferroelectrics in particular, containing lead have received mention in connection with permanent magnetism (12.4), x-ray and neutron diffraction (9.4), production and properties of single crystals useful as transistor devices (13.4), and basically

A new type of chargeable dry cell, using an alkaline lead oxide-silver system, offers exceptionally long shelf and service life, and relatively constant voltage throughout a wide temperature range

In a number of papers presented at the American Ceramic Society meeting, The revelations are indicative of the attention being given to such materials in varied fields and supplement present knowledge.

Lead superconductivity has had practical use and the normal resistivity of lead coupled with this property has made lead and certain lead alloys attractive in many applications. As a result investigations have been made with respect to alloying (1,2, 2.1) and effects of hydrostatic pressure on the transition (2.4). Prati and Templeton (2.3) have made a precise measurement of the superconducting transition temperature for zone-refined lead— $7.175^{\circ} \pm 0.005^{\circ} \text{K.}$ in zero field, compared with Bonner's previous value of 7.22°K. ; the observed transition temperature differential was less than 0.001°K. Exploitation of superconductive phenomena is still in its infancy but startling developments are in the offing.

Still another item of interest is that a lead-niobium alloy exhibits controllable pyrophoricity in massive form. This feature was noted by the military in arctic areas as an efficient and simple means for igniting gasoline fires. Gas ignition is normally a great problem because of low vapor pressure at such low temperatures. As a striker it would appear to be perfect, and, in the fields of general ignition, have many new and potential uses.

Physical Metallurgy. The lithium-lead system has been looked at recently as a possible means to improve lead structurally. It is known, for instance, that lithium acts as a strengthening agent and marked increases in physicals have been noted. Adding to information on the system, the structures of Li_2Pb and LiPb have been determined (1,18). The Li_2Pb cell is f.c.c. with $a = 6.68^{\circ} \text{A.}$ and LiPb is hexagonal with $a = 4.751^{\circ} \text{A.}$ and $c = 8.587^{\circ} \text{A.}$ Studies on the crystal structure of KPt show that for a quenched specimen (300°C.) it is a Laves phase with the MgZn_2 (C_{14} -type) structure. The lattice parameters are $a = 6.66$, $c = 10.76^{\circ} \text{A.}$, $c/a = 1.61$ (28).

Concerning liquid immiscibility in the lead-uranium system, Triel (98) confirmed previous conclusions by describing in greater detail experiments on which they were founded. New data on uranium solubility in lead and on the crystal structure of UPb by neutron diffraction are also presented. UPb is b.c. tetragonal with $a = 11.04^{\circ} \text{A.}$, $c/a = 0.961$. Density is estimated to be 13.7 grams per cc. Certain discrepancies

between the values for the cell size of UPb are discussed.

The similarities between carbon steel and silver-lead alloys, in so far as dependence of modulus on composition is concerned, were made evident by Umetani (108). Young's modulus changes linearly with composition, with its temperature dependence behaving similarly. Elasticity of lead single crystals was studied by the method of diffuse x-ray scattering, using both a Gieger counter and film (18). From known compressibility the various elastic constants are derived and are believed to be more exact than those determined by mechanical methods. Creep of lead single crystals as it relates to activation energies for high temperature creep was studied by Wiseman, Sherby, and Dorn (128). For high purity polycrystalline and single crystal specimens, activation energies of 26,000 and 24,000 cal. per mole, respectively, were reported. It appears that the activation energy is essentially the same for both cases and equal to the respective activation energies for grain boundary shearing. Covering the temperature range of 0° to 250°C. for the entire bismuth-lead system, Muryukov and Tyapulina (78) investigated the temperature dependence of thermal conductivity, electrical conductivity, and specific heat and cited many important anomalies. Thermal expansion measurements from room temperature to 300°C. by dilatometry and with x-rays were found not to be different for lead (118). This was attributed either to the too small concentration of vacancies at high temperatures or the influence of vacancies on the lattice parameter.

On gas content and related property effects, two investigative efforts may be considered. The first is the determination of contents and effects for carbon dioxide, water, and hydrogen (15). Secondly, absorption of hydrogen was shown not to be present in electrolytic or common lead or in lead-copper up to 1% copper (58). Lead-magnesium alloys up to 1% magnesium show an increasing ability to absorb hydrogen, which the authors connect with Mg_2Pb phase. Efforts are being made to connect alloy improvement to the presence of hydrogen or hydrides in the case of alloying with tellurium, selenium, arsenic, and lithium, which form hydrides.

Aging characteristics of lead-antimony alloys with effects of strain were studied by Hooker (18), who explained hardness fluctuations as aggregates of individual changes due to preaging, age hardening, and effects of straining.

Properties observed during hardening were explained as being results of advanced coherent aggregates. The influence of impurities on the macroscopic structures of lead in terms of the properties and characteristics of arrays of low-angle boundaries which appear upon freezing of single crystals was studied by Atwater and Chalmers (18). Effects were shown to be dependent upon the amount and kind of impurities in the freezing liquid.

The application of electromechanical techniques to determine changes of structure in lead due to thermal or mechanical processes was recommended by Kaiser (58) as an excellent complement to conventional methods. Certain representative changes occur during melting and solidification, which are noted and analyzed by atomical apparatus.

Mechanical Metallurgy. The influence of small amounts of lead on the tensile and fatigue properties of SAE 1018, 1045, 8620, 4140, and 4340 steel was determined by Brock and Sinclair (30). Lead has little influence on the tensile properties, and the mean fatigue limit of the leaded steel was significantly reduced only at ultimate tensile strengths above 130,000 p.s.i. Notch sensitivity of the steel was affected only slightly. At stresses above the endurance limit, leaded SAE 1018 and 4140 tended to give longer fatigue life with smooth specimens. Similar information is not available in this report on the other steels or on notched specimens. For cast steels containing from 0.12 to 0.20% lead, higher machinability ratings and improved finishes are noted over similar unleaded steels (50). An excellent treatise ("C") has been published with a view to extending their use in ordnance applications. The economics of this attitude appears irrefutable.

Effects of stress on recrystallization of lead during creep were studied by Gilman (30). The strain necessary to initiate recrystallization of high purity lead during creep has been determined for the range of stresses from 300 to 1500 p.s.i. (constant load). Essentially, recrystallization occurs at higher strains with higher stresses, grain size after recrystallization becoming smaller with increased stress. The performance of soft solders at elevated temperatures and pressures indicates the superiority of high-tin alloys under cyclic loads at 257°F. (50). Small additions of manganese, chromium, iron, nickel, germanium, and cerium improve the performance of a 70 tin-30 lead solder,

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but only the latter three benefited a 90 to 95% lead oxide.

With respect to lead sheath for power cables, Hovner (8) lists the present physical, details of manufacture, working characteristics, and corrosion behavior of the bismuth-lead-uranium alloy. A summary is given of data on electrodeposited metal coatings on uranium with reference to electrodeposits of lead (9C); effects of lead coatings on uranium in wire drawing, roll chalking, soldering, forging, and in various parts such as camera shutter blades, aircraft gears, bearings, guns, hydraulic cylinders, and landing gears.

Nuclear Physics and Nucleonics

The effectiveness of lead as a gamma shield has been manifestly demonstrated by the attention it is receiving in the literature. One could almost say that if the shield never, it has lead in it. Studies on the shielding requirements for shipment of metallurgical samples were outlined (5D). Tanks and corrosion samples, irradiated at Materials Testing Reactor for long periods, have to be shipped in carriers to distant laboratories. It is recommended that a lead-shielded carrier be provided that has a minimum lead thickness of 6 inches for the side walls, 6 inches for the top, and 5 inches for the bottom. The inside container space should have a minimum diameter of 11 inches and a height of 9 1/4 inches. Such a carrier provides space and shielding for the shipment of two samples. An "exception" permit must be obtained for shipment of samples having an activity greater than 27 curies normally allowed by the Interstate Commerce Commission. No programs for gamma attenuation with build-up in lead give attenuation factors for lead and iron shielding alone used with point gamma sources (2D). Other "mobile" shields are now being used or are slated for use in nuclear-powered ships, aircraft, rockets, and devices essential to the safe movement and handling of isotopes.

Information on the screening effect of lead and other heavy metals against gamma rays (7D) shows that screening ability depends on the coefficient of absorption, which is a function of density and atomic number, and for the following metals increases in the order: iron, lead, tungsten-copper-nickel, or tungsten-nickel, tungsten, uranium.

In primary shield materials studied for SIC (3D), shields of lead, mild steel, and lead mild steel combinations were considered. Large quantities of lead can be saved through the use of borated water, if associated corrosion problems can be solved. Results of

freshness corrosion studies, boron compound additions, and boron compound toxicity studies are given. Corrosion studies, three proposed configurations for the shield tank, all lead, all mild steel, and a combination of mild steel and lead showed corrosion rates in several borated water solutions to be in the order 0.1 to 0.6, 0.0 to 0.1, and 0.3 to 0.9 mil per year. With respect to galvanic action, chemical action was responsible for most of the corrosion. The maximum calculated penetration for any combination of boron solutions and steel configuration was 2 mils per year. Irradiation corrosion tests of the above combination under SIC primary shield tank conditions were also undertaken for two borated solutions. The results, yet to be released by the Atomic Energy Commission, should prove very informative. The anticipated saving in lead is due to the marked reduction of gamma emission emanating from water as the result of neutron knock-on. Lead as a gamma shield per se is not being replaced by borated water in this application. It is a case of reducing the natural of gamma radiation generated or emitted.

Abundant positron and electron fluxes from lead targets irradiated by bremsstrahlung have been observed at angles between 25° and 4° and energies between 50 and 170 m.e.v. (10). By use of several target thicknesses, the fluxes from lead have been separated into large angle positron production and scattering of positron particles originally produced at 0°. Photoneutrons ejected from a small lead target by 2.5-m.e.v. bremsstrahlung x-rays from a betatron were observed by means of recoil protons in nuclear emulsions (4D). The angular distribution of the more energetic photoneutrons was nearly isotropic.

Application of liquid lead and lead alloys for shielding, fuel suspension, and heat transfer has dictated the need for information on working characteristics. Heat transfer coefficients for molten lead-bismuth eutectic are presented for flow in annuli externally heated at a constant rate, by Deban and Casey (4D). Diameter ratios of 1.30 and 1.74 were investigated and results were obtained in Prandtl numbers from 400 to 1600. Bismuth-lead eutectic and pure bismuth penetration of graphite of various densities under 300° C. and pressures up to 20 p.s.i. was of the order of magnitude of 1.0 gram per sq. cm. uniform throughout the graphite (9D). Bismuth was absorbed less than the eutectic but the oxidation rate of bismuth would be prohibitive. There was heavy nitriding in nitrogen atmospheres with the bismuth and the eutectic.

In the studies of possible reactor fuels consisting of the fusible material pos-

sessed in a liquid metal, either as a solute or as particles of compounds containing the fuel, investigation of the lead-uranium system has clarified the discrepancy in the literature concerning the solubility of uranium in molten lead, and an experiment involving lead-uranium alloy in contact with iron at 600° C. for 1 month showed that no gross diffusion of uranium into iron occurs (10). In an effort to understand more fully the problems concerning a slurry reactor, initial experiments using U₃, U₂O₃, and UO₂ in liquid lead, 55% bismuth-lead, and bismuth have been completed.

Corrosion

Three articles on the use of lead chemical construction derive corrosion. The first deals with its use in handling refinery corrosives, mainly sulfuric acid, new developments in lead construction over the past 5 years, and a general treatment of each of the methods for installing lead for corrosive service (5E). The latter treatment divides the conditions which warrant a particular method and procedures for implementing that method. Pertinent methods are described and an informative case history is documented. The second article relates the experiences of a large mining and smelting company with lead and antimonial lead in chemical and metallurgical plant construction (11E). Many successful design innovations are described and particular reference is made to the merits of antimonial lead in many applications over other corrosion grades on a corrosion basis. Lastly, a discussion of lead-lead brick construction offers erroneous conceptions on the limitations of lead as a supplement to acid brick (7E). It further treats the details of this type of construction which contribute to maximum derivable benefit, economics, and the merits of radioactive sources in the location of leaks. Leak detection in this type of construction has always been difficult and has led to considerable hours in down time and to wastage of man-hours. It is, however, seemingly resolvable as a drawback, because of the ever-increasing development and use of isotope techniques.

In building construction, the behavior of lead under various conditions of application, including contact with other building materials, particularly concrete, and methods of avoiding or correcting failure, was treated by Jones (5E). Conclusions were drawn on the most efficient use of lead in relation to corrosion problems. A digest of reports over the past 40 years on corrosion of lead in concrete covers normal concrete and those made with cement or

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special aggregates, effect of addition to molten water, and external agencies promoting corrosion (11).

Electrochemical. Prins (94) has developed an equilibrium diagram for the lead-water system which is useful in estimating the passive or nonpassive behavior of lead in solutions of varying pH and at varying potentials. But, as brought out by various researchers, its use should be modified in terms of the invariable existence of nonequilibrium in practical applications. Burdick (25) has expanded on the potential-pH phase diagram for lead by definition of the crystalline phases present as electrode species. The lamellar nature of the anodic product formed on lead under conditions obtained in the lead-lead cell is further interpreted on the basis of the diagram. On the subject of the electric and electrochemical properties of alpha-Pb and beta forms of Pb, their dependency on their oxygen lattice defect was considered by Ruzicki (115). Anodic polarization of lead in sulfuric acid under conditions of oxygen evolution leads to layers of alpha-PbO₂ with excess oxygen in the surface. The amount of excess oxygen is related to oxygen overvoltage and corrosion; included in the study are a large number of lead alloys.

Underground. An excellent contribution to a better understanding of underground corrosion problems, made by Romanoff (116), comprises the final report on studies conducted from 1910 to 1955 in an extensive field burial program and laboratory studies of electrical and electrochemical aspects. The data on lead are very valuable and recommended to those interested or active in the mitigation of lead corrosion problems.

Adding to the "phenol corrosion" controversy is the paper of Sears and Pichimoy (127). They characterize the white corrosion of lead sheets as the result of attack by volatile fatty acids present in the form of gases in raw waste used to pack caskets. Again, the effect is attributed to the action of bacteria which hydrolyze the coars, and not to phenol.

Environmental. Another extensive investigation initiated in 1940 in the Panama Canal Zone reveals the corrosion properties of lead and other metals and alloys exposed to five natural tropical environments for 1, 2, 4, 8, and 16 years (17). Up to 1957 specimens through the 8-year period were returned and examined. The report compares lead with respect to rates for aluminum, nickel, zinc, copper and structural steel.

Chemical. A relation between the catalytic effect of copper on oil oxidizability and corrosiveness of oils with respect to lead and antifriction bearing

platings of lead and lithium is reported by Doushmane and Larmann (12). A treatment of anticorrosive additions is also given. The corrosion properties and electrolytic deposition techniques for lead-nickel alloys containing up to 6V% lead were outlined by other Russian investigators (118).

That aluminum enamel must corrosion by boiling alkaline water was amply demonstrated by King and others (52). The enamel for aluminum which exhibited corrosion rates of about 1% that of bare aluminum when exposed to boiling water for 100 hours are lead bearing.

Ceramics

Basically, the number of papers presented at the American Ceramic Society's annual meeting and their titles best represent the scope of present lead usage or contemplated future usage in ceramics:

- Phase relations in lead silicate, PbSiO₃ (19)
- Ceramic and dielectric properties of lead metavanadate electrical ceramics (19)
- Polymorphism and ferroelectricity of lead metavanadate (49)
- Dense aluminum-borate bodies having low firing shrinkage (19)
- Phase equilibria in the binary system PbO-Pb₂O₃ and compounds of noble and inactive with PbO, Bi₂O₃, and Bi₂O (19)
- Piezoelectric ceramics (19)

Chemistry

The composition and isotopic constitution of the gaseous products of the pyrolysis of lead carbonate have been studied for reactions between 350° and 625° C. (18). Thermal decompositions of alpha-lead oxide and lead carbonate were studied by Griffiths and Gronow (19) and Perotti (26). The study of lead oxide entails kinetics and the reaction mechanism, whereas for lead carbonate, the solid phases formed by three stages of decomposition were found to be PbO, PbCO₃, 2PbCO₃, PbCO₂, and PbO in order of increasing temperatures. Growth of lead nitrate crystals was further investigated by Williams (27).

New Publications

The Lead Industries Association publishes quarterly the magazine *Lead* (119), which covers many new uses as well as new slants on the old. Annual review articles were prepared for *Engineering and Mining Journal* (121), *Steel* (122), *INDUSTRIAL AND ENGINEERING CHEMISTRY* (123), and the "Year Book of the American Bureau of Metal Statistics" (124). "Use Lead to Control Refinery Corrosion" (98) deals with the latest practices in lead chemical construc-

tion which explain how to get best results with lead-lined equipment. A treatise on leaded steel (125), was published by the Office of Technical Services, U. S. Department of Commerce, with the view of extending industrial uses.

Red Lead Treated Steel (126) offered highway engineers specifications for complete paint systems to be used in protecting highway structural steel. *Lead Building Construction Bulletin* (127) offered specifications for lead chemical laboratory drainage systems. "Lead in the Ceramic Industries" (128) was supplemented by articles devoted to enamel for application to glass, and low-temperature glazes as bonding materials for crystals, information useful in the industry.

The new supplement to the "Encyclopedia of Chemistry" contains a section on annealing metals which gives additional metallurgical data (129). And filling the need for a complete source of data in as far as underground corrosion is concerned is Romanoff's (116) "Underground Corrosion," covering 45 years of research and writing. Finally, another engineering materials handbook contains a section on lead with useful data on corrosion as well as other application areas (130).

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