LEAD INDUSTRIES ASSOCIATION

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SUBJECT: LEAD, ITS ALLOYS AND COMPOUNDS-REVIEW-1960

To Members of the Lead Industries Association:

Attached is a reprint of "Lead, Its Alloys and Compounds," the annual coverage given to lead technical developments by E. J. Mullarkey, formerly technical director of the Lead Industries Association, appearing in the January, 1961 issue of "Industrial and Engineering Chemistry."

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Lead, Its Alloys and Compounds

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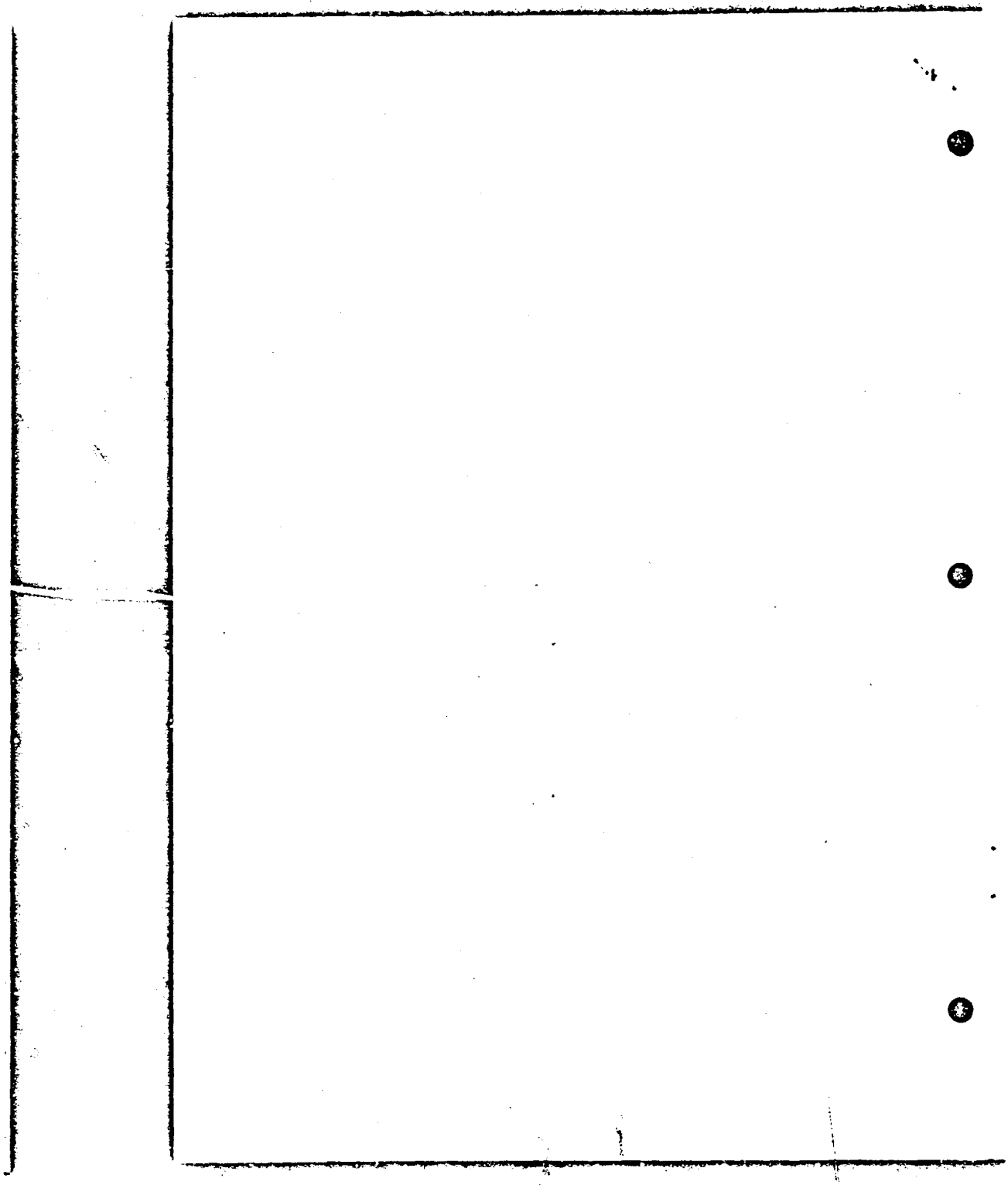

an I/EC Materials of Construction Review

Lead, Its Alloys and Compounds

by Edward J. Mullarkey, *Lead Industries Association, New York, N. Y.*

- Flexible partitions made from nylon-coated lead "fibers" have good acoustical properties
- Polyethylene-lead sandwiches combine the best advantages of each material for efficient nuclear shielding

In an era which places great emphasis on high strength and low weight in its technology, there is understandably some difficulty in promoting lead as a material of construction. However, the current upheaval in materials science, which places emphasis on manipulative microquantities, tends to correct this situation by limiting the parametric importance of tensile and creep strength. Furthermore, the practiced handling of such minute quantities as electrons will, in the future, dominate the applications picture. Current trends in research and development reflect this view more and more.

Metallurgy

There is a pattern which suggests that significant alloy developments are in the offing:

- Favorable sites exist at grain boundaries for solutes of both negative and positive size deviations (*18.4*).
- Consequent improvements could be made in creep behavior by addition of spectral assortments (*1.4, 17.4*).
- A proof of principle for lead has shown it to be a high temperature material subject to the same limitations at room temperature as those imposed on copper above 405° C. and titanium above 773° C.

Others have since established that various solute additions, singly and in combination, caused improvements according to expectation, with some not easily attributable to adsorption alone (Lead Industries Association expanded research). The effects of nickel are particularly important.

Several questions have been posed, however, regarding the need for corroborating the above results with creep tests because their engineering importance militates against acceptance on the basis of tensile data alone. Another question involves the efficacy of internal friction techniques used to validate Winter and Weinig's thesis for establishing with greater surety effects of various solutes, as what constitutes improvement in one parameter may not in another. Finally, there is the relative permanence of selectively adsorbed, assumed beneficial solutes. Would migration proceed away from the grain boundary at too prohibitive a rate, as suggested by Intrata (*11.4*)?

Data from research in an area far afield of alloying offered a plausible answer to questions regarding the anachronistic behavior of nickel. The unusually good strengthening rates obtained with nickel in solute adsorption studies might be better explained on the basis of properties in the liquid state. This is an interesting aside to the equally interesting fact that densities of nickel-lead solutions increased with nickel content. This was determined in an investigation of factors which control mass transfer in heat exchangers of nuclear reactors employing liquid metals as transfer media (*15.4*).

The disparity in density is explained on the basis of carryover to the solid form of structural characteristics peculiar to the liquid—i.e., freezing-in of interstitials which, apart from causing anomalous densities, causes dilation of interstitial sites and related stress field impedances of decided importance to matrix strengthening. Because the structure of a liquid not far from its freezing point is not too different from that of the solid (*4.4*), beneficial effects of various solute additions might be better attributed wholly or in part to matrix strengthening, as indicated, considering as a particular case the duplicity of nickel over identical compositions.

What may be substantive evidence of a carryover from a condition in the liquid state to the solid state is found in experiments with lead melted and solidified in hydrogen and in air. Room temperature aging was encountered which could not be ascribed to oxygen or other impurities. Hooker (*10.4*) suggests that clustering and eventual condensation of quenched-in vacancies were responsible.

Another important disclosure is that metals and alloys with new structures have been created by a technique called splat cooling, representing a rate of 2×10^{6}° F. per second (*13.4*). California Institute of Technology researchers shot droplets of hot metal against the copper rim of a rapidly spinning wheel cooled to liquid nitrogen temperatures. The droplets flattened into pieces of foil 0.5 square inch in area. Several new alloys were created, most of which were of prime interest from the semiconductor standpoint. Gold-silicon, for example, exhibited a novel and complex structure 24 hours after cooling.

Limited in its application because of the small sizes involved, such a technique might nevertheless provide a flake lead suitable for compaction or rolling if there was merit in the resulting [illegible] anical properties. Such a technique might also be of advantage in the synthesis of mixed metal oxide materials incorporating lead. There is much interest in the area already.

Other work in this field is described in Table I.

Steel Making

"Leading" and "getting" are seldom thought of in terms of the same additive in steel making. It is seldom that a development of significance in the

nuclear field, far removed from steel making, can be applied to improving machinability and to removal of deleterious hydrogen, oxygen, sulfur, and certain other impurities in ingot production. When steel makers put lead and lithium together in this sense, they were in effect performing two tasks as one with commensurate cost savings.

Lithium Corp. of America is currently conducting much of the pertinent research with the additional view of promoting markets for lithium (*2B*). Lithium is a remarkable element, and its effects on lead are even more remarkable. The shielding alloy, containing 0.69% (weight) lithium, has a much higher tensile strength than pure lead and takes advantage of the neutron absorption properties of the isotope lithium-6 in combined thermal neutron-gamma shield applications. A great deal is attributed to very little lithium on a weight basis, but, atomistically, there is approximately one lithium atom for every five lead atoms; on this basis effects are more easily understood.

Also, the generally accepted belief that lead is virtually insoluble in liquid iron has been shattered completely by Lord and Parlee (*3B*) and others. It has been confirmed that lead has appreciable solubility in iron and that the true solubility lies in the following range:

Sol., Wt. %	Temp., °C
0.22—0.26	1550
0.27—0.33	1600
0.34—0.40	1650
0.37—0.43	1700

Further inquiry is being made into the mechanism of lead retention in greater quantities. The upper limit of lead content has not been approached yet, according to Inland Steel (*1B*), and such increases are expected to result in still better free-machining characteristics (*4B*).

Powders

When combining virtually insoluble metals with lead via the powder or fiber technique it is at times difficult to determine what is responsible for the resulting properties. There is information which indicates that dispersal of metal powder in lead improves its tensile properties. This is accomplished by stirring in powders of low solubility, such as copper, nickel, and cobalt (*6C*).

On the other hand, there is the allusion to strength obtainable by infiltrating spherical, closely packed, hard-phase powders with soft matrix material carbonyl iron with solder, for example (*8C*). In this case, properties are said to be in agreement with those predicted by theory, but the strength did not reach the strength of hard phase particles because ideally perfect specimens could not be made. Contrast with this the fact that mechanical properties of steel fiber (lead-tin) composites increased almost linearly with increasing steel fiber content but were relatively unaffected by the tin content of the lead (*2C*) and the situation is thoroughly confusing. To think of betterment in terms of one constituent denies that there is a third material or new class of materials combining, it is hoped, that which is beneficial in each.

Practical interest has been evinced, for example, in copper-lead bimetallic strips bonded to steel. The material is produced by sintering uncompacted powder to the steel, compacting by rolling, and resintering (*1C*). Another slant consists of compacting powders coated with another material. Superior products are claimed for this technique, as compared with techniques for standard powder mixtures. Results are given for electrical brushes consisting of Cu-Pb coated graphite (*4C*). There is also some credence given to the reverse—i.e., coating lead powders with other materials, such as an alloy, another metal, or oxide. In the case of oxides, fragmentation occurs which results in a fine, inert dispersal whose influence on properties may be likened to interference hardening combined with an inhibitory effect on grain growth and boundary movement.

Lead powders were treated by Rutkowski (*5C*), who related elastic properties to dimensional changes in compacts. Dimensional changes are increased by a decrease in particle size and an increase in hardness. Effects of different compacting pressures can equalized by compacting additions such as solutions of camphor and alcohol.

Table I. Metallurgy

Subject	Ref.
Effect of solutes on preferred orientation	(*9A*)
Effect of solutes on grain growth	(*5A, 9A*)
Temperature dependence on grain migration	(*5A*)
Boundary migration during creep and grain growth	(*7A*)
Creep under multiaxial stresses	(*9A*)
Effects of physiochemical factors on creep in surface active media	(*10A*)
Creep of lead and lead alloys	(*10A*)
Internal friction of lead	(*5A*)
Grain boundary extrusion and deformation during fatigue	(*11A*)

Bearings

NASA's Lewis Research Center has developed a PbO-based dry film lubricant which provides high temperature (1000° F), high load, and high speed (2400 to 10,000 feet per minute slide velocities) performance. A typical composition contains about 5% SiO_2, 5% Fe_2O_3, and the remainder PbO. This and other formulations have been applied to Type 440C and 304 stainless steels and Inconel X as a dry powder or water slurry and fired at 1650° to 1800° F. (*1D*).

Another bearing that requires no external lubrication has been developed by a British firm, Glour Metal Co. Intended for more commercial uses, such as automobiles, machines, and aircraft, the bearing material, called DU, gives outstanding performance over a range of operating conditions- 10,000 hours of continuous service at 10,000 r.p.m. and better at pressures in excess of 8000 p.s.i. and temperatures of −450° to 536° F. Garlock, Inc., is the exclusive outlet in the United States. In make-up, DU consists of a mixture of lead powder, 60% by weight, and Teflon infiltrating a sintered bronze matrix backed with tin-plated steel. The bronze network and steel backing provides both strength and a heat path which permits cooler operation. Lead-Teflon imparts low friction and nonadherence qualities.

Shielding

Some additional light was shed by Karam and Wethington on the poor understanding of the effects of boundaries between materials and of penetrations in multiple layers (*3E*). Polyethylene, on a mass basis, attenuated gamma rays with energies ≥ 0.55 m.e.v. better than lead, and lead attenuates photons with energies ≤ 0.55 m.e.v. better than polyethylene. These facts, plus knowledge of the Compton and photoelectric cross sections of both materials (*8E*), suggested that a combination of polyethylene followed by lead would make a more efficient shield than an equivalent surface density of each.

Measurements of shielding efficiency in broad beam geometry indicate that for a surface density greater than 17 grams per sq. cm. of polyethylene the attenuation became less than that of lead. For a surface density of 3 to 17 grams per sq. cm. of polyethylene followed by any amount of lead, the combination gave better attenuations than the same mass equivalents of either material. A particular combination, containing 5.58 grams per sq. cm. of polyethylene followed by 7.28 grams per sq. cm. of lead, was reported to provide a shield containing 1.496 mfp of poly-

ethylene and 1.975 m.f.p. of lead. The dose build-up factor for this repeated layer combination was 3.20, which compares with 3.48 for the same thickness of material in combination with all the polyethylene followed by lead. This information is important, particularly as lead is used in intimate combination with a number of organic materials for shielding purposes, either in layers or in mixtures with lead powder or pellets (*1E*, *2E*, *4E*, *6E*, *8E*). The merit in such combinations has been discussed in context with the physics and materials engineering of portable reactor types (*7E*).

Optics

It is practical to consider protracted power needs for the next few decades as depending in large part upon exploitation of the solar region. Indeed, for purely engineering reasons, sources of energy other than the sun are either too expensive, nonrenewable, or both. Lead compounds, basic carbonate of white lead in particular, are suitable for the exploitation of both heat and light and thereby to involvement in a futuristic power outlook. Basic carbonate of white lead is virtually black throughout the heat spectrum and nearly opaque in the ultraviolet, the latter a seemingly incongruous but much desired property.

Recently, the National Aeronautics and Space Administration (NASA) became aware of basic carbonate of white lead as enabling maintenance of temperatures tolerable for both man and instrument in space (*4F*). A payload, heated by solar radiation on the one hand and cooled by radiation on the other, must be protected from widely varying temperature differentials. Constant temperatures are presently maintained by painting, polishing, and shuttering. In the ultimate, a means is sought based solely on surface emittance and reflection characteristics (*1F*). Selective scattering may be feasible, in addition, by controlling the surface texture of crystals otherwise operating on absorption or interference phenomena or both. With thin, platelike crystals, it is possible to exploit orientation, size, width, and surface texture.

Commercial significance lies immediately in the areas of aeronautics, astronautics, solar energy utilization, air conditioning, and boiler-type furnace design among others. It is significant that the atmosphere is transparent to radiation with wave lengths in the range from 8 to 12 microns, which is enough to cause speculation as to the merits of basic carbonate of white lead, whose absorptivity peaks at 7.1 microns, as the perfect solar collector. Some adjustment may be possible, thereby achieving close approximation of the selective black for solar heating.

Multiple coating systems can be used to achieve very low absorptivity-emissivity ratios. A first coat might be a basic carbonate of white lead applied at relatively high film thickness. The top coat might consist of a rutile TiO_2 pigmented, water white vehicle at 25% poly(vinyl chloride) with optimum pigment particle size distribution. The white first coat is necessary to reflect that portion of the light transmitted through the rutile paint. The coating will still possess a high absorption in the ultraviolet. Therefore, application of a very thin third coat of white lead paint should optimize the reflection of ultraviolet while remaining transparent to longer wave lengths.

An extension of this idea to incorporation of interference phenomena in platelike crystal formulations might allow complete control over spectral admittance and rejection and perhaps amelioration of yellowing. Ultraviolet radiation causes a decrease in the reflected blue light and, for most paint vehicles or binders, a significant increase in solar absorptivity results (*8F*). It is conceivable that a Murano formulation chemically identical to basic carbonate of white lead can be made to reflect blue selectively - a fourth coating that negates solarization by virtue of eliminating blue. This would be preferable to costly research into synthesizing resistant silicones and other polymers.

Murano colors, marketed by Mearl Corp., are basic carbonate of white lead crystals of high index of refraction produced in varying interference widths which allows a two color play in crystals devoid of pigmentation - i.e., colors produced by an optical effect rather than by absorption of a particular band of wave lengths, as occurs with ordinary pigments and dyes; one color is observed by reflected light, the second by transmitted light (*2F*). A spectral assortment of decorative two color plays is possible on any curved surface from automobile fenders to furniture legs and in flat transparent plastic sheets and films provided that a mottled orientation is achieved, all modified or intensified by resorting to varying background colors. A polyester sheet or film containing a blue pigment, as a pertinent example, appears blue when held against white. However, even with a white undercoat the reflection color, or blue, is still seen at the gloss angle or highlight where specular reflection occurs directly from the crystal layer.

Inasmuch as Zerlaut's work (*1F*) involves basic lead carbamate ball-milled for 40 hours, a procedure tending to destroy the platelike character of crystals, there is little likelihood that crystal orientation was a factor in his results. Researchers at New York University are determining characteristics of hexagonal platelets, in methyl Cellosolve, 10 microns in diameter and less than 0.1 micron thick. If preliminary work is promising, a variety of dimensions will be investigated along with some further perfection or smoothness of the hexagonal faces.

Table II. Liquid Metal Properties

Subject	Ref.
Sound attenuation	(*1G*)
Solubility of krypton (inert cover)	(*2G*)
Electrical resistance	(*3G*)
Russian counterpart of "Liquid Metals Handbook"	(*4G*)
Low temperature coefficients of solubility vs. interstitiality	(*5AA*)
Structure of liquid lead	(*4A*)

Liquid Metals

A study of the effects of reactivity between liquid metals and the equipment handling them as heat removal media or as nuclear power plant working fluids is being supported by NASA at the University of Michigan Research Institute. In fact, most of the interest in liquid lead or lead alloys at present seems to stem from work in this field, a fact made particularly evident by preoccupation with lead-bismuth (*4G*, *5G*). Other work in this area is listed in Table II.

Thermoelectricity

All of the earth's tellurium and selenium could not together satisfy material requirements for large-scale introduction to consumer markets of thermoelectric refrigerator-freezer appliances, a market potential now standing at approximately 5 million units per year. Exploring the facets of lead's use as the heavy metal component in Peltier devices is therefore of minor importance. Units that do appear will be expensive, primarily military, and will undoubtedly involve bismuth, scarce itself but preferable from the figure of merit standpoint as economics will not forestall its selection.

For power generation, the supply seems adequate for all anticipated needs. Here, PbTe is most efficient in the temperature range of interest, as evidenced by its use in a variety of SNAP devices. Commercially, forced air fans in home heating units will most certainly use thermoelectric power (waste heat), and perhaps there will be self-propelled spits for outdoor rotisseries. Other use is likely in the realm of d.c. power supplies for regions remote to

purchasable power thermoelectric generators for cathodic protection of pipelines and buried structures of all kinds and for aircraft and ship beacons. For the latter, Martin Co. has a 100-watt isotopic unit on the verge of introduction, with a 10-year anticipated life. Many of similar construction are slated for satellite reconnaissance and space probe function and perhaps for purposes under the seas.

Studies on semiconductor properties of lead compounds are contained in Table III.

Superconductivity

A 10-fold improvement in accuracy in measuring magnetic fields associated with lead at 4 milligauss has been reported by the Jet Propulsion Laboratory. They have mapped magnetic fields in lead tubes 18 inches long by 4 inches in diameter with thicknesses of 10^{-4} and 10^{-1} meters to determine homogeneity of the fields in large space volumes. Such tubes have application in furnishing magnetic fields for masers (solid state amplifiers) and for electron paramagnetic studies. Also claimed is a new figure for resistivity—6×10^{-20} ohm-meters.

Niobium has all but eclipsed lead as a high speed rotor material for "cryo gyros" based on the Meissner effect. Both materials, lead and niobium, exhibit transitions above 4° K. and a field strength of at least 500 gauss at 4° K. Oddly enough, in an area where everything is seemingly odd, lead is considered least preferable because low density is a desirable property along with high tensile strength, good machinability, low modulus of elasticity, high hardness, and isotropic thermal expansion. Some effort is being made to investigate clad materials, perhaps lead on niobium (*IJ*).

Work on superconductor properties is listed in Table IV.

Acoustics

A grant from the Ford Foundation led to development of a new material for operable partitioning of spaces in schools (*IK*, *3K*). Growing out of a desire for flexibility in meeting a variety of instructional requirements, the material is based on the principle of achieving great density without porosity in a woven fabric that retains the linear qualities of draping and the pleasant visual and tactile rewards of more traditionally woven fabrics. A nylon-sheathed lead "fiber" is used to provide adequate sound blocking at efficiencies better than those predicted on the basis of weight alone. Still in the development stages, tests at the Riverbank Acoustical Laboratories rate a single layer at an average 34-decibel transmission loss. Two layers with a 2-inch air space between provided an average transmission loss of 43 decibels.

A survey on what may best be described as the internal damping characteristics of structural systems has been compiled by the American Society of Mechanical Engineers, for the purpose of gathering information for eventual inclusion into one convenient reference. It does much to clear away many misconceptions regarding the nature of damping and what may be expected from different designs using different materials (*4K*). A further enlightening article by Hutchison (*2K*) treats ultrasonic absorption in solids, specifically delving into the question of how sound is damped by imperfections.

Table III. Semiconductor Properties

Subject	Ref.
Compounds and solid solutions in metallic systems, SnTe-PbTe	(*1H*)
Properties of the system AgSbTe$_2$-PbTe	(*2H*)
Thermoelectric properties of PbTe	(*3H*)
Reaction of O$_2$ with PbSe	(*4H*)
Mobility of electrons and holes in PbS, PbSe, and PbTe	(*5H*)
Optical properties of PbS, PbSe, and PbTe	(*6H*)
Crystal size vs. spectral response limit of evaporated PbTe and PbSe	(*8H*)
Solubility limits of PbTe	(*9H*)

Table IV. Superconductor Properties

Subject	Ref.
Annealing of thin lead films	(*8J*)
$n(E)$ vs. E curves for lead and alloys with Tl or Bi	(*9J*)
Optical constants of lead films in air and vacuum	(*4J*)

Corrosion

Except for use in certain environments such as soils and highly resistive polluted waters, lead anodes seem firmly entrenched as cover-all sources of mitigative current in cathodic protection circuits. A report of Subcommittee T-2B-7 presented at the March 1960 National Association of Corrosion Engineers (NACE) meeting in Dallas served to acquaint corrosion engineers with the details of lead anode use in various electrolytes, including feasibility in soils as well as the relationships between occurrence of PbO_2 and the extraneous influences of electrolyte resistivity and capacitative effects in the developing film (*7H*). It also included a description of cast "shrinkage" electrical connections used to install a string of 12 duct anodes at Portsmouth, N.H. This joint holds promise of high reliability, having resisted boiling seawater for six months to date (*1L*). If proved, such a joint will greatly simplify anode fabrication.

Examples of good practice are the fender protection system of the Sidney Lanier Toll Bridge, Brunswick, Ga.; the water boxes and heat exchange equipment in a number of utilities; tests conducted at the Port of New York Authority's Pier 11; and a number of others protecting ship hulls—all engineered by the Electro Rust-Proofing Corp. Apart from proponents in England, it has been the first to introduce lead anode systems in this country. Important among the other successes was the use of lead anodes in handling several refinery problems (*5L*).

Increased current-carrying capacity has been afforded by bimetallic anodes—i.e., lead anodes with platinized microelectrodes imbedded in their surfaces. Platinized lead anodes have been used for protection of seawater intakes in Malaya for the past two years with 100% success. Cable type anodes with platinum electrodes have also been utilized both in Kuwait and Aden for protection of jetties and have proved highly satisfactory, although in use for only 15 months. They have also been used for protecting a steel cooler box—25 amps per sq. ft. in practically boiling seawater. These anodes are only satisfactory in salt water, and breakdowns in water with over 100 ohm-cm. resistivity can be expected (*5L*).

The exact mechanism of platinum's beneficience has been established by Grennell and Wheeler as essentially that of a metallic conductor between the peroxide layer and the substrate layer (*1L*). Additional studies relating to corrosion are found in Table V.

Porcelain Enameled Magnesium

A special surface treatment for magnesium developed by the Dow Metal Products Co. has made possible the use of standard lead oxide frits as used on aluminum (*1M*). The enamels provide

Table V. Corrosion

Subject	Ref.
Soil corrosion of cable sheathing characterized as stress corrosion	(*7L*)
Penetration of cable sheathing by marine organism	(*11L*)
Polyatomic phenols as salt water inhibitors	(*9L*)
Oxidation in water in presence of atmospheric O$_2$	(*1L*)
Anodic oxides of lead	(*2L*)
Lead anode corrosion in ZnSO$_4$	(*6L*)
Pb-Sn-Ba alloy positive grids for lead-acid cells	(*12L*)
Orientation effects on single crystals	(*13L*)
Chemical construction	(*11L*, *13L*)

excellent resistance to abrasion and chemical attack without permitting undercutting at bare or exposed edges. Magnesium alloy panels were successfully protected against 20% salt spray for periods up to 500 hours. In addition, good thermal shock resistance is provided—i.e., no spalling when water quenched from 800° F., which promises wider application for such high temperature parts as jet engine components.

Books

A state of the art treatise on inorganic chemistry is the first in a series presenting collections of articles written by leading researchers. It includes a treatment of mixed metal oxides (*IN*). A new publication on reactor shielding is an excellent effort aimed at encouraging further investigation into the basic physics of shields. It supplies an exposition of the fundamental shielding process which forms the basis of reactor shield design (*3E*).

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