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LEAD INDUSTRIES ASSOCIATION

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REVIEW - 1959

Attached is a reprint of "Lead and Its Alloys," the annual coverage given to lead technical developments by E. J. Mullarkey, Technical Director of L.I.A., appearing in the September issue of "Industrial and Engineering Chemistry."

Additional copies will be available as long as our supply lasts.

Very truly yours,

Secretary.

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AN REPRINT

Lead and Its Alloys

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

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MATERIALS OF CONSTRUCTION

Lead and Its Alloys

Changes in our environment influence to a large extent the progression of ideas, processes, and products.

Lead's behavior in cold is a first example. Developments in [illegible] brought forth as a material which, contrary to general expectation and to the exclusion of many so-called structural materials, increases in strength and ductility, and exhibits superconduction, thermal nonconduction, or their converse conductivity, perfect electrical shielding properties, weight-damping, reduced thermal expansion, and transmission of ultrasound up to 1000 times normal, and possibly out of the ordinary shielding properties. Another example is interest in lead as a cosmic and nuclear ray shield.

Another is miniaturization, in terms of compact refrigerators, home appliances, and power sources for the military, generating interest in the thermoelectrics—lead selenide and telluride. The fact that we are noisily going about our business prompts further interest in lead as a low frequency sound absorbent. Its use for noise abatement is growing in the aircraft, mechanical design, and architectural fields. And with wide-screen theaters, the ever-increasing number of amateur photographers and projectionists, and superabundance in lighting, come problems of vision and interest in the optical properties of nacreous pigments. They, too, are gaining the attention of optic specialists everywhere.

General Developments

Following success with lead foil in abating noise in air passenger liners, a

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successor material involves lead powder-impregnated vinyl coating on cotton duck and glass fabrics, which combines the acoustic properties of lead (loss decibel increase per pound) with the strength of glass or cotton fibers. The material achieved rather wide acclaim, and a manufacturer used it for sound abatement in an electric typewriter. Its use for general area isolation is envisioned. Manufactured by Curdo Chemical Corp., Countlab is a noteworthy development.

Pearl pigment manufacturers have extended a purely decorative medium to some rather sophisticated optical applications, from movie screens to area lighting, to road marking to sewer, and to more effective packaging schemes.

Exmet offers expanded lead in a variety of fine meshes. Used in the battery field, such a material undoubtedly has other uses—perhaps electrochemical, structural, or, in combination with other materials, composite use. The surface-volume ratio is of particular interest.

Many manufacturers are producing portable packaged research and training reactors, but none involve as novel an application of lead as Atomics International's L-77 homogeneous solution type, following the first nuclear reactor employing lead as the only reflector material. Unique in its field, the primary shield comprises three concentric regions and makes extensive use of lead pellets. The innermost region, composed of a mixture of lead and diphenyl, surrounds a stainless steel sphere containing 20% enriched uranyl sulfate. It acts primarily to attenuate γ-radiation, reduce neutron leakage, making use of lead's reflector characteristics, and moderate escaping neutrons. The central region is composed of borated paraffin which further moderates, capturing a significant portion of thermalized neutrons. The third region features a mixture of lead and borated paraffin and functions as a neutron-gamma shield. Because it is a feature of low power research and training reactors to expel heat by conduction, radiation, and convection, pelleting of the γ-ray shielding combined with intimate mixing with a moderator improves over-all conduction, raises heat capacity, and reduces the factors contributing to hot spots.

New ways of using a generally accepted shield material with increased advantage are possible. Handling massive lead elements and mastery of casting techniques are made eminently unnecessary, contributing to a lower cost "consumer" directed product. All such reactors, each representing sophisticated yet simple design, are meeting a wide demand in education here and abroad, with lead helping to satisfy cost and mobility requirements.

Lead pellets are also used by the Martin-Denver Co., contractor for the Titan ICBM, to simulate G-loads during static testing of fuel tanks. With the tanks in mockup location, liquid nitrogen is introduced to simulate a liquid oxygen ambient, whereupon lead shot is fed into the tanks at up to 1200 pounds per minute, flowing freely under the extreme cold.

In contrast is Bell Telephone Laboratories' solution to the problem of detecting aging—i.e., loss of polarity with repeated use, in ferroelectric crystals, and in computers with the help of red lead. A suspension of electrostatically charged particles of sulfur and red lead in hexane is applied to the face of a crystal to allow for assessment of polarity changes and a quick check of crystal uniformity. Applied separately, the sulfur is attracted to the negatively charged domains and red lead to positive domains, with the hexane evaporating to leave a bright pattern.

Cryogenics

Lead's low temperature behavior is receiving wide attention. Galvanomagnetic properties of lead sulfide, lead selenide, and lead telluride between 4.2° K and room temperature have been covered (7) in addition to thermal conductivity (13). K below the transition is insensitive to the amount and type of impurity (1 atom % bismuth, tin, or thallium), dependent upon specimen geometry for sufficiently thin specimens, and sensitive to plastic deformation. Lead takes on the character of dielectric crystals at approximately 1.4° K.

A redetermination of the absolute scale of thermoelectric power (4, 17) shows that small amounts of impurities do not cause notable variations as a function of composition or temperature, except for a 0.1 atom % cadmium alloy, 0.6 µv per °C in the range of 10° to 20° K. The shape of the Fermi surface was determined for single crystals between 1.2° and 4.2° K at fields up to 80 kg (9). Attention was paid to variations of periods (0.11 × 10⁻⁹ to 5.4 × 10⁻⁹ G⁻¹)

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with field orientation relative to crystal axes.

Study of the frequency dependence of electrical conductivity indicates independence of film resistance, thickness, degree of anneal, and alloying (3). Electron diffraction photos below 20° K were made of thin films and correlated with tempering above 20° K and approaching room temperature (3); grain size recovery runs parallel to recovery of resistivity and there is no connection between grain size after tempering and the transition. Volume changes and associated anisotropy in the area of the transition (5, 7, 14, 15) were noted, covering magnitudes approximating 2×10^{-9} cm. Differences in critical fields were established for different isotopic masses (6, 10), and ultrasonic sound attenuations for longitudinal and transverse waves show a considerable drop from values at room temperature at the transition (2, 12). There was also strong dependence of damping in the normal state on purity. The existence of a discontinuity in Young's modulus at the transition was noted (16).

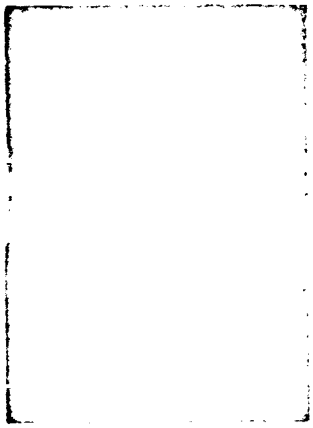
Acoustics

An excellent treatment is given physical and environmental factors in general by Mason (14), in a rather definitive treatment of both the practical and scientific.

Thermoelectricity

There are several developments. First there is the much-publicized Snap III atomic generator with its dependence on p- and n-type lead telluride. Much can be said in favor of the design slated to provide auxiliary electric power for space missile and vehicle systems. It won the 1958 Miniaturization Awards Contest. The basic features of lead telluride, most efficient in the range of 1000° to 1200° F., are being worked into prototype designs for direct conversion reactors.

Because the economic success of large scale power reactors rests with the maximum temperatures attainable, it was thought that lead compound thermoelectrics would be circumvented by the higher ambients of so-called mixed valent types. However, it now appears that a cascade principle is favored, which relegates position, either near or away from the heat source, according to maximum temperatures for efficient operation, thus achieving comparative efficiencies in tandem. Lead compound thermoelectrics should therefore figure in any eventuality. Apart from military use and envisioned use in atomic reactors, commercial use of lead telluride has its first example in Baso's Remotrol Appliance control system, completely flame-powered and independent of an outside



Symbols of ... in new arrangements—a lead ball suspended in space by the action of persuasive currents

power source. A lead telluride thermocouple provides as much as 140 mv. in combination with a valve, thermostat, and pilot burner. The control is adaptable to a wide variety of appliances and heating installations.

One major refrigerator manufacturer expects to have a thermoelectric in its line in about two years, but there is some leaning toward other materials. This is attributable to the background of work in this country with bismuth compounds. The Russians have passed the stage of commercial introduction with what appears to be sole dependence on lead telluride or selenide. However, the prime consideration seems to be the availability of tellurium or selenium in sufficient quantities and at costs to permit large scale introduction to consumer markets. The noiselessness, reliability, space advantage, and prestige of thermoelectric refrigerators and coolers are sufficient motivating influence to press industry's conclusion shortly. It is estimated that the average refrigerator will incorporate at least 50 pounds of thermoelectric materials.

In the realm of basic information, radiation damage in isotope- or reactor-powered systems is being ascertained. It is not known whether or not there is damage tending to subtract from efficiencies. Otherwise, New York University's thermoelectric materials and devices conference (5B) acquainted industry with current theory and the present state of the art, including a session devoted to lead telluride.

Thermoelectric Properties	Ref.
Alloy junctions in semiconducting devices	(8B)
Heat conductivity of PbTe	(1B)
Photoelectric properties of PbS single crystals [illegible] method	(2B)
Photoconductivity in chemically deposited PbSe films	(4B)
Antireflection coatings for indium antimonide and other semiconductor films	(5B)

Physical Metallurgy

In great part developments cover annealing behavior—i.e., recovery, recrystallization, and growth, and the morphology of solidification. Recent investigations divulge the structure of liquid lead (7C). Distribution of atoms is connected in a known way with packing in the solid state.

At the melting point, using thermal expansion data and x-rays, the vacancy concentration for lead is $<1.5 \times 10^{-4}$ and the corresponding energy of vacancy formation is >0.53 e.v. (4C).

The solid solution of gallium in lead rises from 0.02 weight % at 110° C. to 0.17 at the eutectic temperature (6C).

In the lead-silicon system, tensile and other mechanical properties at room temperature improved with silicon content up to 0.035% (9C).

After rolling in liquid air, chemical lead shows two recovery steps in the ranges −130° to −100° and −40° to −10° C., with the latter at −30° C. attributed to recrystallization (−100° C. for pure lead) (3C).

The morphologies of the growth forms associated with the solidification of lead indicate that solidification produces the (111) orientation in zone refined lead and with 5×10^{-4} weight % silver produces a (100) orientation (8C).

All this is seemingly important to studies of acoustic inspection technique refinements for lead nuclear shields. The effect on recrystallization of annealing conditions takes into account grain size, purity, and the influence of solid solution additives of thallium (3C), and grain boundary migration in high-purity lead and dilute lead-tin alloys shows tin to have a large effect on boundary migration (1C, 2C), the addition of 0.001 weight % tin reducing it by a factor of 10 with 0.015% the stopping it altogether.

A 200% increase in the density of lead has been achieved at pressures of 400,000 atm. or more, according to Soviet scientists. Studies at 5,000,000 atm. bear out findings in the United States that ultrahigh pressures can cause changes in electrical conductivity and light sensitivity as well as in such physical properties as density. Using explosives as a pressure source, the Soviets have been concentrating on studies of lead and bismuth because these metals show the greatest increase in density, over 200%, at pressures of 400,000 atm. or more. One interesting finding is that structural rearrangement at ultrahigh pressure is "unbelievably rapid," and a

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Lead alloy anodes are gaining widespread attention in corrosion circles while research portends greater developments

matter of slow adjustment to changed conditions.

Mechanical Metallurgy

Tests on the surface deformation differences between lead fatigued in air and in partial vacuum (5D) suggest that fatigue in vacuo causes more surface deformation, even though fatigue life is greater. Other tests show the dependence of microcreep properties on fatigue development (3D). With regard to compression data (4D), lead's resistance to deformation increases with alloying additions up to 3 atom % in the sequence copper, zinc, bismuth, tin, antimony, cadmium, lithium, tellurium, and silver. Copper and zinc are insoluble, with the remainder forming solid solutions. Compression strength decreases with increasing temperature but tellurium additions as low as 0.005% to a lead-1.5% antimony alloy minimize such behavior.

More specific work related to nuclear shield requirements treats the mechanicals of high purity lead, 0.058% copper-lead (7D), and 0.69% lithium-lead (1D). Work on the first two covers test temperatures of 100°, 175°, 250°, and 325° F., and corresponding tensile, compression, and creep properties. Work on the lead-lithium alloy covers alloying, casting, mechanical properties, and corrosion. The lead-lithium alloy has superior strength at room temperature but the lead-copper alloy has superior creep strength at 110° C. Corrosion resisting properties of the lithium alloy were inferior. It oxidizes more rapidly than pure lead in air (110° C.) and is attacked by water with a volume increase and cracking.

Finally, experimental and theoretical pressures and velocity fields for various lead extrusions were determined for the inverted extrusion of commercially pure lead through sharp-edged circular dies (6D). Extrusion ratios varied from 1.51 to 18.5 and billet reduction velocities between 0.03 and 0.24 inch per minute. Two lead solders (97.5 lead-2.5% silver and 95 lead-5% tin) have been found suitable for cold environments—i.e., liquefied gases at temperatures between −450° and −300° F. (2D). Based on tensile and impact properties, these alloys increase in both strength and ductility with falling temperature.

Nuclear Physics and Nucleonics

Shielding. Some attention is given to lead as a reflector in a report covering critical mass determinations of lead reflected systems (2E). A series of experimentally determined critical masses of cylindrical and spherical lead reflected or alloy systems is presented, and for reflected and unreflected ends in the cylindrical case. Experimental methods are described and details of the assembly machine are included.

A problematical treatment of eight lead and water shield configurations is given by the Oak Ridge National Laboratory (2E). The energy of the incident radiation, the angle of incidence, the thickness of the shield, and the percentage of lead preceding or following water were varied, with the source assumed to be a monodirectional beam having energies of 1, 3, 6, and 10 m.e.v. Incident angles were chosen to give slant thicknesses 1, 2, 3, and 4 times the normal. The infinite slabs had linear normal thicknesses of 1, 2, 4, and 6 free mean paths. Results included dose rates and energy flux throughout the slab and at the rear, dose-rate build-up factors, the heat deposited throughout the slab, and the energy (with angular distribution) reflected and transmitted.

Supplementary corrosion data (7E) cover the effects of lithium chromate as an aqueous corrosion inhibitor on carbon steel, copper, and lead tank materials. Standard coupons and some soldered couples were tested in lithium chromate solution (1630 p.p.m.) with different pH values. The solution is effective for at least 6 months.

For combined function, a lead-0.69% lithium alloy is promising (1D). Lithium because of its thermal neutron absorption properties is effective in suppressing secondary γ-production resulting from neutron interaction with lead, where the capture gammas produced are emitted instantaneously, unlike those liberated by decay. Use of such an alloy would tend to reduce secondary emission problems to consideration of hydrogenous materials in combination shield designs. This development might be pertinent to portable research and training reactors like the L-77, which utilizes pelletized lead intimately mixed with diphenyl and paraffin. The alloy's deficiency—corrosion in air or water—would be negated possibly by introduction into hydrocarbons, because lead is virtually inert to nonpolar hydrocarbons and only slightly attacked by the majority of polar substances. The further suggestion of lead-boron alloys was short-lived because of the development of data showing boron to be only slightly soluble. Attempts with powder metallurgy were not pursued to any length because of segregation. Otherwise use of the lithium alloy is recommended in a 15-ton spectrometer shield for the Bulk Shielding Facility at Oak Ridge.

Ashley (1E) puts forth a shield design simplification involving the conversion of spectral energy to an effective photon energy as an approach to shielding of mobile carriers for spent fuel elements. A fast determination of shield sufficiencies is made possible with the aid of suitable graphs. With an effective photon energy figure for different lead thicknesses and corresponding linear absorption coefficients, flux-dose conversion factors, and build-up factors, the effectiveness of lead shields can be computed with fair accuracy.

Power Sources. In addition to the evaluation of the Snap III generator and the recognition of the possibilities for direct power conversion reactors via the cascade principle, both involving the use of thermoelectrics, there is the suggestion of a photogalvanic system—i.e., a radiation source coupled with the electrochemical features of the lead-acid cell. It is based on the observation that copious quantities of red lead and litharge were produced in a reactor irradiation can containing moisture (4E–6E). Lead reacted with moisture under irradiation in the manner of reactions induced by applied potentials. It is reasonable to imagine that radiation flux-pH constitution diagrams for the lead-water or sulfuric acid system can be developed which parallel electrochemical counterparts and that a dischargeable peroxide can be produced. For all intents the system would be self-shielding with continuous irradiation, short wave length, electromagnetic or particular, used in place of or in addition to the charge cycle.

Corrosion

Waters. The superiority of lead over 15 other metals and alloys was reported in an article covering corrosion conditions in the waters of Port Hueneme Harbor, Calif. (3F). Conducted by the Naval Civil Engineering Research and Evaluation Laboratory, the study enables a more exact selection of construction materials.

In duct systems where the picture changes because of extraneous influences, —i.e., stray current electrolysis—protection by impressed current cathodization becomes necessary. Cable sheath can be protected in a constant current system even in the presence of high salt content and pH (4F). The behavior is substantially the same as that inferred from the Pourbaix diagram for lead.

Anodes. Rather than receiving protection, lead appears promising as an inert impressed current cathodic protection anode. Prompted by a number of developments in connection with success in the protection of ship hulls, additional ship, bridge, pier, and heat exchanger trials are under way in salt, brackish, and fresh (industrial) waters. There has been talk of extension of such anodes to soils having low resistivities—for example, salted mud and sands where parallel performance is indicated.

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Underlying developments include first, the work of the Naval Research Establishment, Nova Scotia, in evaluating cast lead-2 % silver alloy anodes on a high speed Royal Canadian Navy destroyer over a two-year period (*1F, 4F*). Since installing them in 1956, the ship has been docked twice and the hull was found to be rust-free and in excellent general condition. Economic and functional comparison with more conventional types has shown lead anodes to be superior to graphite, steel, platinum, or galvanic magnesium, with 10+ years' life envisioned. Concurrent work in England explores the theory and practice of lead anode systems with emphasis on practical designs and their estimated performance (*10F*). Fundamental discussion of the nature and kinetics of formation of the essential peroxide is included.

The National Association of Corrosion Engineers has concurred with industry's view regarding the practicability of lead anodes by placing their consideration on the agenda of committees T-3G-4 "Cathodic Protection of Hull Bottoms of Ships" and T-2B "Pipe-Line Corrosion Anodes for Impressed Currents." And the Navy has been investigating a series of lead alloys. A naval scientist reports that preliminary data are "promising."

In the realm of research, interesting developments apparently lift the lid on current densities heretofore considered practical, at least in so far as behavior in waters is concerned. According to the recent work of Shreir and Weinraub in England (*14F*), the insertion of a very small microelectrode or "tack" of platinum into the anode surface prior to polarization results in the formation of an adherent coating of peroxide and maintenance of the layer at remarkable current densities. Although several factors may give rise to this effect, one explanation considers platinum's function as an internal "potentiostat" and another, the view that platinum's sole function is to make electrical contact between lead and peroxide (*13F*). There may be an additional consideration in behavior in the standby or disconnected state which appreciates the existence of an electrochemical couple between the peroxide and platinum and the possible consequence of discharge or loss of peroxide in the neighborhood of the tack if not over the anode surface.

Lead-0.69% bismuth alloy offers distinct advantage as a combination neutron-γ-ray shield but not without contaminant. It corrodes with great rapidity in water or in air at rates above those for pure lead. Because bismuth leads the list with regard to e.m.f. it is reasonable to suggest that lead has been less noble via alloying or possibly more electropositive than iron or steel, implying possibilities for a sacrificial anode and the invasion of markets pertinent to common galvanic types. It is doubtful if anyone has given more than academic attention to the feasibility of decreasing lead's corrosion resistance or of increasing e.m.f. drastically by alloying. Such an attitude might be reexamined, if not the semantics of properties in general.

Chemical. Anode compositions and applications in the plating industry were treated (*7F*), with emphasis on the significance of reactions in plating processes which parallel the importance of the deposition mechanism.

Chemical corrosion by bromine has been shown to be due to the presence of hydrobromic acid (*2F*). Prevention is discussed in terms of additions of oxidizing agents, especially lead peroxide, or by anodic oxidation of lead surfaces.

Corrosion rates in dilute aqueous organic acids, 0.0001 to 0.1*N* acetic, propionic, butyric, succinic and lactic, are very similar for equinormal solutions (*5F*). Passivation was evident in pyruvic acid, however. Attack was intercrystalline, with products covering the transition from basic carbonates deficient in carbon dioxide to the basic carbonate $2PbCO_3 \cdot Pb(OH)_2$. The purpose of such studies was to obtain data on corrosion of lead sheaths for electric and telecommunications underground cables wrapped in bitumen reinforced with cellulosic materials.

Chemical Construction. Two articles put forth the interesting points that the main objective in every case must be the provision of a membrane with complete impermeability and adequate chemical resistance (*9F*) and that there is still no accepted technique of lead fabrication (*12F*). Such comments indicate the existence of room for improvement in design of lead-lined equipment. Another article covers the properties and uses of lead and its alloys, including specifications (*6F*).

In the area of improvements, the latest factors and experiences common to lead-acid brick construction are discussed (in extracted form) (*11F*). The article also refers to a plausible solution to the problem of locating in-service leaks quickly with the aid of radioisotopes. A typical example of successful employment of a lead-acid brick system (*15F*) refers to a lead-lined steel, acid-proof brick, carbon brick mortar lining in a Moa Bay nickel extraction plant. Interesting comments are made regarding the durability of homogeneous bonded linings which led to the use of supplemental brick.

Chemistry

A new ultraviolet luminescent material developed by North American Philips has received a patent (*3G*). The material is essentially a lead-activated barium-zinc silicate which emits ultraviolet in the range 2900 to 3500 A. with peak emission at about 3000 A. In work sponsored by the Department of Agriculture, lead hydroxide has been found useful in increasing durability of cotton. Basic lead carbonate and lead monohydrogen phosphate, pigments for decorative and optical effects, are given deserved attention in an article on improved pearl essences (*1G*). It traces the history of synthetic pearl essence (as achieved with lead compounds) with interesting comments on morphology and lists several applicable patents. Another article goes into properties of pearl pigments in more detail (*2G*).

Basic lead carbonate may also have a role in finding the "selective black," a problem very close to proponents of solar energy conversion. The fact that the properties of the material are in accord with the requirement of being black in the infrared and "white" in the ultraviolet has opened a line of inquiry at New York University into the nature of heat emission-absorption of lead compounds and how it might be controlled. Outside the earth's atmosphere 50% of cosmic radiation is infrared up to 2.5 microns and represents 6 to 13 watts per sq. foot of tappable power (based on solar constants). It is likely that a solar collector having the attributes of selectivity with good heat stability when coupled with photovoltaic or thermoelectric generators would be most efficient without the need for glass shields. Considering space probes and satellites, selectivity enables a surface to accept infrared and not remit to any great extent, thus lowering collector efficiencies, as would not be the case if the energetic wave lengths (ultraviolet) were absorbed in black body fashion. Reemission would occur and heavy shields become necessary (the shields cut down re-emission). Interesting in itself is the morphology of basic lead carbonate. It can be an amorphous conglomerate, tabular or lenticular, and consequently orientable. This behavior may represent an additional contribution to collector efficiencies, as reflectivity becomes important in materials having multiple boundaries and crystal interfaces. An orientable pigment could effect a high degree of regularity between particles, thus further approaching the perfect collector with high absorption and transmissibility. Single crystals exhibit the latter property, but the prospects of producing economically large crystal masses, sheets, or coatings make their use improbable.

Heat dissipation in space vehicles and associated equipment must be handled by emission or some evaporative technique since convection is precluded in airless environs. Therefore the emissivity of basic lead carbonate might also deserve

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attraction. Involvement with Rochelle salts, a common storage medium for solar heating installations, might also be indicated. A white lead surfaced mass would considerably enhance heat capacity characteristics by the addition of excellent absorption and emission properties.

Ceramics

Volatility of lead silicate series (2H), high dielectric permeability, and puncture voltages in a group of boron-lead-titania glasses (4H), the lowest dielectric loss characteristics yet noted in lead fluxed wollastonite bodies (3H), and a patent covering leaded vitreous low temperature coatings for steel (1H) comprise the significant developments in this area.

Publications

The economic picture was covered in an annual review article (4J), and the "Yearbook of the American Bureau of Metal Statistics" (2J). The technical picture was covered in INDUSTRIAL AND ENGINEERING CHEMISTRY (3J).

The Lead Industries Association publishes quarterly the magazine *Lead* (1J), which covered many of the developments herein referred to and a variety of others. Mason's "Physical Acoustics and the Properties of Solids" (1A) is a source of practical and theoretical data on lead's acoustical behavior under the influence of various physical and environmental conditions.

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