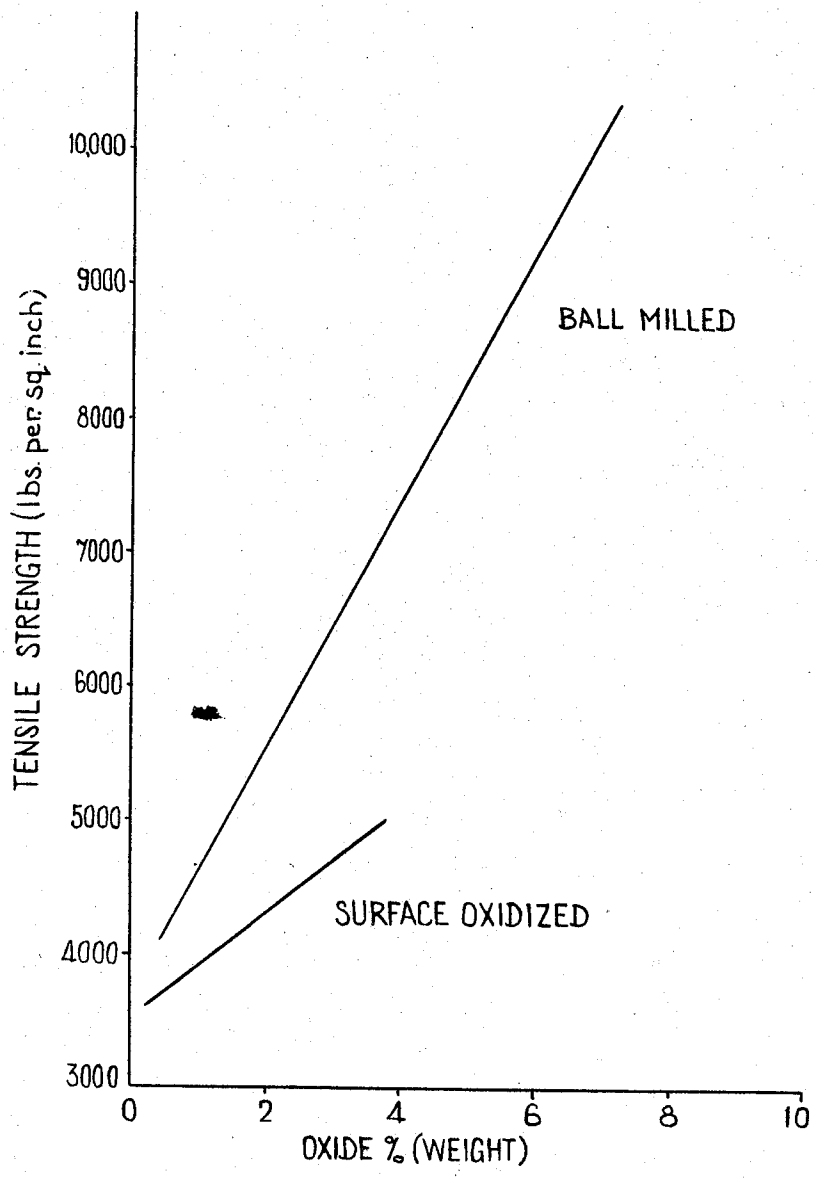
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DISPERSION STRENGTHENING OF LEAD

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DISPERSION STRENGTHENING OF LEAD

David Henry Roberts, Harlow, England, assignor, by mesne assignments, to St. Joseph Lead Company, New York, N.Y., a corporation of New York

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This invention is concerned with the production of hardened lead for industrial purposes.

The special properties of lead make it valuable in the electrical industry for cable sheathing and the construction of batteries. It is also useful for radiation shielding. Further, its resistance to corrosion makes it valuable in the chemical industry. Its disadvantage, however, is its very low tensile strength, which means that a structure made of ordinary lead has to be supported by parts made of another metal e.g. copper or steel, otherwise the structure would suffer serious distortion from the effects of its own weight in the course of time. It is therefore desirable to be able to produce lead in a strengthened form.

A method has already been proposed for forming sheets of dispersion-strengthened lead from lead powder by extruding the lead powder through a die and then rolling the resultant bar along and across the extrusion direction. This procedure breaks up the oxide coatings on the lead particles, distributes the resultant fragments of oxide throughout the lead, and causes the lead particles to become welded together.

The expression "oxide coatings" in the foregoing means coatings formed on the surfaces of the lead particles as a result of the exposure of the powder to the action of the atmosphere. Throughout the remainder of this specification the expression is to be taken as meaning coatings either formed in the same way on the lead particles by the action of the atmosphere on them or else formed on particles by the actions, simultaneously or in succession, both of the atmosphere and one or more other agents which tend to cause oxidation of the lead. Such coatings consist substantially of one or more of the oxides of lead and are commonly called "oxide" coatings but other materials, such as the carbonates and hydroxides of lead may also be present in them. Similarly, "oxide" particles through the remainder of this specification is to be taken as meaning particles of the "oxide" coatings as described above.

We have discovered that dispersion-strengthened lead may be obtained from partly oxidised lead powder by simply extruding it, provided that the extrusion ratio is such that the process of extrusion breaks up the oxide coatings on the lead particles, distributes the resultant fragments of oxide throughout the lead and causes the lead particles to become welded together. The degree of deformation to which the lead particles need to be subjected during extrusion in order to produce a dispersion strengthened extrusion product depends mainly upon the mean thickness of the oxide films on the particles. The proportion of dispersed oxide in the extrusion product, however, and hence the strength of this product will depend both upon the means thickness of the oxide films on the particles and upon their mean initial size. The larger the mean particle size for a given film thickness, the smaller the proportion of dispersed oxide in the extrusion product and conversely. In a typical procedure for the fabrication of a bar of dispersion strengthened lead, lead particles which will pass through a 300 B.S. Sieve and which have a mean oxide film thickness of 0.3μ are extruded at an extrusion ratio of 20:1. No subsequent rolling of the extrusion product will be necessary in order to complete the dispersion-strengthening process.

We have also discovered that the strength of a dispersion-strengthened lead article formed by simply extruding lead powder can be still further increased by subjecting the lead powder to be extruded to a process whereby the lead oxide content of the powder is increased without any significant increase in the thickness of the oxide coatings on the surfaces of the individual lead particles.

The present invention provides a method for producing dispersion-strengthened lead from lead powder, which comprises subjecting the lead powder to a mechanical process whereby the oxide coatings on the lead particles are broken up and the resultant oxide fragments forced into the lead particles themselves, while further oxidation takes place on fresh surfaces, and thereafter extruding the lead powder to produce solid lead containing a dispersion of oxide.

Typically the mechanical process comprises milling the lead powder in a vibration ball mill and this process should be carried out under such conditions that the lead exposed as a result of the break-up of the oxide coatings will readily re-oxidise.

The purpose of the initial mechanical treatment is to increase the oxide content of the lead powder in such a way that the proportion of dispersed oxide in the extruded lead is increased. This results in an increase in the strength of the dispersion-strengthened lead. Experiments have shown that there is a substantially linear relationship between the ultimate tensile strength (U.T.S.) of the extruded lead and its content of dispersed oxide, so that to achieve the highest strengths high oxide contents are necessary. Further, it is found that, for a given oxide content, dispersion strengthened lead formed by extruding lead powder which has been subjected to a mechanical process of the type described above, is stronger and more ductile than dispersion strengthened lead formed by simply extruding the surface oxidised powder. This is illustrated in the graph shown in the accompanying drawing.

Atempts to increase substantially the dispersed oxide content of the extruded lead, and thereby to increase its strength, by simply increasing the thickness of the oxide coating on the lead particles by heating the powder to be extruded, have not proved successful. This is because the thickened coatings are too tough to be broken up properly by the extrusion process with the result that unbroken networks of oxide are formed in the extrusion product. Consequently, oxide dispersion and particle bonding in the extrusion product are poor.

Preliminary ball-milling of the particles avoids this difficulty by breaking up the oxide coatings initially present on the particles, and punching the oxide fragments into them. The milling also exposes clean lead on which, provided oxygen is present, a new thin layer of oxide is formed and then in turn broken up and punched into the lead and so on. In the presence of oxygen the process is thus a continuous one. The evidence for this is that when lead powder is treated in a closed air-tight ball mill, large agglomerates are formed due to the cold welding of the clean oxide-free surfaces of the lead. Further, experiments on ball milling lead powder in an inert gas have shown that no oxidation occurs and material extruded from this powder has only a very slight increase in strength. This slight increase probably results from a better dispersion in the extrusion product of the oxide initially present on the particles due to the fragmentation of the oxide coatings in the ball mill.

Experience with the ball-milling process has shown that

(a) the higher the proportion of the balls in the charge the faster the oxidation,

(b) the higher the temperature, the faster and more extensive the oxidation, and

(c) if continuous oxidation is possible then there is no agglomeration of the powder particles due to cold welding.

For the initial ball-milling step a vibration ball mill is preferred since it gives more severe working of the lead powder than a simple tumble ball mill. As shown above, the particles of lead powder treated in this way will contain oxide particles within them, in addition to a surface coating (due in part to oxidation during ball-milling) and it is found that the oxide dispersion in material extruded from this powder is very good. It has so far only proved possible to extrude lead powder containing up to 7% by weight of oxide and thereby to produce a consolidated extrusion product, when the extrusion is carried out at room temperature. It is possible however successfully to extrude powders containing larger proportions of oxide by raising the extrusion temperature.

The practice of the invention will be better understood from the following description of the typical conditions for the production of a bar of dispersion strengthened lead.

(a) 1 volume of commercial air atomised lead powder which will pass a 300 B.S. Sieve (particle sizes 53μ down to dust) is ball-milled at about 70° C. for 6 hours in a vibration ball mill containing 1 volume of ½" diameter steel balls. By this is meant that, for example, a beakerful of powder and a beakerful of balls are placed in the mill. In the case of the balls, therefore, it is the "gross" volume rather than the actual volume of balls themselves we are concerned with, in view of the large spaces between them. The design of the ball-mill must be such that the oxygen used up during the ball-milling is continually replaced.

(b) The ball-milled powder is extruded at 25° C. through a 0.1" diameter extrusion die, the extrusion ratio being 40:1. (This is the ratio of the cross-sectional area of the chamber to that of the extrusion product.)

Some typical results are given in Tables 1 and 2.

TABLE 1.—OXIDATION RATE FOR BALL MILLED LEAD POWDER

Conditions for milling	Milling time, hours	Wt. percent Oxide
Charge 1 volume Lead powder to 1 Volume of ½" diameter Steel balls Total charge 300 gms	0	0.9
	1	1.7
	2	2.4
	3	3.1
	4	3.8
Atmosphere: Oxygen	5	4.8
	6	5.8
	7	6.8
	8	7.7

TABLE 2.—STRESS-RUPTURE PROPERTIES OF LEAD AND DISPERSION-STRENGTHENED LEAD

Material	Applied Stress, lb./sq. in.	Time to Fracture, min.	Elongation at Fracture, percent on 1 inch
Lead (Cast and Wrought)	1,300	200	20
	1,400	30	25
	1,500	8	29.5
Dispersion-Strengthened Lead (1.5% oxide)	2,000	(1)	
	3,000	10,080	
	3,850	39	6.7
	4,000	34	7.9

[1] No failures after 1,000 hours.

The present invention is not confined to the production of dispersion-strengthened lead from lead powder. It may equally be employed for the production of dispersion-strengthened lead alloy from a powdered alloy. It is found in general however, that particles of a lead alloy oxidise less readily than lead particles so that in some cases lead alloy particles need to be subjected to mechanical treatment for a longer time than lead particles, or under conditions which increase the rate of oxidation of the alloy particles, or both. The rate of oxidation of the particles may be increased by ball milling them in oxygen at a temperature above room temperature. A typical alloy which may be hardened by the methods of this invention is the so-called cable sheathing alloy B containing 98.2% lead and 0.8% antimony.

What I claim is:

1. A method for producing dispersion-strengthened lead from lead powder, which comprises subjecting the lead powder to a mechanical process whereby the oxide coatings on the lead particles are broken up and the resultant oxide fragments forced into the lead particles themselves, while further oxidation takes place on fresh surfaces, and thereafter extruding the lead powder to produce solid lead containing a dispersion of oxide.

2. A method according to claim 1, in which the lead powder is subjected to milling in a vibration ball mill.

3. A method according to claim 2, in which arrangements are made for continual changing of the air or oxygen in the ball mill to promote oxidation of the lead powder.

4. A method according to claim 1, in which a dispersion-strengthened lead alloy is produced from a lead alloy powder.

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JOHN F. CAMPBELL, *Primary Examiner.*

P. M. COHEN, *Assistant Examiner.*