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A Thesis
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INTRODUCTION

From the standpoint of its practical usefulness, the most important characteristics of metallic lead are its cheapness, resistance to corrosion, plasticity, high specific gravity, low melting point, and its ability to form alloys in which some properties are modified by the addition of other elements, while other properties remain the same.

Statistics show that the annual production of lead is about the same as that of copper, but its applications are not so striking in comparison with copper. This is mainly due to the fact that a large proportion of lead produced is used for compounds, and most of the remainder is buried in cable-sheaths, pipes, and storage batteries. Lead is also an important alloying element; it is used in type metals, bearing alloys, solders, etc.

The main reason why lead is unsuitable for many uses is that it cannot be hardened by cold-work. All work done on lead at room temperature is hot-work, since the recrystallization of lead proceeds fairly rapidly at room temperature. Therefore, strain-hardening takes place only to a small extent and is not permanent at room temperature; in other words, as soon as strain-hardening takes place, recrystallization destroys the hardness.

It must be noted here, although pure lead recrystallizes very rapidly at room temperature, the time required for this to take place increases when impurities are present.

Since the recrystallization temperature of lead is below room temperature, it is very susceptible to recrystallization and grain growth at ordinary temperatures. It is evident that a structure containing grains which vary widely in size does not behave uniformly under straining and corroding conditions. Consequently, lead shows inter-crystalline fractures when subjected to stress.

Recently the mechanical properties of lead have received a considerable amount of attention because of cable-sheaths under very low stresses. Several alloying elements are available for adding to lead to increase its strength without destroying the plasticity that makes the extrusion and subsequent manipulation of pipes and cable-sheaths possible.

Singleton and Jones of England, and the British Non-Ferrous Mètals Research Association have studied the effect of the addition of small amounts of other metals to lead. These investigators have found that the addition of about 0.07 to 0.1 per cent tellurium to lead increased the corrosion resistance to concentrated sulphuric acid, and improved the mechanical properties. The temperature of recrystallization is increased so that most of the increase in strength produced by cold-work is permanent at ordinary temperatures. For instance, the tensile strength of tellurium-lead may be increased to 4000 lbs. per sq. in. by cold-working the tensile strength of pure lead being about 2000 lbs. per sq. in. Its elongation is greater due to an increase in the uniform extension, its endurance limit is nearly three times that of ordinary lead. Furthermore, they found that the work hardening can be regulated so that a rolled sheet with a wide range of physical properties can be produced. As a result of this work, lead with less than 0.1 per cent tellurium has been patented in England by Singleton and Jones, and is being sold under the trade name of "Telledium." It is used extensively for sheet, pipe, and cable-sheathing.

It was the purpose of this thesis to study some effects of the addition of small amounts of tellurium to lead alloys. Of the lead-base alloys, the binary alloy systems that are important in connection with industrial alloys are those formed by lead and the metals antimony, tin, bismuth, and cadmium.

Because of the shortness of the time and for the sake of convenience, the effect of the addition of tellurium was studied on the alloys of lead-antimony and lead-tin containing 10 per cent antimony and tin respectfully. At this point it is therefore desirable to consider the constitution and some properties of these alloys.

Constitution and Properties of Pb-Te Alloys

Lead and tellurium form a series of alloys completely soluble in the liquid state and completely insoluble in the solid state. They form a chemical compound of lead telluride PbTe, which forms a eutectic temperature of 412 degrees centigrade. No eutectic is formed with lead telluride and lead.

When tellurium is added to lead, the melting point of lead is raised until the alloy is all PbTe, then as the tellurium content increases, the melting point is lowered until the eutectic of PbTe-Te is formed. The addition of lead to tellurium decreases the melting point until the same eutectic is reached.

As the tellurium content increases, the alloy becomes harder, and finally brittle.

Constitution and Properties of Pb-Sb Alloys

Lead and antimony form a series of alloys completely soluble in the liquid state and partially soluble in the solid state, consequently, solid solutions are formed to the extent of a few per cent at both the lead and antimony ends of the system. These two solid solutions form a eutectic at about 12.5 per cent antimony with a eutectic temperature of 247 degrees centigrade.

The solubility of antimony in lead decreases according to the equilibrium diagram, from 2.45 per cent at eutectic temperature to about 0.20 per cent at 20 degrees centigrade. This decrease makes possible the dispersion hardening of lead rich alloys by suitable treatment, namely, by heating the alloy to a temperature above the solubility curve and cooling rapidly to a temperature below this curve and aging. The degree and rate of hardening becomes greater with increase in antimony content, with increase of impurities, and with increase in the rate of cooling. However, the aging may include over-aging i.e., the antimony which is precipitated in a highly dispersed state gradually agglomerates during continued aging and thus the alloys gradually soften. Therefore, the lead rich alloys are of greatest practical importance.

Lead-antimony alloys containing up to 12 per cent antimony have good corrosion resistance, good mechanical strength, and good casting properties. Therefore, they are used in chemical industry, in machinery, and apparatuses. The effect of antimony in lead in increasing the corrosion resistance is probably due to its position in electro-chemical series. The changes in mechanical properties are due to the hardness and brittlness of antimony.

Lead with 1 per cent antimony is used for cable-sheaths, and lead with 7-12 per cent antimony is used for storage batteries.

Constitution and Properties of Pb-Sn Alloys

As with antimony, lead forms a complete series of alloys with tin completely soluble in the liquid state and partially soluble in the solid state. The solid solutions form a eutectic with 62 per cent tin at eutectic temperature of 183 degrees centigrade. The solubility of tin in lead decreases rapidly from 19.5 per cent at eutectic temperature to about 13 per cent at 155 degrees centigrade. The tensile strength increases as the tin content is increased from zero to about 65 per cent.

As tin is a plastic metal like lead, the alloys throughout the entire system are plastic and readily workable. They can be rolled, extruded, and stamped, even in the cold state. The alloys have also a notable corrosion resistance.

Lead containing 1-3 per cent tin is used for cable-sheaths, and in manufacturing lead foil. Lead containing 3-8 per cent tin is used for sheet and pipe in chemical plants. Terne plate is made by dipping steel in an alloy with 15-25 per cent tin.

Experimental

The lead used for making the alloys was high grade test lead. The tellurium, antimony, and tin were chemically pure.

Preparation of Master Alloy: The required amounts of lead and tellurium to make a 10 per cent tellurium alloy after being mixed thoroughly were placed in a graphite crucible. To prevent oxidation the charge was covered with powdered charcoal and the crucible was kept covered. The crucible was placed in an electric furnace. When the charge was molten, the cover was removed and the charge stirred with a carbon rod to insure a homogeneous mixture. The alloy was then cast in a small fire clay mold prepared by hand in the laboratory. From this master alloy three series of alloys were prepared, carrying out the same procedure as is making the master alloy. The alloys made are of the following composition:

No.			
1.	Pb	Pb 1%Te	Pb 0.1%Te
2.	Pb 10%Sb	Pb 10%Sb 1%Te	Pb 10%Sb 0.1%Te
3.	Pb 10%Sn	Pb 10%Sn 1%Te	Pb 10%Sn 0.1%Te

Preparation of Specimens for Microscopic Examination:

Because of the softness of lead and its alloys the preparation of specimens for microscopic examination is quite difficult. The finished surface is usually too heavily scratched, and the surface metal flows forming a layer of cold-worked metal which masks the true structure. Usually, these difficulties were overcome by deeply etching as to get below the surface imperfection. However, it is most desirable to finish the sample with a minimum of scratches and flowed metal as to permit light etching. There are a few basic methods for overcoming these difficulties. The special technique, suggested by Metals Handbook for polishing lead and its alloys, was applied which can be carried out with ordinary polishing equipment. The surface of the specimens were smoothed with a file and then ground on No. 1, 0, 00, 000, and 0000 emery papers which have been previously smeared with a concentrated solution of paraffin in kerosene. This prevents the particles of alloy from adhering to the paper and forming a glaze which drags and distorts the surface of the alloy.

Wet grinding was done on a broadcloth smeared with plenty of soap using an abrasive of levigated alumina. At this stage a black smudge often appears on the surface of the specimen which can be eliminated by keeping the cloth thoroughly wet by alumina and soap. The final step in polishing was done on a felt wheel using distilled water.

The surfaces were etched with a solution of 3 parts glacial acetic acid and 1 part hydrogen peroxide, and some with dilute hot nitric acid.

As a matter of fact even a well prepared surface will be seldom free from a film of cold-worked metal. Therefore, alternate polishing and etching methods were used in order to show the true structure of the alloy. By doing this, the film of distorted metal is gradually removed by the etchant, while the succeeding light polish prevents a long continued attack.

Fig. 1 Pb - 10%Sb, as cast, etched with acetic acid - H_2O_2. 100x

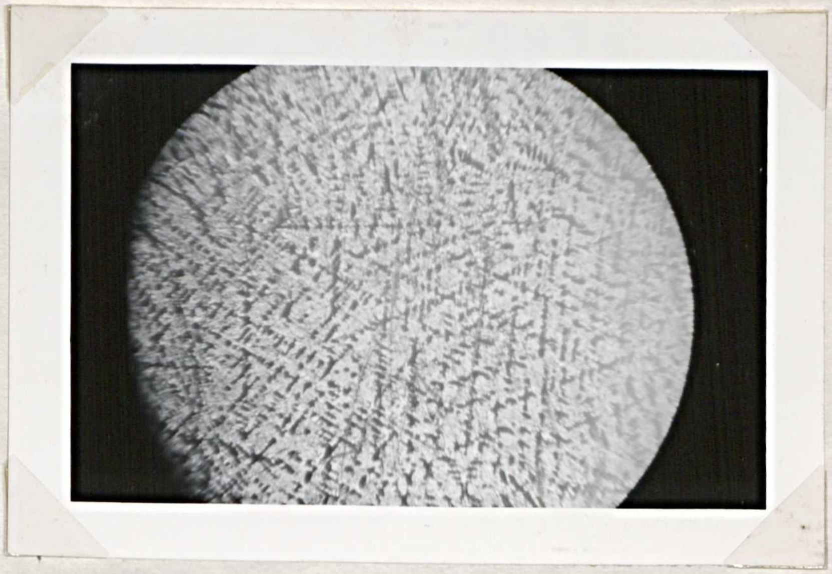

Fig. 2 Pb - 10%Sb - 1%Te as cast, etched with acetic acid - H_2O_2. 100x

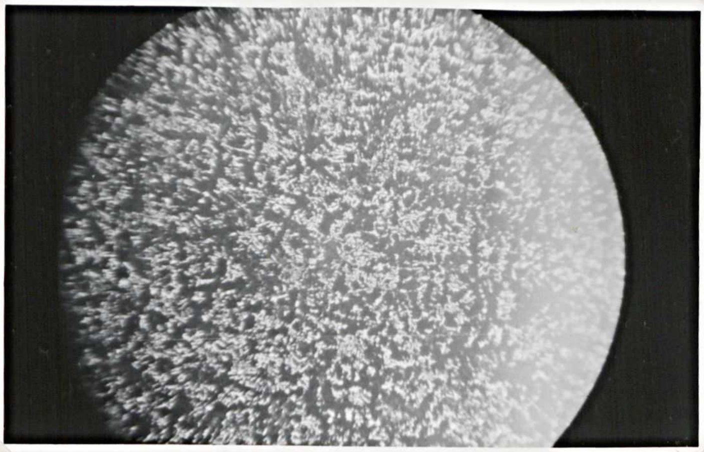

Fig. 3 Pb - 10%Sb - 0.1%Te, as cast, etched with acetic acid - H_2O_2. 100x

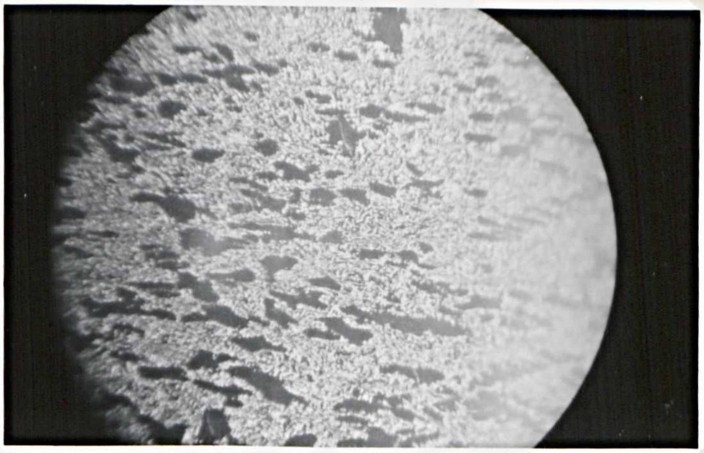

Fig. 4 Pb - 10%Sb, cold-rolled, etched with acetic acid - H_2O_2. 100x

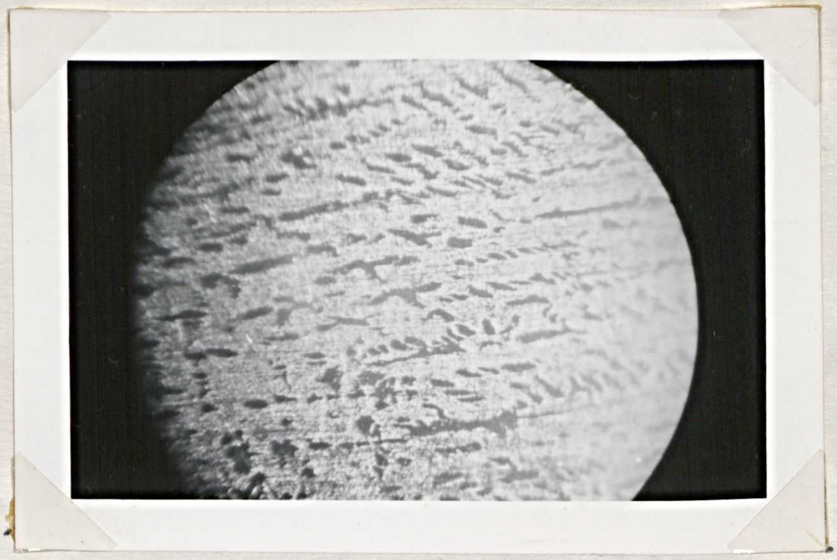

Fig. 5 Pb - 10%Sb - 1%Te, cold-rolled,
etched with acetic acid - H_2O_2.
100x

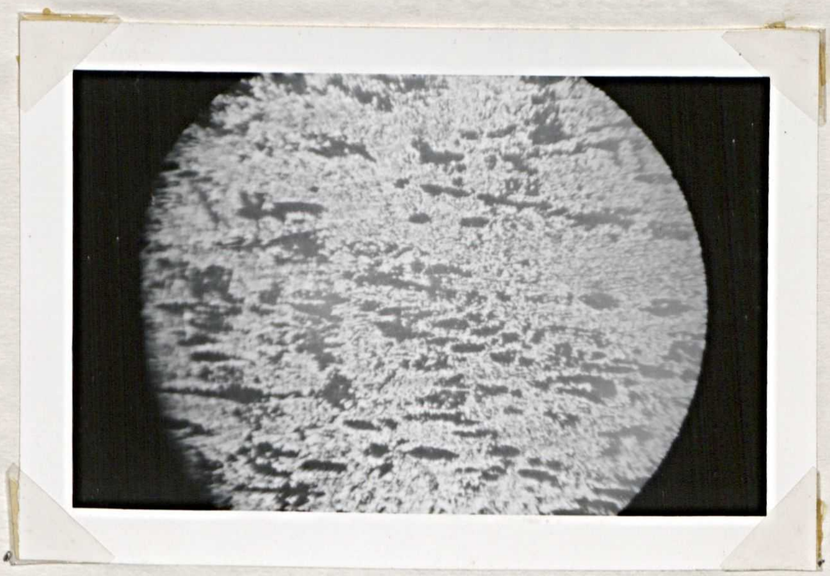

Fig. 6 Pb - 10%Sb - 0.1%Te, cold-rolled,
etched with acetic acid - H_2O_2.
100x

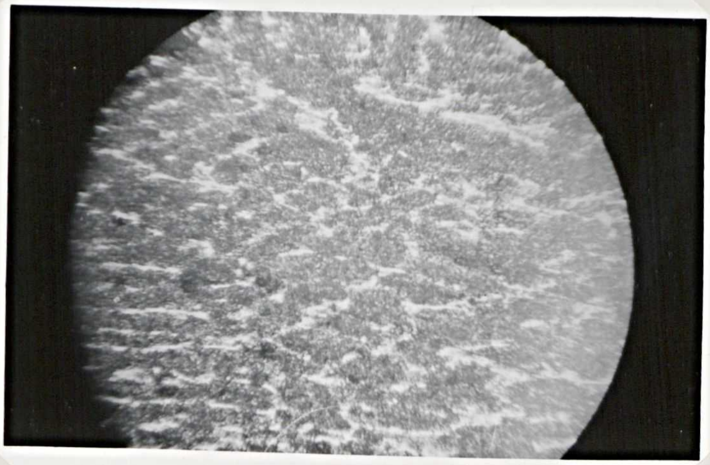

Fig. 7 Pb - 10%Sn, cold-rolled,
etched with $AgNO_3$. 100x

Corrosion Resistance to Sulphuric Acid:

Tests were made for corrosion resistance against sulphuric acid on lead and its alloys as such and containing 1 and 0.1 per cent tellurium. The lead and lead-antimony and lead-tin alloys containing 0.1 per cent tellurium were found to be more corrosion resistant to sulphuric acid. Cold rolled sheets having approximately the same surface area after being annealed, were weighed and exposed to the action of hot concentrated sulphuric acid for a short time. The sheets were then removed, washed, dried and re-weighed. The weight losses were as follows:

Pb	12.3	per	cent
Pb 0.1%Te	4.4	"	"
Pb 10%Sb	5.9	"	"
Pb 10%Sb 0.1%Te	3.3	"	"
Pb 10%Sn	4.2	"	"
Pb 10%Sn 0.1%Te	3.0	"	"

Note: After the exposure the sulphuric acid had a pink color, showing the presence of tellurium.

CONCLUSION

1. The binary lead-antimony and lead-tin alloys containing small amounts of tellurium show a structure in which the matrix of lead around the eutectic is more evenly dispersed.

2. The addition of small amounts of tellurium to the binary lead alloys improves their physical properties.

3. The addition of 0.1 per cent tellurium to the binary lead alloys increases the corrosion resistance to concentrated sulphuric acid.

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