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Alloys of Lead and Tellurium

Ettore A. Peretti

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ALLOYS OF LEAD
AND TELLURIUM

by
ETTORE A. PERETTI

A Thesis
Submitted to the Department of Metallurgy
in Partial Fulfillment of the
Requirement for the Degree of
Bachelor of Science in Metallurgical Engineering

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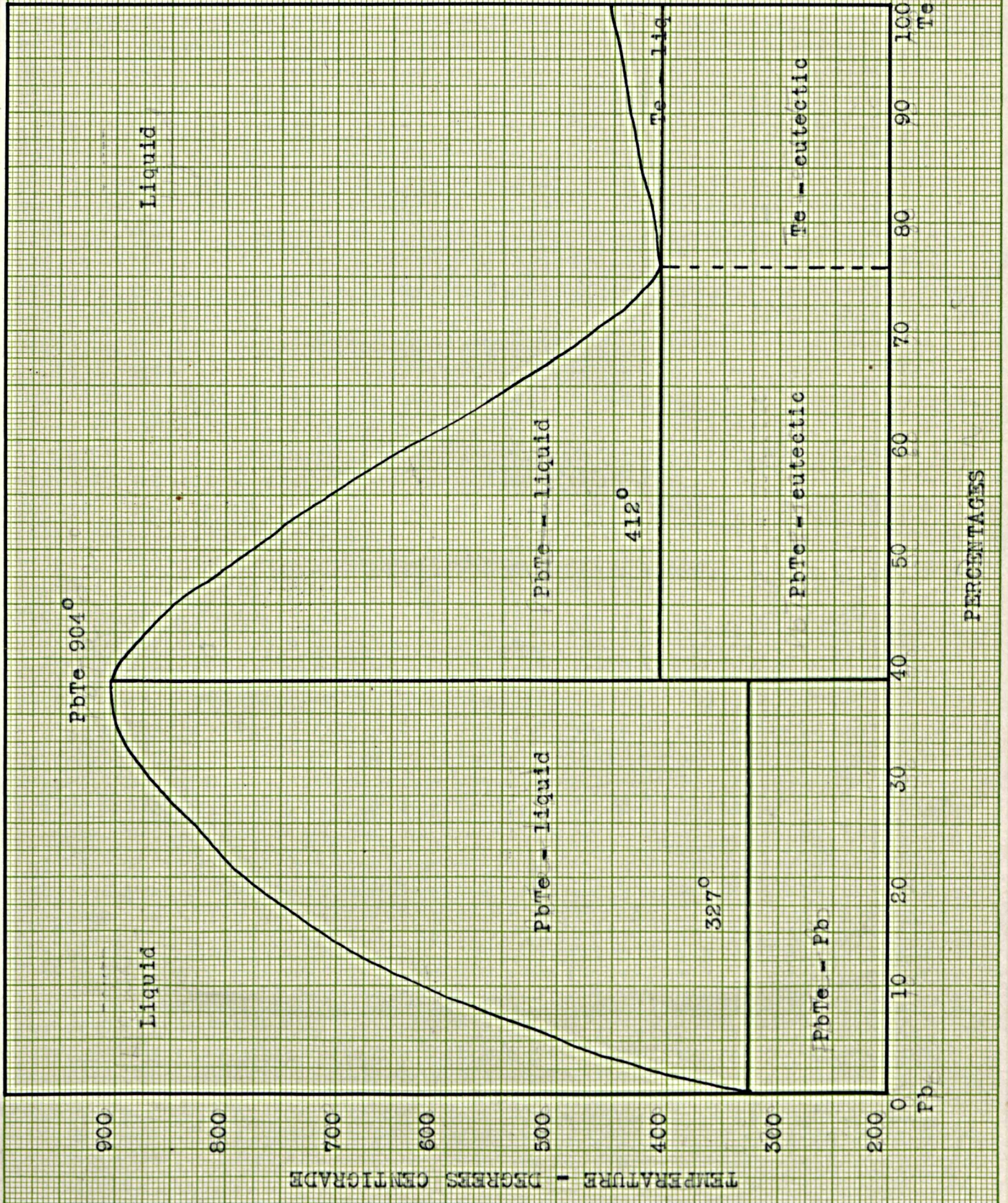
INTRODUCTION

Prior to the last few years little practical use was made of the element tellurium, which is obtained from gold and silver tellurides and from the slimes of electrolytic copper refineries. Lately, however, more study has been made of its properties when alloyed with other metals. It was the purpose of this thesis to study the effects of the addition of tellurium to lead, particularly in small amounts.

The equilibrium diagram for lead and tellurium was worked out by Henry Fay and C.B. Gillson in 1902⁽¹⁾. As shown by figure 1, when tellurium is added to lead, the compound PbTe is formed, which raises the melting point of the lead. As the tellurium content increases, the melting point is raised until the alloy is all PbTe, when a greater addition of lead lowers the melting point. The addition of lead to tellurium decreases the melting point until the eutectic of tellurium and lead telluride is reached at 24% lead. The alloys are completely soluble in the liquid state and completely insoluble in the solid state. No eutectic is given as existing between lead telluride and lead.

As the tellurium content increases the alloys become harder, and finally brittle.

Figure 1.



KEUFFEL & ESSER CO., N. Y. NO. 358-14
Millimeters, 10th lines heavy.

EXPERIMENTATION

The lead used in making the alloys was high grade test lead. The tellurium was secured from the Raritan Copper Works of Perth Amboy, New Jersey.

The required amounts of tellurium and lead to make a 10% tellurium alloy and a 90% tellurium alloy were placed in graphite crucibles in an electric furnace. The crucibles were kept covered, and to prevent oxidation, the charge was covered with powdered charcoal. When the alloy was molten, the cover was removed, and the charge stirred with a carbon rod to insure a homogeneous mixture. The alloys were then quenched to prevent segregation. They were analyzed for both lead and tellurium. Scott's method was used for the determination of the tellurium ⁽²⁾. From these master alloys there were then prepared alloys containing 0.1%, 0.06%, and 0.01% tellurium, the same precautions being taken as in making the 10% alloys.

Specimens were sawed from the alloys for microscopical examination. The surface of the alloy was smoothed with a file and then rubbed on No.'s 0, 00, 000, 0000 emery paper. The final polish was obtained on a felt wheel with levigated alumina. The surfaces were etched with dilute, hot nitric acid, with the exception of the alloys with less than 1% tellurium. These were etched with 75% acetic acid

plus a few drops of hydrogen peroxide.

Pieces of pure lead and lead containing 0.01%, 0.06%, and 0.1% tellurium were cold rolled and annealed for one hour at 250° C. The annealing was done in an electric tube furnace. A stream of natural gas was kept flowing through the furnace to prevent oxidation.

Photomicrographs were taken at 100 diameters and 400 diameters, and photomacrographs were taken at 3 diameters magnification.

DISCUSSION

Figure 2 is a photomicrograph, at 100 diameters, of an alloy containing 10% tellurium. It shows crystals of lead telluride with a matrix of lead. The PbTe crystals show up very prominently upon etching with nitric acid.

Figure 3 is a photomicrograph of an alloy with 90% tellurium, showing crystals of tellurium with the lead telluride-tellurium eutectic as the matrix. The crystals of tellurium and the eutectic matrix are very nearly the same color, and consequently, a sharp contrast is not obtained. The alloy is very brittle and has a silvery color.

Lead as a structural material for the chemical industry has two particular features of fundamental importance: (a) the grain size is liable to and does vary within wide limits; (b) it is very susceptible to grain growth

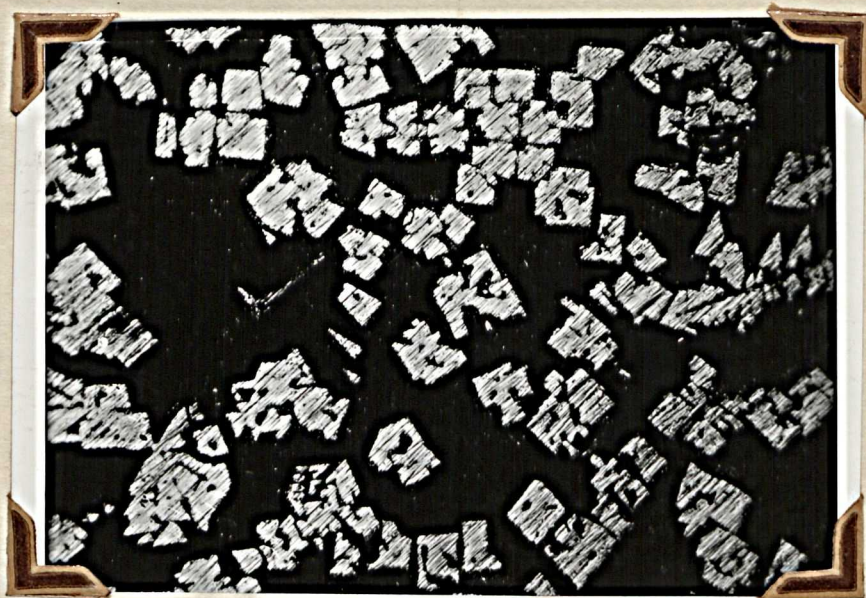


Fig. 2 10% Te - 90% Pb, 100x
As cast, etched with
hot dilute nitric acid.

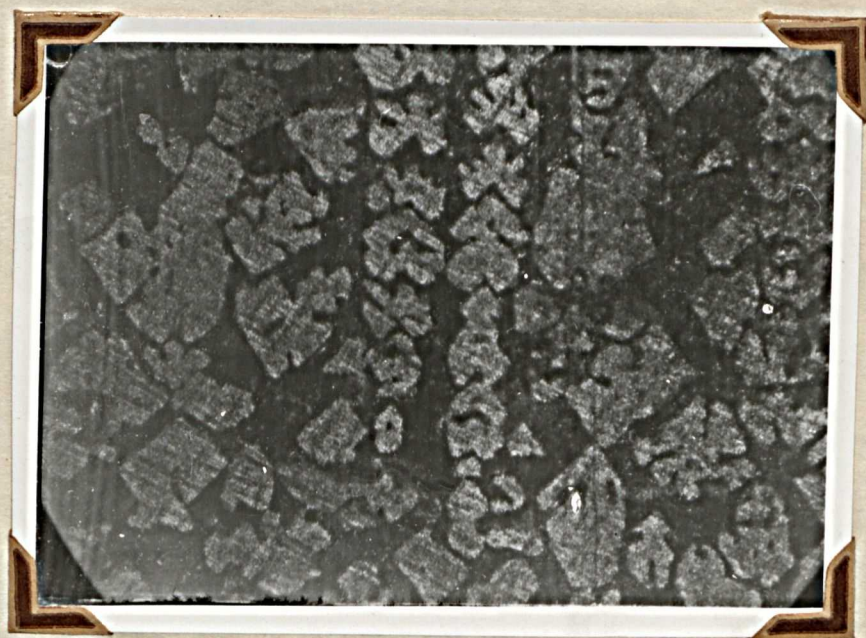


Fig. 3 90% Te - 10% Pb, 100x
As cast, etched with hot
dilute nitric acid.

and recrystallization at ordinary temperatures. It is obvious that a structure containing grains which vary widely in size does not behave uniformly under straining and corroding conditions, and in such cases metals are predisposed to intercrystalline cracking. No definite grain size can be ascribed to ordinary lead since grain growth is usual and variable, both in service and shortly after rolling.

Grain growth in lead is accelerated at elevated temperatures and by strain. Figures 4 to 7 show grain growth occurring in lead as a result of heat treatment at 250° C for one hour compared with that in lead containing 0.1% tellurium after similar treatment. The alloys containing 0.01% and 0.06% tellurium were also annealed, but they did not show as marked a degree of prevention of self annealing as the 0.1% tellurium alloy did. The 1% alloy did not differ from the 0.1% alloy, consequently, it seems as though the effect of tellurium to prevent grain growth in the lead is complete at 0.1% tellurium.

Some of the mechanical properties of lead containing 0.1% tellurium have been studied by Messrs. Singleton and Jones of England ⁽⁴⁾ and the British Non-Ferrous Metals Research Association. Their results show that the ratio of strength of tellurium-lead sheet to that of lead sheet is greater at 100° than at ordinary temperatures. Specimens of extruded ordinary lead and extruded tellurium lead were over-strained $12\frac{1}{2}\%$ in tension and then tested at

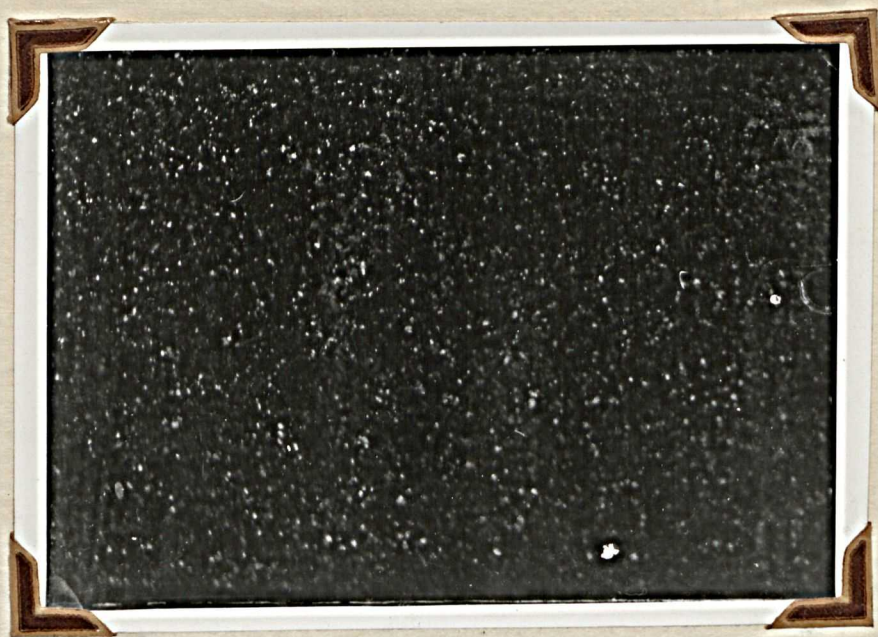


Fig. 4 Pb as rolled, 3x
Etched with 75% HAc, H₂O₂



Fig. 5 Pb after annealing for one hour
at 250°C. Etched with HAc, H₂O₂.



Fig. 6 Pb plus 0.1% Te, 3x
As rolled, etched with
HAc plus H₂O₂.

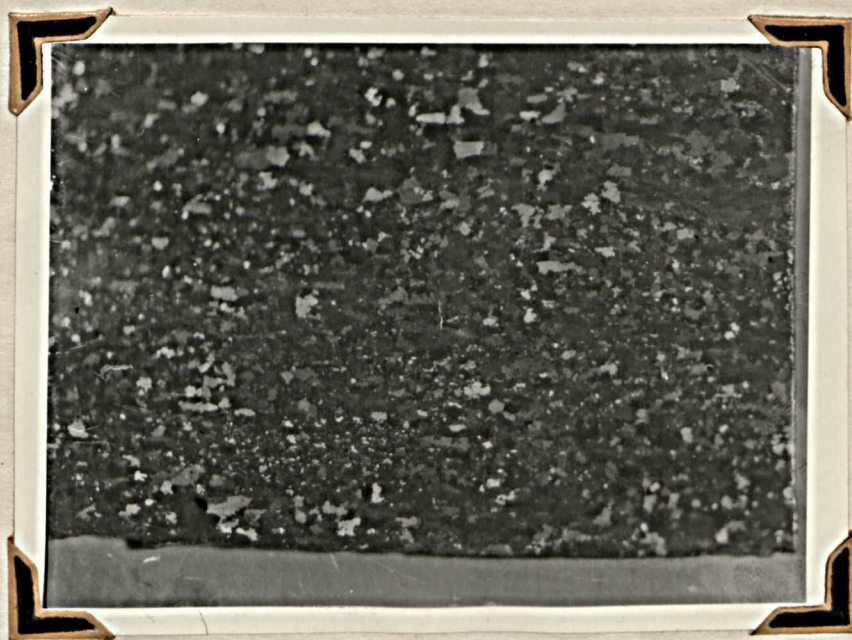


Fig. 7 Pb plus 0.1% Te, 3x
After annealing for one hour
at 250°C, etched with HAc, H₂O₂.

various periods after being allowed to rest in a condition free from stress at room temperature. The tests show the following changes occurring during a period of 6 months after straining: (1) the strength of lead shows an increase after 3 minutes but then commences to decrease, until after 6 months interval the strength is 12% less than the original value, (2) extruded tellurium lead shows a considerable increase in strength after 3 minutes. The increase continues to develop over a period of 6 months, when it has reached a strength 21% greater than the original value, showing that strain develops strength in tellurium-lead. Lead with less than 0.1% 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.

Figure 8, a photomicrograph of a section of lead at a magnification of 400 diameters, shows the uniform structure of the individual grains, each of which has a definite orientation. Each grain is oriented in a different direction from that of the adjacent grains, and so there is a sudden change in the direction of orientation at the grain boundaries. Because of the low temperature of recrystallization, there is a tendency for the grains to orient themselves in the same direction and thus unite to form one large grain. This is what is occurring in figure 8. This tendency for grain growth is increased by conditions of strain and rise of temperature. During the

period of instability that exists in lead under these conditions, the grain boundaries are in an unstable condition; the knowledge that intercrystalline cracking is the most common form of breakdown of lead sheet and pipe is an indication of this.

Cook and U.R. Evans ⁽³⁾ observed in lead the growth of old grains, the shrinkage and disappearance of others, and the birth of new grains. They found that the main changes which took place during the annealing of lead consisted of the birth of new grains followed by their growth at the expense of the old grains. My observations are in accord with Cook and Evans as is shown by figure 8.

The practical effects which result from the foregoing are clearly illustrated in the behavior of chemical lead ⁽⁴⁾ and tellurium-lead, when subjected to strain. In the case of chemical lead, where the structure is irregular and variable in size, strains are unevenly distributed and are concentrated in certain grains. These grains recrystallize and favorable conditions for intercrystalline cracking are established. In the case of lead with 0.1% tellurium, which has a refined grain and is uniform (See figure 7), strain is more evenly distributed and the majority of the grains are affected, so that the concentration of strain is absent in tellurium-lead, and the possibility of intercrystalline cracking is markedly reduced. The effect of tellurium to prevent self annealing seems to be complete at 0.1% tellurium.



Fig. 8 Pb at 400x
Etched with dilute, hot
nitric acid.

The lead containing 0.1% tellurium was also found to be more resistant to corrosion by sulphuric acid. Two cold rolled sheets, one of pure lead and the other of lead containing 0.1% tellurium and having approximately the same surface area were annealed at 250°C for one hour, weighed, and then exposed to the action of hot sulphuric acid for a short time. The sheets were then removed, washed, and reweighed. The lead sheet had lost 15.9% of its weight, while the tellurium-lead lost only 6.8% of its weight.

SUMMARY

1. The addition of tellurium to lead raises the temperature of recrystallization, thus making it stronger at ordinary temperatures than pure lead.
2. The effect to prevent self annealing after cold work is complete at 0.06 - 0.1% tellurium.
3. Tellurium-lead has increased resistance to corrosion by sulphuric acid.

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