

The Low Temperature Properties of Tin and Tin-lead Alloys

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INTRODUCTION AND PREVIOUS WORK

THE determination of the low temperature tensile properties of tin and tin-lead alloys was initiated as part of an extensive research program on the phase equilibria of tin binary systems below 13.2°C and the property changes associated therewith. A survey of the literature revealed that, although some room temperature tensile data were available for tin and a few tin-lead alloys, there existed no survey report of the low temperature tensile properties of these alloys. Accordingly, the tensile data for tin and a series of pure tin alloys containing from 0.01 to 50 pct lead were measured at eleven temperatures from -196 to +20°C and are presented here in survey form.

In addition to the importance of these tensile data as a preliminary to certain fundamental research, this information promises also to be of commercial importance because of the extensive use now being made of tin-lead solders in refrigeration and gas liquefaction equipment. Occasional failures of the solders in low temperature service have been attributed by some metallurgists to low temperature

embrittlement, while others hold that failure results from the transformation of the tin-rich phase of the solder from the solid white (beta) to the powdered gray (alpha) form. From the data previously available in the literature, neither of these explanations can be completely corroborated.

Cohen and van Lieshout¹ have established the beta to alpha transformation at 13.2°C and have shown that strain accelerates the transformation. Further, they showed that although 1 pct lead retarded the transformation, it was not eliminated. It will be pertinent, then, to note whether the beta to alpha transformation of the tin-rich phase will occur in a short-time tensile test.

Polanyi and Schmid² studied the tensile properties of tin single crystals at -185°C and report that tin is brittle at this temperature. In 1946, Kostenets³ reported that pure tin was brittle at -196 and -253°C, while lead remained ductile at both these temperatures. He also studied the tensile properties of tin-lead alloys containing from 10 to 75 pct lead at -253, -196 and +17°C. The ultimate tensile strength increased more than two times in going from room temperature to -196°C and about three times that value on further cooling to -253°C. All the alloys were brittle at -196°C except those containing more than 75 pct lead. Even at -253°C, the 75 pct lead alloy retained 36 pct elongation in a 30 mm-ga. length.

Creep is also an important phenomenon

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¹ References are at the end of the paper.

which must be considered in the tensile testing of tin-base alloys. Chalmers⁴ found that polycrystalline tin shows macro-creep above 57 psi at room temperature. This minimum creep stress threshold may be higher for the tin-lead alloys and at lower temperatures, but with data now available, this cannot be forecast with any certainty.

MATERIALS AND PROCEDURES

Preparation of Alloys

Tin and lead of extremely high purity were used in all the alloys. The tin was obtained from the Vulcan Detinning Co. and was produced by an electrolytic process. Spectrographic analyses on the tensile bar castings themselves showed that all impurity elements analyzed for were less than the detectable limit. The lead was an extremely pure commercial grade containing less than 0.009 pct residual impurities and was obtained from the American Smelting and Refining Co. The lead content of the alloys with 1 pct or less was determined by spectrographic methods or by wet methods in the case of higher lead contents. The commercial solder included in the test analyzed 49.82 pct Sn, 1.57 pct Sb and 0.01 pct Cu.

The pure tin and the series of tin-lead alloys, containing 0.01, 0.05, 0.1, 0.35, 1.0, 1.66, 5.0, 10.0, 38.7 and 50.0 pct lead, were melted in a graphite crucible under a protective cover of cottonseed oil. The molten metal was then teemed into the mold from a graphite bottom-pour ladle. A split steel mold, designed to feed the casting from the bottom to prevent cold shot, was used to prepare the cast tensile bars. The metal was poured at 300°C and the mold was heated to 200°C by platen heaters fitted to each half of the mold. Immediately after pouring, the mold was water quenched from the bottom to insure sound castings as well as to obtain a reasonably fine grain size. When this casting technique was rigorously followed,

each casting produced six tensile bars of good surface which could be tested without further machining except for threading the grips. The standard tensile specimen was 0.25 in. in diam, of 1 in.-ga. length with a 1½ in. shoulder radius.

Heat Treatment and Microstructure

The tin-lead system is a simple eutectic with solid solubility of both tin and lead in each other. Stockdale⁵ has established the eutectic composition as 38.14 pct lead, and the eutectic temperature at 183.3°C. Homer and Plummer⁶ and Stockburn⁷ found the maximum solid solubility of lead in tin to be 1.6 pct and 2.0, respectively, at the eutectic temperature, while the solubility of tin in lead is 19.5 pct at this temperature. The solvus line on the tin-rich side has not been accurately determined, but from work now in progress, it is believed the solid solubility of lead in tin at room temperature is less than 0.3 pct lead. The solubility of tin in lead at room temperature, although not of primary interest here, is about 0.4 pct tin.

All the alloys were homogenized at 170°C for 200 hr in a Silicone bath prior to testing to eliminate segregation which occurred during cooling from the melt. Metallographic examination showed that alloys containing less than 1.6 pct but greater than 0.1 pct lead were cored in the as-cast form as shown in Fig 1a. However, the above homogenization treatment entirely eliminated coring and produced the uniform spherodized structure shown in Fig 1c. Annealing at lower temperatures and shorter times, for example, 150 hr at 125°C, did not eliminate segregation, as shown by Fig 1b. The 5 to 10 pct lead alloys had a typical dendritic microstructure in the as-cast condition. Annealing resulted in agglomeration of the second phase as seen in Fig 2a, but its distribution throughout the structure was little affected. The as-cast eutectic structure was lamellar

near the outside surface of the casting, but even water quenching the mold did not cool the casting fast enough to prevent general spheroidization, as shown in Fig 2a.

proeutectic lead was obtained which, upon annealing, formed large massive particles.

The relatively fine grain size of the cast structure was retained by all the alloys

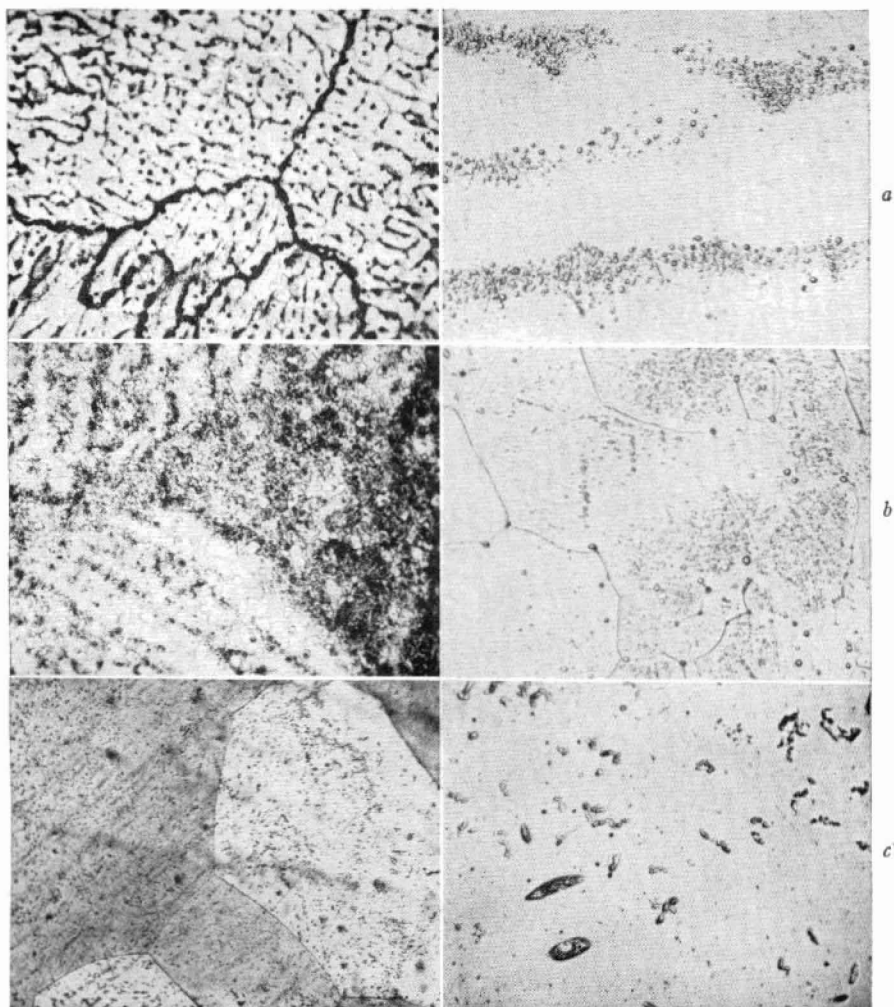


FIG 1—MICROSTRUCTURES OF A 1 PCT LEAD TIN-LEAD ALLOY IN THE AS CAST AND ANNEALED CONDITIONS.

a. As cast. *b.* Annealed 125 hr at 150°C. *c.* Annealed 200 hr at 170°C. Etched in 5 parts glycerine, 3 parts acetic acid, 1 part nitric acid. Left 50 X. Right 500 X. Reduced approximately one third.

Rather large particles of proeutectic lead also appear in this microstructure because the alloy contained about 0.6 pct lead in excess of the eutectic composition. The 50 pct lead alloy was essentially the same as the eutectic structure except that more

during homogenization at 170°C except for those containing 1. and 1.6 pct lead. In these cases, strain imposed by the differential cooling of the mold and specimen appeared to accelerate grain growth so that an average of only 5 to 8 grains

in a 0.25 diam cross-section was ultimately obtained. The effect of this abnormal grain size will be considered in the discussion.

The device ultimately developed is a modification of the usual "weigh-bar," consisting of a carefully machined thin

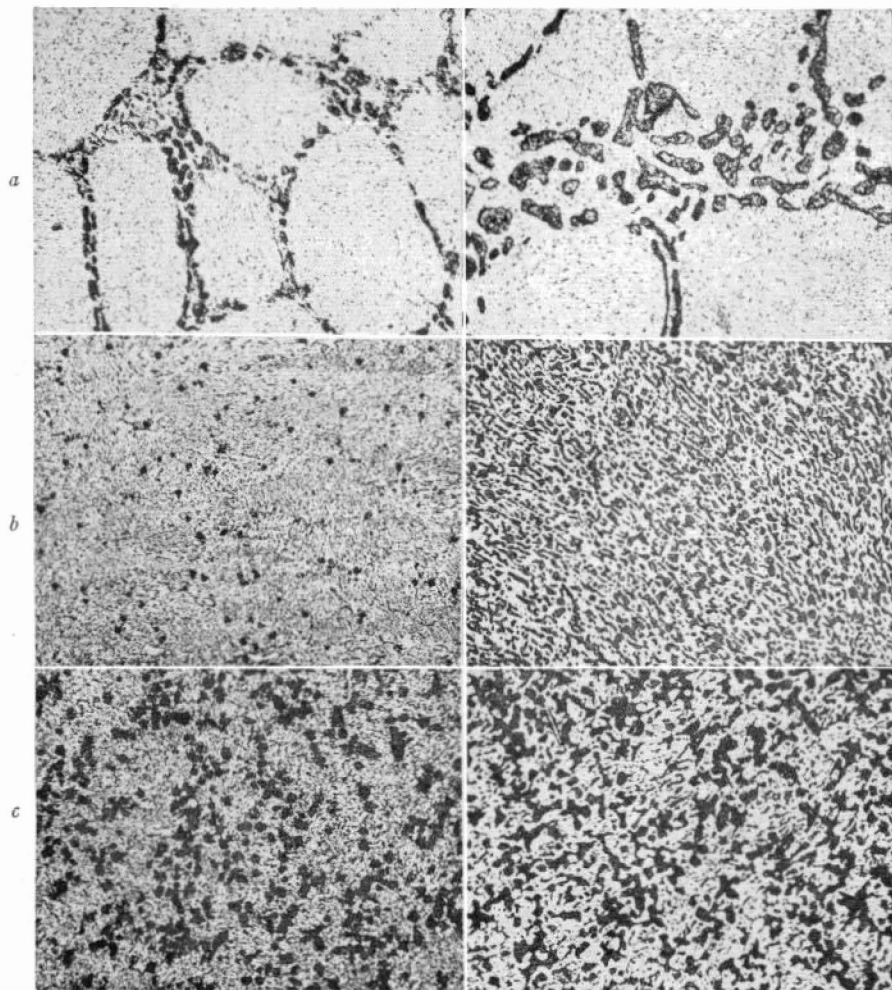


FIG 2—MICROSTRUCTURES OF AS CAST AND HOMOGENIZED TIN-LEAD ALLOYS.
a. 10 pct lead, 500 X. *b.* 38.8 pct lead, 100 X. *c.* 50 pct lead, 100 X. Etched in 8 parts glycerine, 1 part acetic acid, 1 part nitric acid. Left: as cast. Right: annealed 200 hr at 170°C. Reduced approximately one third.

Testing Methods

The tensile tests were made on a Baldwin-Southwark tensile machine of 60,000 lb capacity, equipped with a 6,000 lb scale which could be read only to ± 5 lb. Since the loads needed for these tests were quite low, it was necessary to design a special device for accurately measuring the loads.

walled 75 S-T aluminum tube, 0.02 sq in. in cross-section, with two longitudinal wire strain gauges mounted on opposite sides and wired in series to compensate for any lack of axially. A calibration curve was established for load vs. the apparent strain on the weigh bar by taking strain readings at small load intervals up to 1,000 lb.

The weigh bar unit was threaded into the upper grip at the top and into a $\frac{3}{4}$ in. diam stainless steel extension rod at the bottom to which the specimen was attached. The stainless steel extension rod reduced the heat leak along the grip so that during the low temperature tests, the weigh bar did not fall below room temperature. The bottom grip was threaded through the bottom of an insulated brass container for holding the liquid in which the specimens were immersed for the low temperature tests. The vessel was also equipped with a stirrer, copper cooling coils and a bimetallic thermost switch. The thermost switch actuated an on-off solenoid which admitted compressed air to the Dewar of liquid air and forced the latter through the copper cooling coils. This, along with a slight steady flow of coolant controlled by a bleeding by-pass around the solenoid in the compressed air line, permitted temperature control to within $\pm 1^\circ\text{C}$ down to -165°C . This assembly was then attached to the testing machine by Robertson shackles which gave ball point contact and thus insured axiality of loading. Also, the coolant container, being threaded to the lower grip, was prevented from preloading the specimen by supporting it with springs.

Petroleum ether was used as the immersion liquid down to -125°C , while isopentane was found most satisfactory for the immersing bath between -125° to -165°C . The temperature control mechanism described above was used to maintain the test temperatures above -165°C , while at -183 and -196°C , liquid oxygen and liquid nitrogen, respectively, were used directly in the container with the thermost switch removed. The temperature of the bath was measured by a copper-constantan thermocouple placed near the specimen.

The selection of the proper strain rate which would permit comparison of the tensile data both as a function of the

composition and temperature was difficult because of the influence of strain rate on creep rate and extent of work hardening. In pure tin, any load in excess of 57 psi (3 lb on 0.25 diam test bar) results in macro-creep at room temperature while no creep data on tin-lead alloys are available. Thus, if the creep rate at low loads is significant at a given testing temperature, a very slow strain rate would indicate nil tensile strength. Equally important is the effect of strain rate on the extent of work hardening. Since tin and presumably the tin-lead alloys will be below their recrystallization temperatures at most of the testing temperatures, work hardening will occur as long as the metal retains any ductility. The increase in strength caused by work hardening, which can be realized before the metal reaches its ultimate tensile strength, will depend upon the strain rate.

It was decided, accordingly, since neither creep properties nor work hardening characteristics of these alloys were available, that a strain rate of 0.2 in. per in. per min., which was primarily a practical one, would be used. This rate gave sufficient time for measuring the load by the "weigh-bar" method when brittle fractures occurred and, further, was fast enough to minimize creep phenomena.

This experimental set-up permitted determination of the ultimate tensile stress to within ± 1 lb in temperature ranges where plastic deformation occurred, and possibly within about ± 5 lb when the specimens were brittle. Per cent elongation was measured in the one inch gauge length and the reduction in area data was obtained from measurement of the initial and final diameter at the point of fracture. Attempts were also made to measure yield strengths, but neither the "weigh-bar" nor the "drop-of-the-beam" method gave reliable results. Therefore, only low temperature ultimate tensile strength, per cent elongation in one inch,

and reduction in area data were obtained for these alloys. Single specimens of each composition were tested at each of eleven temperatures between -196 and $+27^{\circ}\text{C}$.

inclusively, the ultimate tensile strength, the reduction in area and elongation in a 1 in. ga. length being plotted as functions of both temperature and composition.

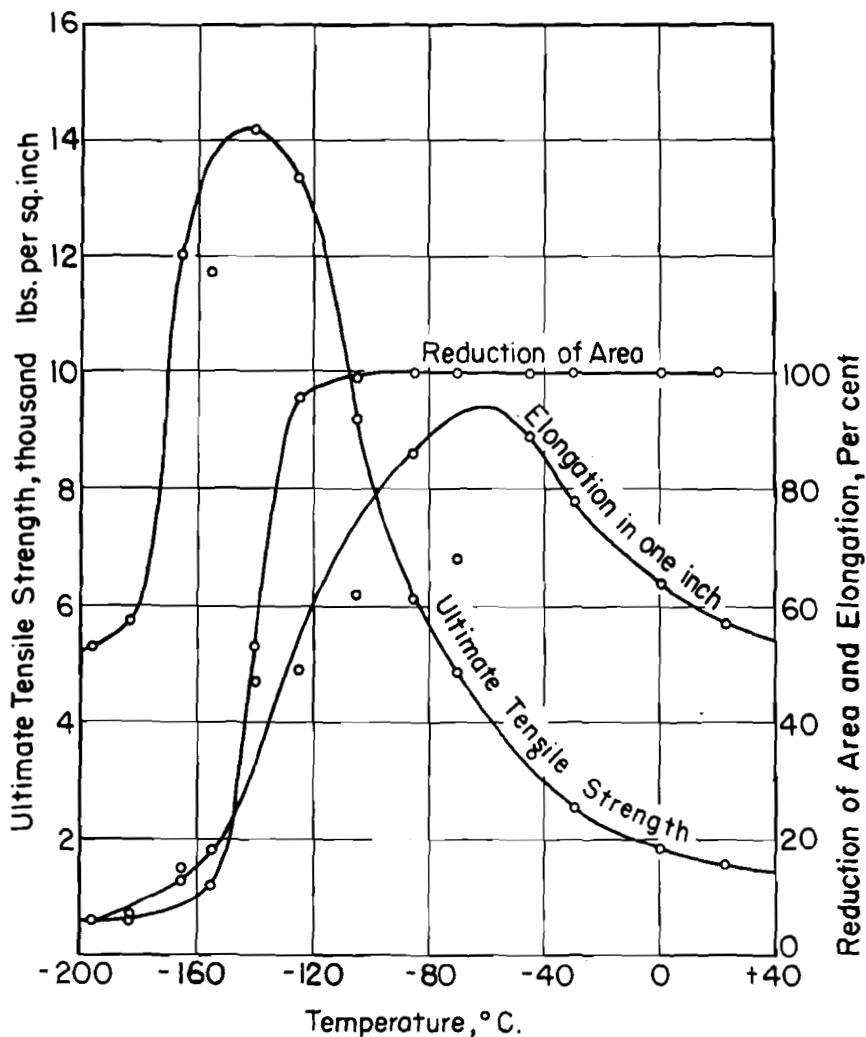


FIG 3—THE EFFECT OF TEMPERATURE ON THE TENSILE PROPERTIES OF PURE TIN.

A single test at each temperature and composition level was considered sufficient for this survey because the temperature and composition intervals were small.

DISCUSSION OF RESULTS

The data obtained in this investigation are presented graphically in Fig 3 to 10

As explained earlier, yield strength data were not obtained because the methods of measurement were not reliable. Although more fundamental data would be required for a rigorous analysis, it is believed that these low temperature tensile properties can be adequately explained in terms of the effect of decreasing

temperature on (1) the critical shear or flow stress, (2) the extent or amount of cold work, and (3) the technical cohesive strength⁸ (fracture strength at nil cold

of the second phase will influence the magnitude of the net temperature effects. In addition, the possibility that the beta to alpha transformation of the tin or tin-

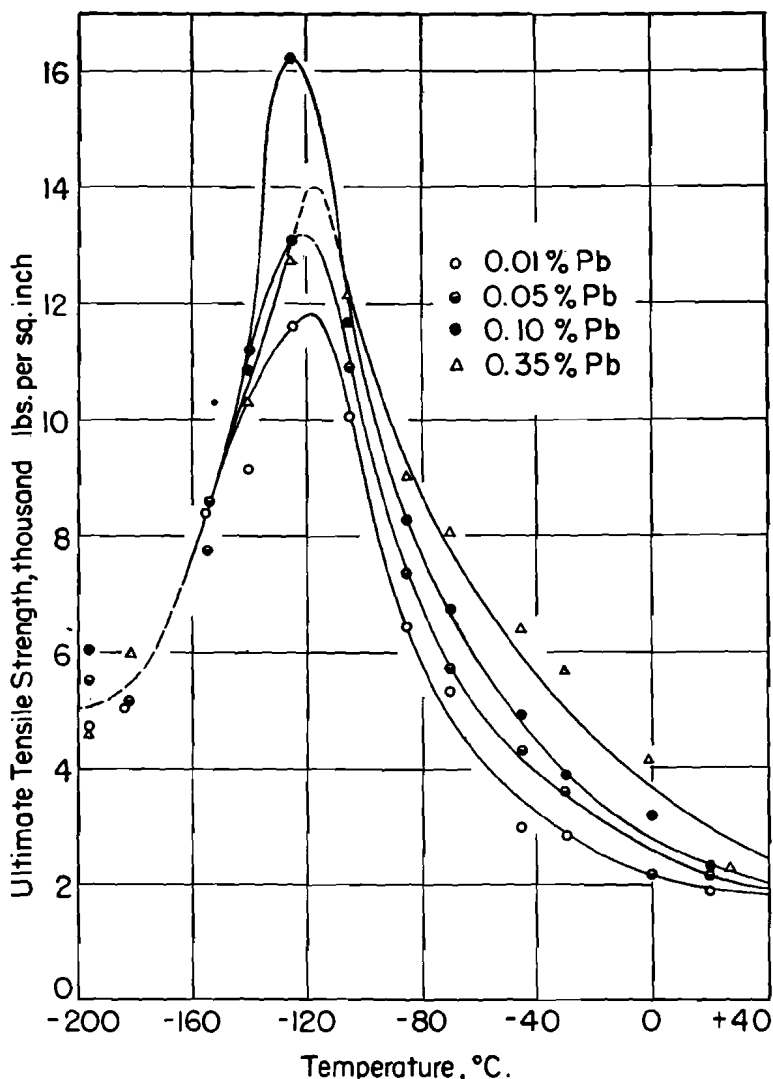


FIG 4—THE EFFECT OF TEMPERATURE ON THE ULTIMATE TENSILE STRENGTH OF TIN-LEAD ALLOYS CONTAINING FROM 0.01 TO 0.35 PCT LEAD.

work). In the case of the alloys containing two phases, the tensile properties will be the composite of these three factors operating on the separate phases, and thus the amount, the form and the distribution

rich phase would occur during the tests below 13.2°C had to be considered as previously mentioned. Careful macro-examination, however, of the fractured surfaces revealed none of the gray (alpha)

modification, so that the transformation was eliminated as a variable or contributing factor.

Attention is also called to the use in this discussion of certain terms which differ from their generally inferred mean-

ing. It was found convenient, for example, to use only the reduction in area as the principal measure of ductility, instead of elongation. The temperature of embrittlement T_B as used here means the temperature at which 50 pct of the maximum

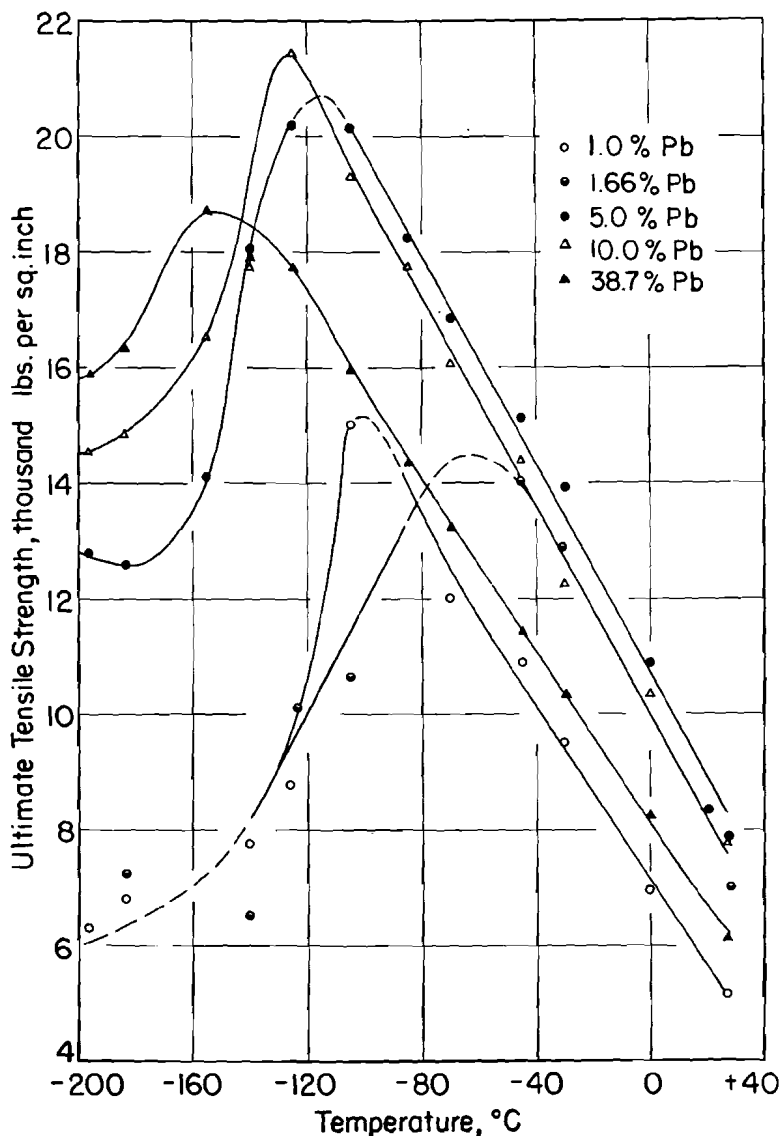


FIG 5—THE EFFECT OF TEMPERATURE ON THE ULTIMATE TENSILE STRENGTH OF TIN-LEAD ALLOYS CONTAINING FROM 1.0 TO 38.7 PCT LEAD.

ductility as defined by reduction in area is lost. The self-defined term, temperature of maximum ultimate tensile strength, T_M , is also a new and convenient expres-

to $+23^{\circ}\text{C}$. The ultimate tensile strength, starting at 1600 psi at room temperature, increased rapidly as the temperature decreased, reaching a maximum value of

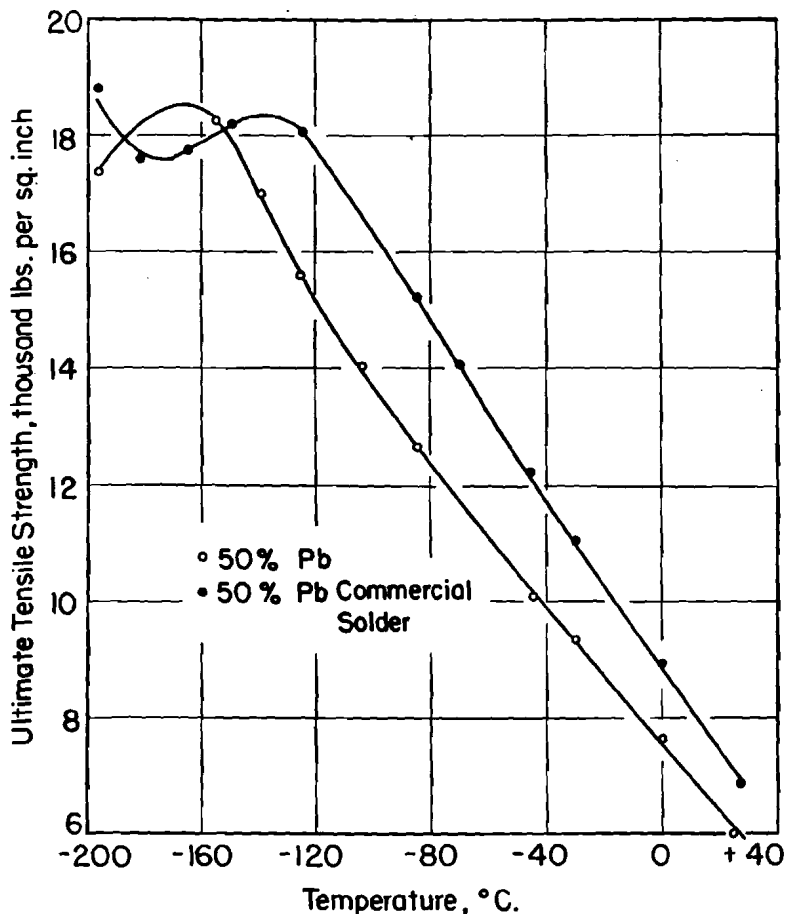


FIG 6—THE EFFECT OF TEMPERATURE ON THE ULTIMATE TENSILE STRENGTH OF PURE 50-50 TIN-LEAD ALLOY AND 50-50 COMMERCIAL TIN-LEAD SOLDER.

sion used throughout the discussion. Technical cohesive strength means the fracture stress at nil cold work or nil ductility. The critical flow stress is defined as the applied stress required to start flow.

PURE TIN

Fig 3 is a graphical summary of the tensile properties of pure tin from -196

$14,200$ psi at -140°C , and then decreased abruptly to 5300 psi at -196°C . The reduction in area remained at 100 pct to -125°C , then dropped sharply and approached a value of 6 pct at -183°C . The elongation, on the other hand, increased steadily to a maximum of 94 pct, then gradually decreased to a value of 6 pct at -196°C .

In terms of the three fundamental phenomena mentioned above, these data can be explained as follows. Fracture at temperatures down to -125°C occurred

strength. Since the tensile strength continued to rise from -60 to -140°C , it must be presumed that the critical flow stress also rises in this temperature range.

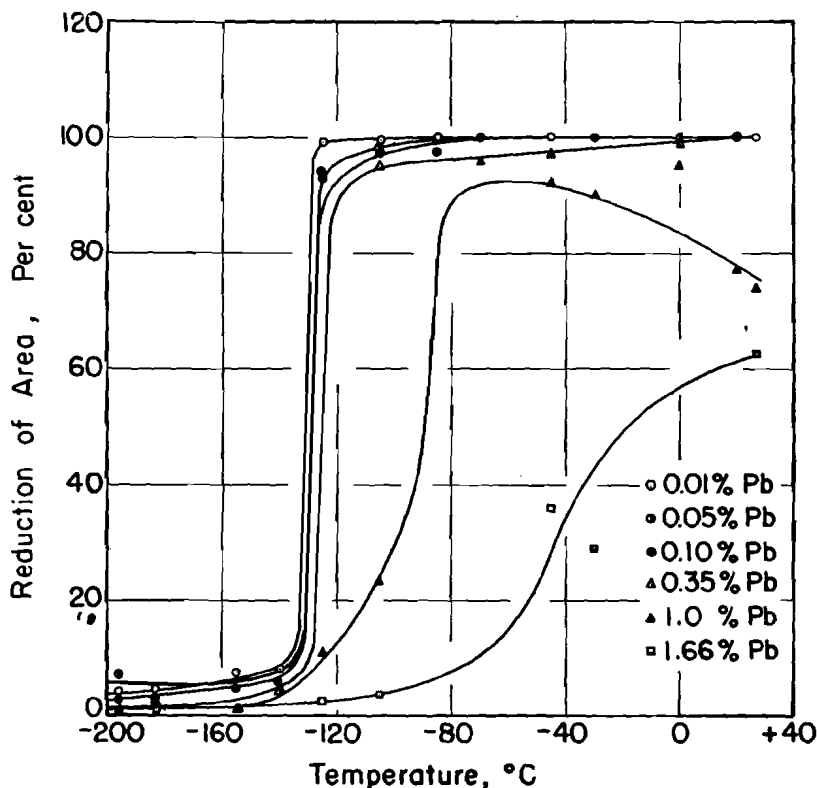


FIG 7—THE EFFECT OF TEMPERATURE ON THE REDUCTION IN AREA OF TIN-LEAD ALLOYS CONTAINING 0.01 TO 1.66 PCT LEAD.

principally by flow, since the metal remained essentially 100 pct ductile at the point of failure. The rapid increase in ultimate tensile strength as the temperature decreased was a result, then, of an increase in both the critical flow stress and work hardening. Down to -60°C , work hardening was probably increasingly important, as evidenced by the increase in elongation to a maximum at this temperature. Below -60°C , the amount of work hardening apparently decreased and the critical flow stress became more important in establishing the ultimate tensile

Now as the ductility began to fall off at -130°C , failure in tension occurred partially by cleavage and partially by flow. At the temperature of 50 pct reduction in area, for example, failure probably occurred half in cleavage and half by flow, and this, then, represented the defined temperature of embrittlement, T_B . At this point, the cleavage stress was being increased by work hardening because considerable ductility still remained. Then the contribution of flow stress to the ultimate tensile strength fell off as the ductility decreased until failure occurred

principally by cleavage. Simultaneously, the cleavage stress decreased and approached the true fracture stress at nil

ture on the low temperature tensile properties will be emphasized.

In order to avoid confusion, the rela-

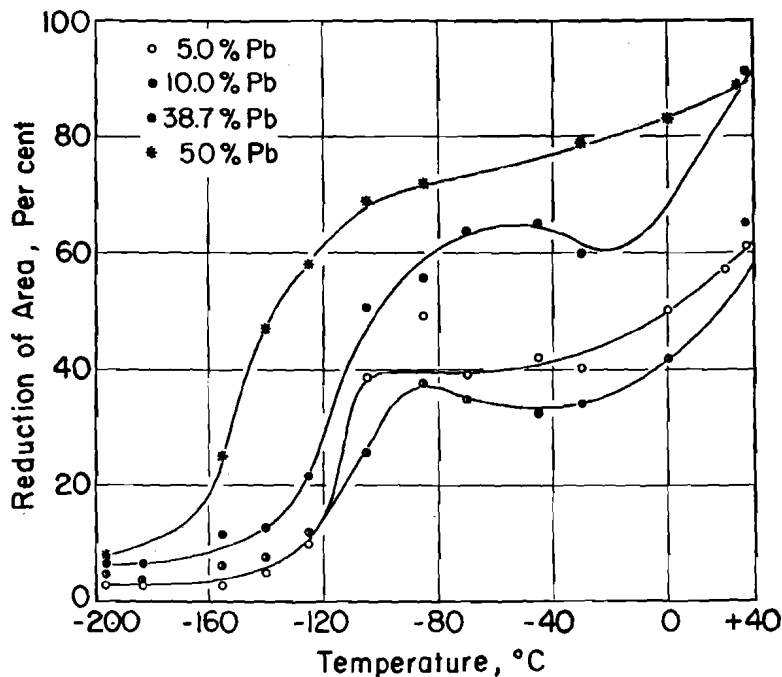


FIG 8—THE EFFECT OF TEMPERATURE ON THE REDUCTION IN AREA OF TIN-LEAD ALLOYS CONTAINING 5 TO 50 PCT LEAD.

cold work, that is, the technical cohesive strength.

TIN-LEAD ALLOYS

Fig 4 to 10 contain the tensile properties of the tin-lead alloys from -190 to $+27^{\circ}\text{C}$. From the general shapes of the curves, it is believed the same general phenomena which occurred in pure tin also took place in the alloys. The appearance of increasing amounts of the second lead-rich phase, however, caused definite differences in the magnitude of the changes in tensile strength and ductility which occurred as the temperature decreased. Therefore, since the temperature effects differ only in degree, their explanation in terms of the three fundamental phenomena will not be repeated, but rather the influence of the composition and resulting microstruc-

tionships of composition and low temperature tensile properties will be discussed in the following sequence: First, the general effect of composition on (1) the temperature of maximum ultimate tensile strength, T_M , (2) the embrittlement temperature, T_B , (3) the value of the maximum ultimate tensile strength and (4) the ultimate tensile strength both above and below T_B . Second, the specific influence of the microstructure of each alloy on these four functions or properties will be considered in the order mentioned.

Fig 4, 5 and 7 show that the first additions of lead had little or no effect on either the temperature of maximum ultimate tensile strength, T_M , or the temperature of embrittlement, T_B . Higher lead contents, however, affect both T_M and T_B

and, in order to show these effects more clearly, Fig 11 was constructed from the tensile and ductility vs. temperature plots. Here it is noted that T_M and T_B are not

at 0.1 pct lead, it rises from 20 to 30° above the latter and finally at 60 pct lead, they coincide. The maximum ultimate tensile strength increases steadily from 0.01 to

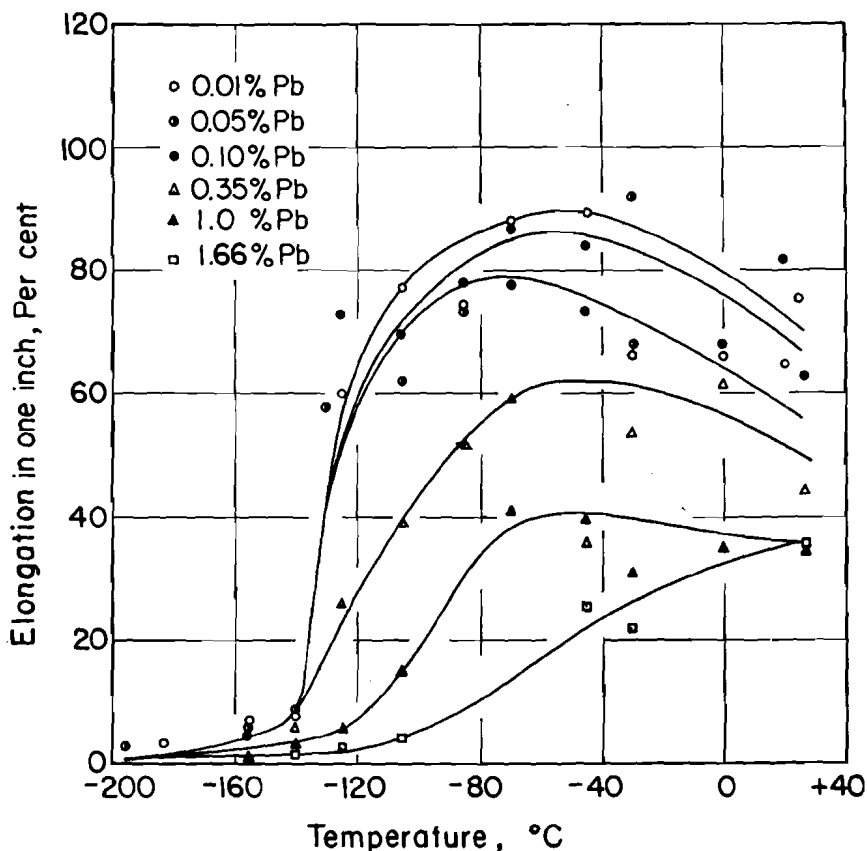


FIG 9—THE EFFECT OF TEMPERATURE ON THE ELONGATION IN ONE INCH OF TIN-LEAD ALLOYS CONTAINING 0.01 TO 1.66 PCT LEAD.

synonymous as in the case of pure tin. This is believed to be caused by the interaction of the three fundamental phenomena mentioned above operating simultaneously on the two phases present in most of the alloys.

It can be seen in Fig 11 that T_M remains essentially constant up to 0.3 pct lead, rises sharply to a maximum at about 1.6 pct lead, then gradually decreases toward absolute zero. T_B follows the same general trends but falls about 10° below T_M until,

0.1 pct lead, drops slightly at 0.35 pct lead, and then increases slightly at 1.0 pct lead. Then from 1.0 to 1.6 pct lead, the strength decreases about 400 psi, followed by a rapid increase to a maximum at 10 pct and a subsequent decline at 38.7 and 50 pct lead. In order to explain these effects adequately, the variations of ultimate tensile strength with composition must also be considered. Fig 12 and 13 are cross-plots of ultimate tensile strength vs. the logarithm of the lead content for the testing

temperatures above and below T_B , respectively. The curve for -183°C is also included in Fig 12 for comparison purposes. Above T_B , lead contents up to

pretended in terms of the influence of the amount, the size or form, and the distribution of the second phase. The first additions of lead did not affect T_M or T_B

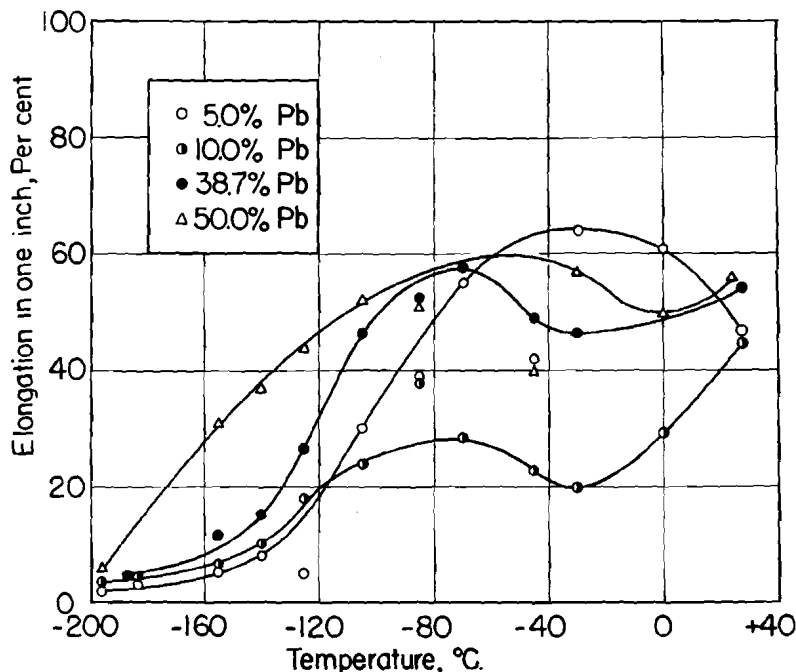


FIG 10—THE EFFECT OF TEMPERATURE ON THE ELONGATION IN ONE INCH OF TIN-LEAD ALLOYS CONTAINING 5 TO 50 PCT LEAD.

about 0.3 pct increase the ultimate tensile strength slightly. Between 0.2 and 0.3 pct lead, a sharp break occurs for all the temperatures and increasing lead improves the tensile strength to a maximum between 5 and 10 pct. The values then fall off for the 38.7 and 50 pct lead alloys. In Fig 13 it is seen that below T_B , a slight strengthening occurs up to about 0.1 pct lead at -125 and -140°C , then an apparent minimum is seen between 1 and 1.6 pct lead. At -183°C there is a slight decrease, and at -196°C a slight increase in strength for lead contents to about 1.6 pct. Above this composition, the ultimate tensile strength increases steadily and then either attains or approaches a maximum.

These data can be quite logically inter-

as long as the lead remained in solid solution. The solid solubility of lead in tin at and below room temperature is not known precisely, but the variation in tensile strength with temperature, as shown in Fig 12 and 13, indicate it probably lies between 0.1 and 0.3 pct. Further, if these data up to 1 pct lead are replotted with composition on a linear scale, as in Fig 14 and 15, a very definite change in slope is observed at about 0.1 pct lead for all temperatures. On the basis of these data and certain other work in progress,⁹ the solid solubility of lead in tin was assumed to lie between 0.1 and 0.3 pct lead. This leads to the following explanation. Lead in alloys up to 0.1 pct had no effect on either T_M or T_B because the lead was in solid

solution. Between 0.1 and 0.3 pct lead, however, fine particles of lead-rich phase appeared throughout the tin matrix and in the grain boundaries to cause precipitation

was an extrapolated value, it is not considered significant. For temperatures above T_B , the ultimate tensile strength continued to increase because of either more lead in

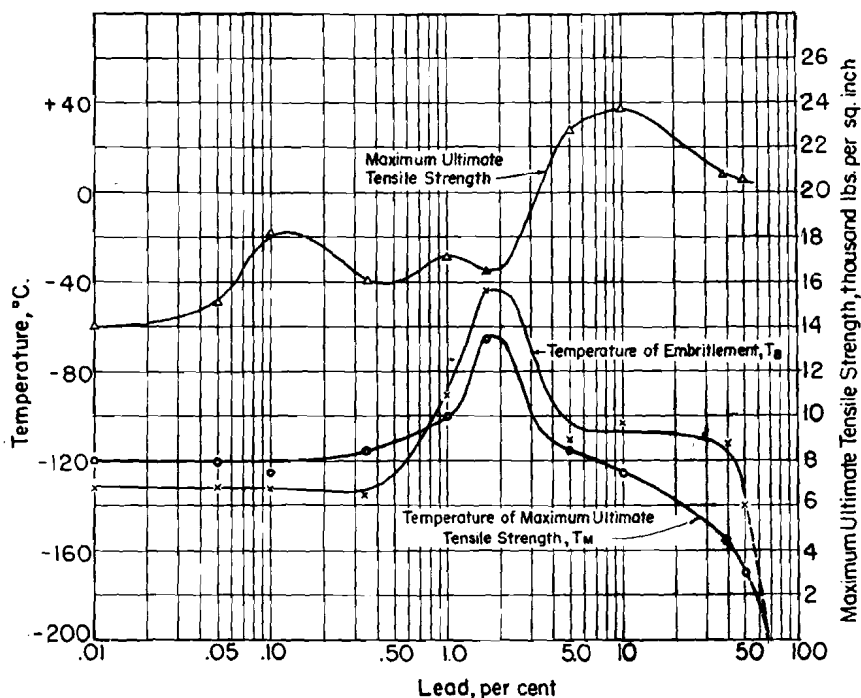
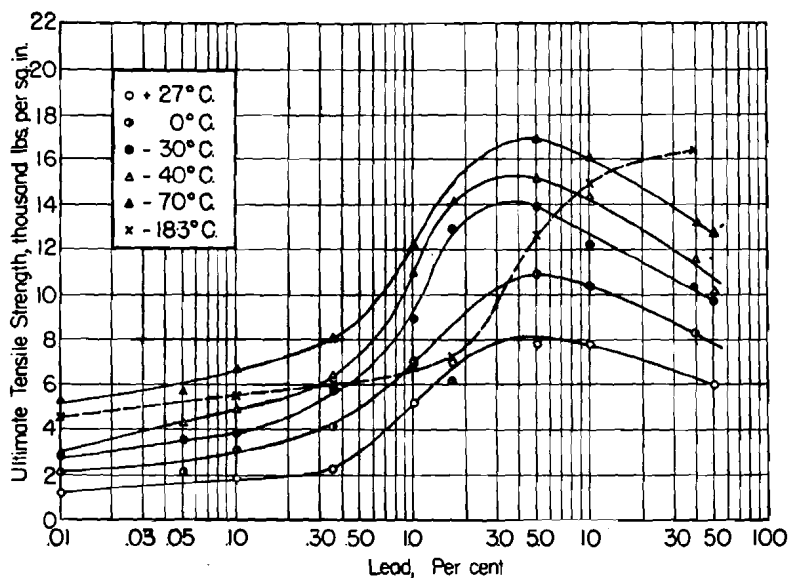
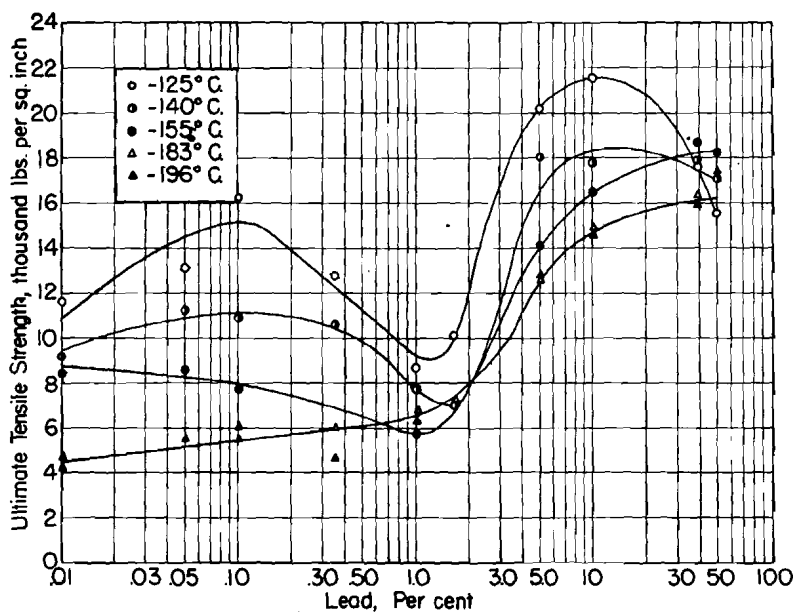


FIG 11—THE EFFECT OF COMPOSITION ON THE TEMPERATURE OF MAXIMUM ULTIMATE TENSILE STRENGTH, THE TEMPERATURE OF EMBRITTLEMENT, AND THE MAXIMUM ULTIMATE TENSILE STRENGTH OF TIN-LEAD ALLOYS IN THE COMPOSITION RANGE 0.01 TO 50 PCT LEAD.

hardening. The particles were too small, however, to affect T_M or T_B ; yet, as seen in Fig 11, the maximum tensile strength noticeably increased at about 0.1 pct lead, probably as a result of precipitation hardening. Similarly, the ultimate tensile strength at all the testing temperatures apparently increased for 0.1 pct lead alloy. This is in agreement with other work which shows marked effects of small additions of various residual elements in alloys on the room temperature tensile strength.^{10,11,12,13,14} T_M for the 0.3 pct alloy is slightly higher than for lower lead alloys, while T_B seems unaffected. The maximum tensile strength dropped slightly, but since this

solid solution or precipitation hardening. At -125°C in the 0.1 to 0.3 pct range, the tensile strength decreased sharply, but since this is based on only one point, the validity and significance of the abrupt rise and decline of the curve of Fig 14 may be questioned.

In the 0.3 to 1.66 pct lead range, the lead-rich phase precipitated principally in grain boundaries, the particles becoming larger as the amount of lead-rich phase increased. At about 1.0 pct lead, the particles exceeded the critical maximum particle size for hardening, slip was facilitated and T_B and T_M thus increased rapidly. Up to 1.0 pct lead, the maximum

FIG 12—THE EFFECT OF COMPOSITION ON THE ULTIMATE TENSILE STRENGTH FROM -183 TO $+27^{\circ}\text{C}$.FIG 13—THE EFFECT OF COMPOSITION ON THE ULTIMATE TENSILE STRENGTH FROM -196 TO -125°C .

tensile strength increase, if real, probably was caused by precipitation hardening. However, from 1.0 to 1.6 pct lead, the particles at the grain boundary became larger and more numerous and acted as

discontinuities, as reflected in Fig 11 by the slight drop in maximum tensile strength. The ultimate tensile strength, however, continued to increase with lead content in this range above T_B , as seen

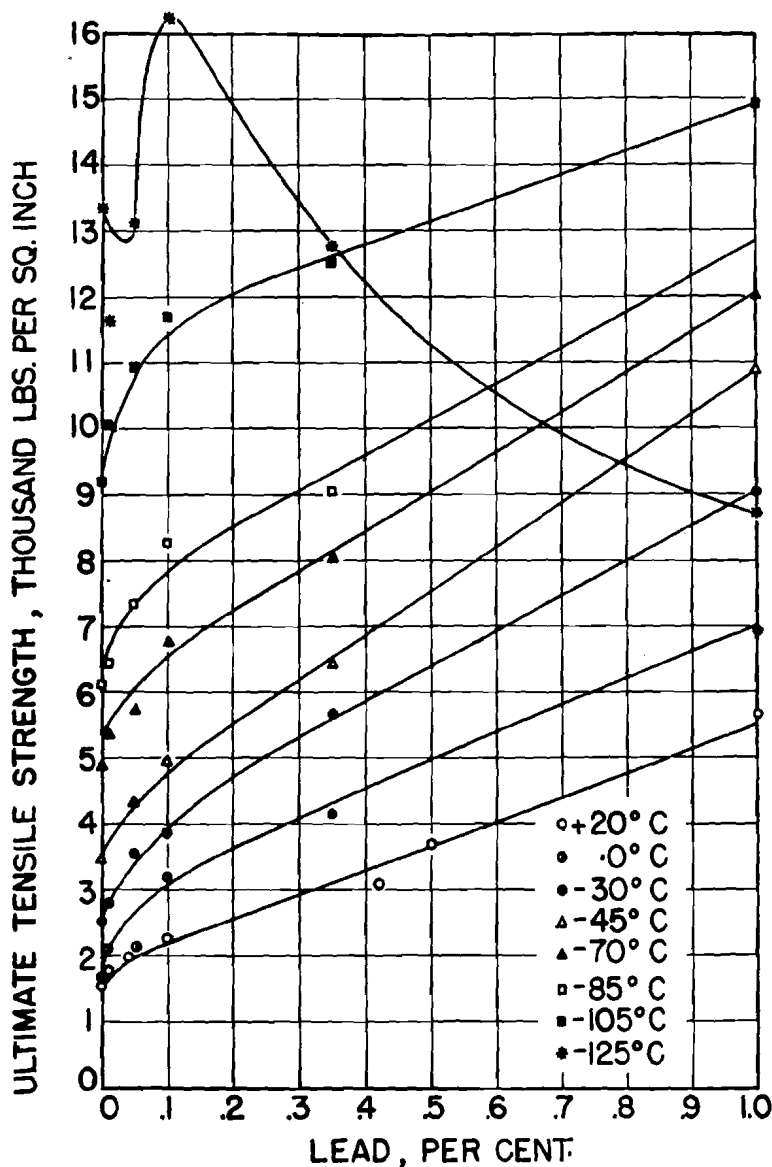


FIG 14—THE EFFECT OF SMALL AMOUNTS OF LEAD ON THE ULTIMATE TENSILE STRENGTH OF TIN FROM -125 to $+20^{\circ}\text{C}$.

in Fig 12, and increased slightly or remained essentially constant for temperatures below T_B , Fig 13. In the 1.66 pct alloy, primary lead-rich phase appeared at the grain boundaries, since this composition is slightly higher than the solid

solubility limit at the eutectic temperature.⁶ This, along with the grain boundary precipitate, formed a film which was too thin to flow. It acted, therefore, as a discontinuity and fracture occurred at the grain boundaries. Examination of the

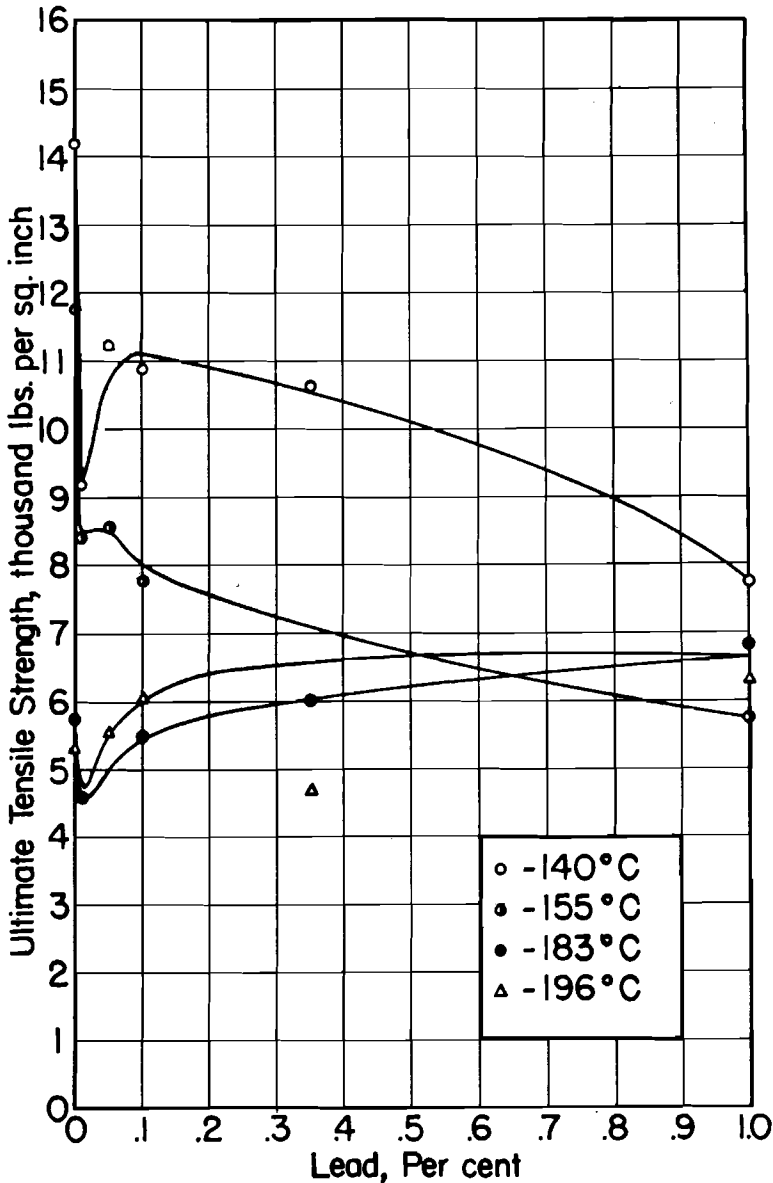


FIG 15—THE EFFECT OF SMALL AMOUNTS OF LEAD ON THE ULTIMATE TENSILE STRENGTH OF TIN FROM -196 TO -140°C .

fractures confirm this reasoning, for a conchoidal or grain boundary fracture was obtained for the 1 and 1.6 pct lead alloys only. This, then, accounts for the maxima of T_M and T_B at or about 1.6 pct lead.

As the second phase increased in the grain boundaries, its thickness increased so that it could flow. T_M and T_B thus decreased as the lead content exceeded 1.6 pct, becoming essentially constant for the 5 and 10 pct lead alloys. In these alloys, the intragranular lead-rich phase was quite massive, as seen in Fig 2, and, being ductile, was subject to work hardening at temperatures below its recrystallization temperature. As a result, the maximum tensile strength and the ultimate tensile strength above and below T_B (-110°C) both increased to maxima. That work hardening is obtained is definitely indicated by the ductility which remains and the elongation which was observed, and shown in Fig 8 and 10, in these alloys down to temperatures even below T_B . In the 38.7 pct alloy, the tin-rich phase was still continuous, but the lead phase, because of spheroidization and agglomeration of the eutectic structure, was now massive and uniformly distributed. Thus, although the ductility was improved appreciably, as shown by Fig 8, T_M and T_B decreased only slightly because of the uniform distribution of the second phase. Both the maximum tensile strength and ultimate tensile strength above T_B decreased from 10 to 38.7 pct lead, probably because the tin-rich phase in some areas may have fractured, leaving the lead phase to determine the strength of the alloy. If this occurred, metallographic examination of the deformed section immediately after fracture would be expected to reveal micro-cracks.

Above 38.7 pct lead, T_B and T_M decreased sharply, because the amount of ductile lead-rich phase was increasing. The maximum tensile strength and the ultimate tensile above T_B for the 50 pct

alloy, that is, -140°C , decreased appreciably, presumably because the continuous tin-rich phase had become brittle and the critical flow stress of the predominant lead-rich phase is low. Below T_B , for this alloy, there was little change in tensile strength. However, considerable ductility still remained at T_B , so that the cleavage stress of the alloy was conceivably being increased by work hardening.

A comparison of the tensile properties of the 50-50 pure alloy with the 50-50 commercial composition, Fig 6, shows that the commercial alloy has a consistently higher ultimate tensile strength at any given temperature, while its T_M is about 30°C higher. Embrittlement of the solders at subatmospheric service temperatures such as those encountered in refrigeration equipment does not, therefore, appear feasible from these data. Tin-lead solders containing about 70 pct or less lead, however, would become brittle in gas liquefaction installations (operating below -160°C). Creep rupture or transformation of the tin-rich phase below room temperature is another possible cause of failure. The solution of the problem from these two approaches requires creep and phase equilibria data, and at present none of this information is available.

From an extrapolation of the T_B and T_M vs. composition curves of Fig 11, one would predict that higher lead contents would remain ductile to increasingly lower temperatures. This is in agreement with Kostenets³ who found that both 75-25 Pb-Sn and pure lead retained good ductility down to -253°C .

Thus, although it was not possible to isolate the specific effects of all the variables, the interpretation of the low temperature tensile properties of tin-lead alloys in terms of their microstructure seems to be logical and straightforward. As in any survey investigation, however, questions have arisen which cannot now be answered.

True stress strain, yield strength and certain other fundamental data are needed to answer these questions and confirm the basic reasoning used above. It is hoped that such data can be obtained in the near future.

CONCLUSIONS

1. Pure tin and all the tin-lead alloys containing up to 5.0 pct lead become brittle at about -110°C , except for the 1.0 and 1.66 pct lead alloys which give brittle fractures at higher temperatures. Alloys containing more than 10 pct lead remain ductile at successively lower temperatures as the lead content increases. Since tin is body-centered tetragonal and lead is face-centered cubic, this low temperature behavior substantiates the prediction of Seigle and Brick¹⁵ that only face-centered cubic metals remain ductile at temperatures near absolute zero.

2. Tin and tin-lead alloys increase in strength as the temperature is decreased prior to embrittlement. This increase in tensile strength in the ductile state has been attributed to the increase of the critical flow stress with decreasing temperature, the effects of work hardening, and the increase in technical cohesive strength with decreasing temperature.

3. Additions of lead up to 1.6 pct increased the ultimate tensile strength slightly at any temperature level between $+27$ and -196°C . Above 1.6 pct lead, the ultimate tensile strength both above and below the temperature of embrittlement is improved markedly to a maximum between 5 to 10 pct lead, and then decreased for all the temperatures except -183 and -196°C . At these temperatures, the maximum ultimate tensile strength occurred in the 38.7 and 50 pct lead alloys. The initial increase in tensile strength of lead up to 0.1 to 0.3 pct was caused by solid solution effects. Past the solid solubility limit and up to 1.6 pct lead, the effects on low temperature properties were caused

by precipitation of the lead-rich second phase in the tin-rich matrix. Above 1.6 pct lead, work hardening of the massive and ductile second phase caused the increased tensile strength for all the test temperatures. The decline in strength was believed to result from embrittlement of the continuous tin-rich phase before the fracture stress of the ductile lead-rich phase was attained.

4. Lead in tin is responsible for a loss of ductility up to about 1.6 pct and a gain in ductility above this composition at any temperature between -196 and $+27^{\circ}\text{C}$.

5. Lead in solid solution does not change the embrittlement temperature. As a dispersed second phase, lead raises the embrittlement temperature while, as a uniformly distributed massive second phase, it decreases the temperature of embrittlement.

6. The solid solubility of lead in tin at or about room temperature appears to be between 0.1 and 0.3 pct lead as shown by significant changes of the tensile properties with composition at all temperatures between -196 and $+27^{\circ}\text{C}$.

7. A 50-50 tin-lead commercial solder was brittle below -150°C , but at this temperature its ultimate tensile strength increased to 18,500 psi. Solders of higher lead content will become brittle only at successively lower temperatures until at about 70 to 80 pct lead, the tin-lead alloys should remain ductile down to temperatures near absolute zero.

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REFERENCES

1. E. Cohen and A. K. W. A. van Lieshout: *Ztsch. Physik. Chem.* (1935), **173**, 32.

2. M. Polanyi and E. Schmid: *Zisch. Physik* (1925), **32**, 684.
3. V. I. Kostenets: Mechanical Properties of Metals under Static Load at Low Temperatures. *Jnl. Tech. Phy.* (U.S.S.R.) (1946), **16**, I, IV, 515.
4. B. Chalmers: Precision Extensometer Measurements on Tin. *Jnl. Inst. Metals* (1937), **61**, 103.
5. D. Stockdale: The Constitution of the Lead-Tin Alloys. *Jnl. Inst. Metals* (1932), **49**, 267.
6. C. E. Homer and H. Plummer: Embrittlement of Tin at Elevated Temperatures and Its Relation to Impurities. *Jnl. Inst. of Metals* (1939), **64**, 172.
7. A. Stockburn: The Solubility of Lead in Tin. *Jnl. Inst. Metals* (1940), **66**, I, 33.
8. D. J. McAdam, Jr.: Technical Cohesive Strength and Yield Strength of Metals. *Trans. AIME* (1942), **150**, 311.
9. Unpublished work, Dept. of Met., Univ. of Pennsylvania.
10. D. Hanson and W. T. Pell-Walpole: A Study of the Mechanical Properties of Tin-Rich Antimony-Cadmium-Tin Alloys. *Jnl. Inst. Metals* (1937), **61**, 123.
11. D. Hanson, E. Sandford and H. Stevens: Some Properties of Tin Containing Small Amounts of Silver, Iron, Nickel or Copper. *Jnl. Inst. Metals* (1934), **55**, 115.
12. D. Hanson and E. Sandford: Some Properties of Tin Containing Small Amounts of Aluminum, Manganese or Bismuth. *Jnl. Inst. Metals* (1935), **56**, 191.
13. D. Hanson and W. T. Pell-Walpole: The Effect of Small Additions of Tellurium on the Mechanical Properties of Tin. *Jnl. Inst. Metals* (1938), **63**, 109.
14. D. Hanson and W. T. Pell-Walpole: A Study of the Mechanical Properties of Tin-Rich Antimony-Tin Alloys. *Jnl. Inst. Metals* (1938), **63**, 87.
15. L. Seigle and R. M. Brick: Mechanical Properties of Metals at Low Temperatures; A Survey. *Trans. Amer. Soc. for Met.* (1948), **40**, 813.