

## TELLURIUM—TIN ALLOYS.

BY HENRY FAY.

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So far as the author has been able to learn, no effort has been made to make any compounds of tin and tellurium nor do any minerals containing these two elements occur in nature. The alloys of tellurium with lead<sup>1</sup>, and with antimony<sup>2</sup> have been described by the writer, and more recently the freezing point diagram for tellurium and bismuth has been worked out by Mönkemeyer<sup>3</sup>.

The tin-tellurium freezing point diagram is very similar, as might be expected, to the lead-tellurium diagram. Tin and tellurium unite to form the compound, SnTe, which melts undecomposed at 769°. This compound forms a eutectic with tellurium, which contains 85 per cent. of tellurium, and a eutectic with tin of very low concentration. In the lead-tellurium series there is the compound, PbTe, forming a eutectic with tellurium containing 78.50 per cent. of tellurium. At the time the lead-tellurium series was worked upon it was supposed that the compound formed a solid solution with lead, but this view is no longer tenable, and it is now believed that lead-telluride forms a eutectic of low concentration with lead. The reasons for this belief will be discussed later in the paper.

The method of work has been in all respects similar to that described in the previous papers on tellurium alloys, with the exception of the preparation of the metallic tellurium. It has been the custom in the past to recover the tellurium from the dried mud obtained from the Baltimore Copper Works by extraction with concentrated hydrochloric acid. On account of the large amount of silica present there was much difficulty not only in the extraction but in the separation of the tellurium from the gelatinous silica. Recently it has been found more expeditious and convenient to distill the selenium and tellurium from the dried mud in a current of hydrochloric acid gas. The chlorides of these two elements with traces of ferric chloride distill over and are subsequently separated by the method described by Norris, Fay and Edgerly<sup>4</sup>. This method involving the purification of the tellurium by means of the basic nitrate gives metallic tellurium of a high degree of purity. The whole process is very much simpler and more economical than that previously described.

The freezing points of the various alloys were determined by means of a thermo-electric pyrometer. The couple was composed of a pure platinum and a platinum ten per cent. iridium wire bound tightly together at their ends and both enclosed in thin pieces of hard glass tubing. These

<sup>1</sup> Fay and Gillson. *Am. Ch. J.*, 27, 81 (1902).

<sup>2</sup> Fay and Ashley. *Am. Ch. J.*, 27, 95 (1902).

<sup>3</sup> *Z. anorg. Chem.* 46, 415.

<sup>4</sup> *Am. Ch. J.* 23, 105.

two were again surrounded by a single piece of hard glass. The junction was standardized and calibrated in the usual manner.

A number of alloys, each weighing about twenty grams were made. The tin and tellurium were both carefully weighed, placed in a porcelain crucible, and covered with a layer of powdered charcoal to prevent oxidation. The crucibles were cautiously heated until combination took place and then the heat was raised until the whole button was completely melted. The glass tubing covering the junction was warmed in the flame and then inserted into the molten alloy, which was stirred during the cooling. In those alloys containing approximately equal amounts of the two metals the heat of combination was so great in some cases as to project some of the metal out of the crucible. In these cases it was necessary to add the tellurium slowly, and in small pieces. During the cooling process readings of the galvanometer scale were made at regular intervals and these readings were subsequently plotted. The results obtained are shown in the table and are plotted diagrammatically in Fig. 1.

Tin per cent.	Tellurium per cent.	Freezing points.	
.....	100	446°	....
2.50	97.50	438°	399°
5.00	95.00	432°	399°
10.00	90.00	419°	399°
20.00	80.00	414°	401°
30.00	70.00	494°	399°
35.00	65.00	536°	400°
40.00	60.00	634°	399°
48.23	51.77	769°	....
50.00	50.00	769°	....
60.00	40.00	761°	....
65.00	35.00	735°	232°
80.00	20.00	590°	232°
90.00	10.00	405°	233°
95.00	5.00	238°	232°
100.00	.....	232°	....

The curve shows one maximum point, indicating a compound corresponding to the composition represented by the formula,  $\text{SnTe}$ , and melting at 769°. This compound forms with tellurium a eutectic which melts at 399°, and which is composed of 85 per cent. of tellurium and 15 per cent. of tin. With tin, the compound also forms a eutectic, but the solubility of tin telluride in tin is so low that it was impossible to locate exactly the concentration of this eutectic. The melting point of this eutectic of low concentration would appear to be identical with the melting point of tin. This fact, however, does not seem to be an isolated case, as there are some other curves showing eutectics of extremely low concentration and correspondingly having their melting points coinciding with that of one of the constituents. In the lead-tellurium series a simi-

lar curve was obtained and it was thought at the time that the work was done that it indicated a solid solution of lead telluride in lead. The microstructure of both series of alloys, however, would not seem to con-

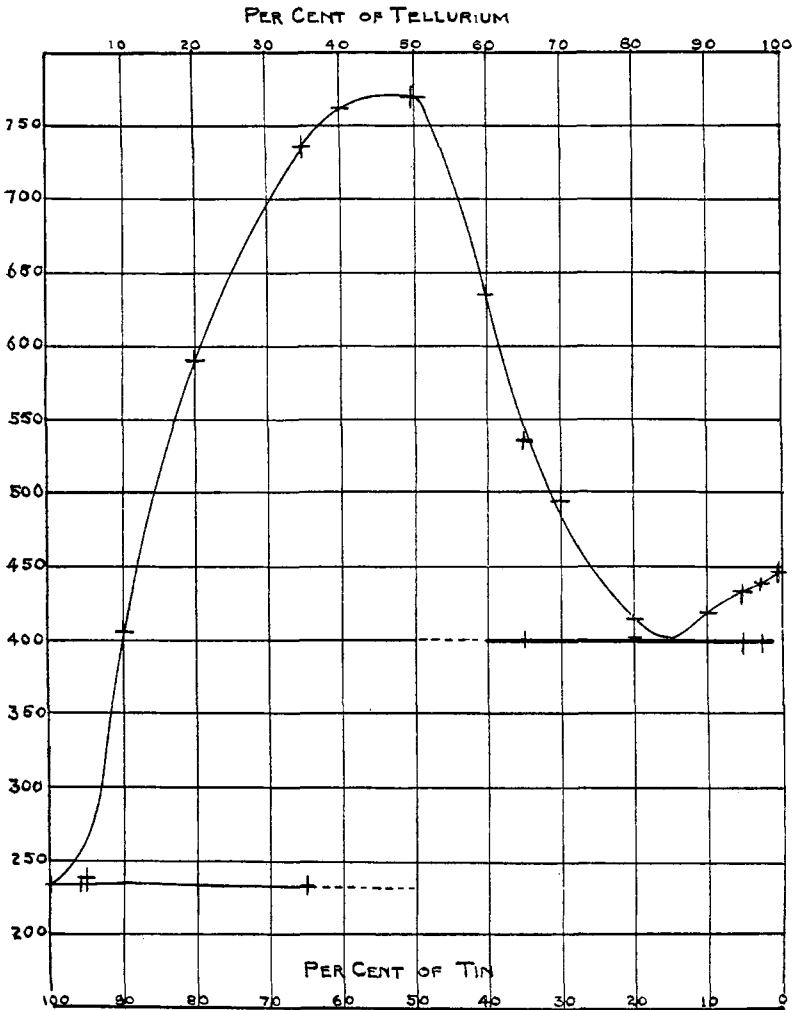


FIG. 1.

firm this belief, although it must be said on the other hand that in neither case does the eutectic structure show itself. It seems highly probable that in both cases a eutectic of low concentration actually exists. In some experiments in which buttons of definite and equal total weight with varying composition were used, the time of the separation of this eutectic was plotted and from the curve thus obtained it appeared that it

exists in the whole field between tin and tin telluride, although the thermal effect near the tin telluride concentration was very slight. That portion of the curve lying between pure tin and tin telluride was very difficult to obtain. While only a few results are given, many other experiments were made. In many cases masses of the high melting telluride would clot together and make a good clear fusion very difficult.

The presence of the compound,  $\text{SnTe}$ , is indicated not only by the fact that the maximum point in the curve coincides with the composition demanded by the formula  $\text{SnTe}$ , but it was also identified by Tammann's thermal method. A line drawn through the tie intervals of the separation of the two eutectics ends in each case at this composition.

Furthermore the compound is clearly indicated by the micro-structure. The alloy containing 48.28 per cent. of tin shows a uniform dark field when etched with nitric acid. With increasing amounts of tellurium more and more of the eutectic shows until the composition of the pure eutectic is reached. Beyond this point crystals of tellurium are found embedded in the eutectic. The microstructure of the alloys varying from 50 to 100 per cent. of tin is very unsatisfactory, as it is only after prolonged annealing that equilibrium is reached, and there is always much oxidation during the heating.

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## A STUDY OF THE CAUSES OF IMPURE NICKEL PLATE WITH SPECIAL REFERENCE TO THE IRON.

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The experiments described in the following article were performed for the purpose of ascertaining the amount of iron present in nickel plate and also to study the conditions that influence the deposition of this undesirable impurity. In the course of this work a point of considerable interest was met with and further investigated as detailed later.

It is a fact of everyday experience that nickel plate rusts on any prolonged exposure to moisture, and this rusting is pronounced to be due to small amounts of iron contained in the plate and coming from the solution during the plating process. This impurity may be conceived as constituting with the nickel a series of galvanic couples that cause rapid rusting in the presence of moisture and carbon dioxide of the air.

The universal method of plating on a commercial scale is to suspend a bar compounded of nickel and iron as anode in a bath of the double salt, nickel-ammonium sulphate, the article to be plated constituting the other pole or cathode.

The presence of the iron in the anode is due to the fact that pure nickel is not corroded rapidly enough to furnish the necessary nickel to