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A Study of the Transformation of a Twenty-Eight Per Cent Tin Bronze

Perry A. Boukind

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A STUDY OF THE TRANSFORMATION
OF A TWENTY-EIGHT PER CENT TIN BRONZE

by

Perry A. Boukind

A thesis submitted to the Department of Metallurgy in partial fulfillment of the requirement for the degree of Bachelor of Science in Metallurgical Engineering

Montana School of Mines
Butte, Montana
May 17, 1949
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Copper, undoubtedly, was one of the first metals known to man. History shows that as far back as the late Stone Age, about 13,000 B.C., some evidences of copper appear. A hunter probably picked up from glacial drift a dark brown, heavy nugget. He found that by scratching its surface it became bright, through curiosity he discovered that the nugget could be worked and formed into desired shapes. The knowledge of the existence of this new material spread and soon the demand exceeded the natural supply of the metal. A native found a small pellet of the material in a fireplace, which had been built over an oxidized outcrop, and this lead to production of the metal from its ore. The Egyptians used copper quite extensively. They used it for knives and even made tubes with it as early as 2750 B.C.

The Egyptians had some knowledge of metallurgy and soon discovered that certain alloys of copper had more attractive properties than the pure metal. Tin was one of the first metals used for alloying. A bronze mirror has been found that

1 All references are listed in the Bibliography
was probably made about 1800 B. C.\textsuperscript{1} These early metallurgists found that a certain amount of tin made the desirable alloy, too little made the alloy soft, too much made the alloy hard and extremely brittle. The reasons for this and many other behavior patterns were unexplained for many centuries. Even today there is considerable discussion on some phases of the alloy.

Metallurgists began to solve these mysteries when they were able to determine the constitutional diagram for copper and tin. There are several methods of determining the positions of phase boundaries in thermal diagrams of alloy systems. The first is by thermal analysis in which the freezing points of alloys of different composition are determined. This method is satisfactory for determining the liquidus line, but is not completely accurate for locating the solidus, and is perhaps very inaccurate for determining phase changes in the solid state. The second method is by the use of the dilatometer. The dilatometer measures the volume change with a change in temperature. As the temperature is increased there is a proportional increase in volume until a temperature is reached that produces a phase change. At this point there is a
sudden drop or rise in the curve depending on if the transformation is accompanied by an expansion or contraction of the lattice. The third and widely used method involves the use of the microscope. The procedure entails the preparation of a suitable sample by polishing and etching followed by a visual examination at a magnification that will resolve the structure. The accuracy of the microscopic method depends on the skill of the investigator and his ability to identify and distinguish the phases present in the alloy system. If studies are to be made of phases existing at elevated temperatures, a special technique is required. The alloy in question is heated to the desired temperature, held until homogenization is complete, and then quenched rapidly to room temperature in order to prevent the high temperature phase or phases from decomposing. Unless the decomposition of the high temperature phase is sluggish, it is sometimes impossible to quench rapidly enough to prevent a phase change from occurring. The fourth and probably the most exacting and positive is by the use of X-ray analysis. Each separate phase has a different crystal-line pattern and this difference is easily noted.
by X-ray studies. From the data received, it is possible to calculate the lattice parameter, which can be plotted against the atomic per cent to give a curve. From this the phase changes can be determined.

In this investigation, attention is directed to the phases covered by a 28 per cent tin alloy. When the investigation was started, consideration was given to the possibility of making a Time - Temperature - Transformation curve for this particular alloy. As the work progressed and further research was carried on, this phase of the work was abandoned. Previous investigators found that the gamma phase was unstable at room temperature. Further inquiry found considerable disagreement about the phases that were being considered. It was then decided to investigate the phase changes on an isopleth for the alloy.

"The amount of published metallurgical data concerning the system is very great. Ever since the publication of the classic researches of Heycock and Neville, whose names will always be associated with this system of alloys, papers have frequently appeared dealing with the whole or with some section of the system. Haughton, Bauer and
Vollenbruck, and Isihara have produced equilibrium diagrams showing the main phase divisions. Bauer and Vollenbruck stated that from their results it is uncertain whether a small range of solubility exists for the gamma phase. Carson gave this phase a solubility over the range 32-33 per cent tin at room temperature. Isihara on the other hand, stated that the gamma phase is a 'compound' and has no capacity for dissolving copper below 510°C., he also drew the alpha phase boundary vertical at about 11 per cent tin from 600°C.

Stockdale carried out a thorough investigation of the alpha phase boundary, for which he concluded that the range of solubility of the pure alpha phase must be extended beyond the boundary shown in the equilibrium diagram (Fig. 1) which is published in the International Critical Tables. The results of the present investigation confirm this conclusion. Stockdale did not investigate the structure of the alloys below 518°C., and he drew the alpha phase boundary vertical below this temperature. Hansen found a sudden change in the direction of the alpha boundary at 518°C., and gave the limits of sol-
ubility at 500° and 400°C. as 15.3 and 14.3 per cent tin, respectively. Matsuda, in a later paper, gave the alpha phase a vertical boundary at approximately 15 per cent tin."
PREPARATION OF THE ALLOY

Preparation of an alloy presented several problems. First, a suitable method of melting the metals had to be found. Second, some way of casting the molten alloy into a usable form was needed. The best available method of melting the constituents was an induction furnace. At first it was thought that the crucibles that could be used would not have capacity enough. An attempt was made to melt the metals in a 10-gram fire-assay crucible that was placed within the coil of the furnace, but it was not possible to get a temperature high enough to melt the constituents in this manner.

A carbon inner liner, from which the center had been machined, was placed in the furnace. The machined hole was just large enough to accommodate a thimble shaped aluminum oxide crucible. The carbon, being a conductor, was heated very rapidly by induction and in turn the copper and tin in the alundum tube was heated by conduction. This worked very satisfactorily. A melt of approximately 400 grams in weight could be melted within ten minutes. The melt was kept under a layer of
powdered charcoal to hold oxidation at a minimum. The alloy contained 112 grams of pure tin shot and 288 grams of pure copper rod and shot.

At first it was thought advisable to cast the alloy into the form of an ingot and then either hot or cold work it into the desired shape. The writer thought that this problem could give some practical experience in the casting of a semi-complicated form. It was then decided to cast the alloy into the shape desired for the samples so that specimens of the desired thickness could be cut from the casting. Two graphite blocks were used to make a suitable mold. The blocks were clamped together and one-quarter inch holes, four inches deep, were drilled on the center line. Seven such holes were drilled which gave the calculated volume of the alloy that could be melted at one time. The bottoms of the holes were joined by one-quarter inch runner to permit the molten metal to enter each opening. One end hole was enlarged to three-eighths of an inch for use as a gate or pouring hole. It was found later that the middle hole had to be used as a gate to obtain an even flow of metal to each of the seven openings.
For preheating, the mold was held together by two iron straps with bolts at each end. The mold was strapped together and placed in an electric furnace and preheated to 800°F. When the mold was thoroughly preheated and the alloy at proper pouring temperature, the metal was poured. The first casting was poured through the gate on the end, but it was found the molten metal cooled too rapidly to allow even distribution. When the molten metal was poured into the center gate, the seven openings were filled before the alloy started to freeze and the desired shape was obtained. When the mold had cooled sufficiently, the cast metal was removed and quenched. The cast metal was so brittle that even a slight blow could shatter it. Even after heating to a high temperature the alloy could not be hot-worked. The alloy was so brittle that it had to be heated to 1200°F and quenched before it could be sectioned with a hacksaw. Sections, approximately one-eighth of an inch thick, were then sawed off. The entire casting gave over one-hundred samples, one-eighth of an inch thick and one-quarter of an inch in diameter. Figure I shows the induction furnace and crucible that were used. In the foreground are the graphite mold and straps used to
hold the mold together.

The saw fillings from the samples were saved for assaying. A representative sample was taken into solution with nitric acid and then converted to a sulfate by fuming with sulfuric acid. Considerable trouble was encountered with the tin oxide that was formed. After much filtering, the tin oxide was thought to be removed, and the solution was diluted to two liters and an aliquot sample removed for analysis. The Fisher Electroanalyzer was used to plate out the copper. Apparent high gas formation took place even with a very low current density. This analysis gave a result of 69 per cent copper which was known to be low. The sample was then given to the Montana Bureau of Mines analyst, Clem Bartzen, who returned a result of 72 per cent copper.
INVESTIGATION OF THE ALLOY

Before further work could be carried on, it had to be certain that the samples were homogeneous throughout. The entire lot was placed into a crucible and annealed at $1300^\circ F$ for 72 hours in the electric furnace. The samples were quenched in an iced salt solution to insure a very rapid and drastic quench. One of the samples was immediately mounted and polished for examination. After polishing, the sample was etched in a ferric chloride solution. The first examination at about 400 magnifications lead to the belief that only one phase was present. Based on this belief, a series of investigations were planned, in an attempt to gather information for a Time - Temperature - Transformation Curve. The original diagram consulted in Dews' "Metallurgy of Bronze" (Fig. II) did not seem to offer any objection to this theory. By heating the 28 per cent tin alloy to $1300^\circ F$, the sample would be in the beta ($\beta$) phase. Then by quenching into a salt pot, that was at a temperature somewhere below $1000^\circ F$, the sample could be held for varying periods of time to allow transformation to take place. The
FIGURE II

Copper-Tin Diagram
After Dews
sample then could be water quenched, mounted, polished, and examined. It was thought that the transformation of the beta (β) into the alpha (α) and delta (δ) phases could be noted in the polished specimen.

A salt bath had to be assembled so that some method of quenching could be affected. A steel shaft with the center machined out was used for a crucible. The crucible containing the salt (Houghton draw temp 275) was placed in an electric pot furnace and cemented in place. The temperature of the salt was determined by a thermocouple placed in a fused quartz tube. The thermocouple and a millivoltmeter were calibrated in a small electric furnace containing a standardized thermocouple.

The quenched samples had apparently remained stable for sometime leading to the belief it might be possible to reheat the quenched samples from room temperature to the desired elevated temperature. The first test was at 700°F. Seven samples were heated to 700°F and held for different periods of time. The first was held for 30 seconds, the second for 60 seconds, the time interval was doubled each time up to 32 minutes. These
first samples were mounted, polished and etched with ferric chloride. The examination of these samples showed that some change had taken place before the 30 second time period had elapsed.

The second test was tried at a temperature 100 degrees lower in an attempt to slow down what was thought to be transformation. The time interval was also cut considerably, taking five second intervals this time. The first, or five second sample, was about the same as the quenched samples. The ten second sample showed some change, but it appeared to be only a coalescence of some of the particles.

At this time it seemed that the technique might be at fault. Apparently, the samples would have to be quenched from the temperature where the beta (β) phase is stable to the temperature desired for transformation. The salt pot was moved next to one of the small electric furnaces, so that the length of time needed to get the sample from the furnace to the salt pot would be at a minimum. The samples were heated to the beta range and quenched to 600°F for time intervals of five seconds, then water quenched. These samples showed no more than the second test. Assuming that the hardness of the
samples might show something, the hardnesses were determined. The samples being very brittle, had to be measured on the Rockwell 15-N scale and converted to Rockwell C scale. The results are as follows:

Quenched to 600°F from 1300°F

<table>
<thead>
<tr>
<th>No.</th>
<th></th>
<th>Time (seconds)</th>
<th>Hardness (Rc)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td>5</td>
<td>34.0</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td>10</td>
<td>38.5</td>
</tr>
<tr>
<td>3</td>
<td></td>
<td>15</td>
<td>41.0</td>
</tr>
<tr>
<td>4</td>
<td></td>
<td>20</td>
<td>42.0</td>
</tr>
<tr>
<td>5</td>
<td></td>
<td>25</td>
<td>45.0</td>
</tr>
</tbody>
</table>

The hardness increased with the time, so there was some apparent change in the structure that could not be accounted for with a microscope.

To see if a better understanding and identification of the phases could be made, a sample was heated to 1300°F, and allowed to cool very slowly, so that all the phase transformations could take place. A photomicrograph of this sample is shown in Fig. III. In an attempt to follow the phase changes on Dews' Diagram, considerable disagreement arose. The samples would not follow the phase changes as given in the diagram. A further check into some of the previous investigations made on bronzes revealed that there were
FIGURE III

Normalized Sample
Magnification 100X
Ferric Chloride Etch
at least five different diagrams, none of which were in complete agreement. At this same time, it was discovered that the beta phase, according to Dews, or gamma phase, according to Smith, is unstable at room temperature. Neither phase can be retained at room temperature by quenching the pure alloys, but a series of complex metastable transition structures are formed. This information made the making of a Time - Temperature - Transformation Curve impossible.

Because of the disagreement in the phases that were being investigated a decision was made to try and follow the transformation of the alloy from the beta phase. The difference and disagreement in diagrams can be seen by comparing Dews' diagrams (Fig. II) with the latest diagram that could be obtained (Fig. IV), taken from the 1948 Metals Handbook. For convenience, the names and designation used on the diagram of Cyril Stanley Smith (Fig. IV) will be referred to in the rest of this paper.

The samples that had been normalized (heated to 1300°F and slowly cooled) was further studied under higher magnification. The grain-boundary material appeared to be made up of two phases.
FIGURE IV
Copper-Tin Diagram
After Smith
From Metals Handbook 1948

-19-
The assumption that was made as to the cooling is as follows.

The sample was heated to 1300°F, and held until a homogeneous alloy was obtained. At this temperature, the sample was completely transformed into the gamma phase. As the cooling progressed, the gamma plus delta region was entered. The delta formed at the grain boundaries and the gamma made up the matrix. When the temperature reached 970°F, the gamma began to break down into alpha and delta. Between 970°F and 660°F the sample consisted of a matrix of alpha and delta with grain boundaries of delta. This structure remained stable to a temperature of 660°F. When this temperature was reached the delta of the grain boundaries transformed into alpha and epsilon accounting for the two-phase structure of the grain boundaries. The delta in the matrix also breaks down into alpha and epsilon. This structure is seen in the photomicrographs in Fig. V.

Further studies were made on the other samples. The as-quenched sample was examined at 1000 magnifications. The photomicrograph in Fig. VI shows that there is a complex structure of more than one phase, the exact phases that are present are not
FIGURE V
Grain-boundary Material
Magnification 1000X
Ferric Chloride Etch
FIGURE VI
As-quenched Sample
Magnification 1000X
Ferric Chloride Etch
known because identification is impossible. A similar sample was heated to 850°F and held for 24 hours and then slowly cooled. It can be seen in Fig. VII that the structure is considerably different. The sample was heated to a temperature that would convert the complex structure into alpha and delta. Upon slow cooling the alpha would remain stable, but the delta would break down into alpha and epsilon.

An attempt to identify the phases present in the as-quenched samples with the aid of X-ray crystal analysis was tried. An extensive investigation of the phases by X-ray analysis would give results by which the phases could be determined. The investigation that was planned was one of comparison of known phases with unknown ones.

The principles of X-ray analysis were covered slightly in the introduction of this paper, but it might be well to elaborate somewhat on these principles. Each crystalline element or compound has a different spacing of its atomic planes. "The X-rays were scattered from these certain planes within the crystal of metal, which contain large numbers of atoms." The angle, \( \alpha \) angles, through which the ray are scattered are always the same
FIGURE VII
Annealed Sample
(850°F for 24 hours)
Magnification 1000X
Ferric Chloride Etch
for each separate element or compound. These angles can be calculated from measurements of the X-ray film and the results substituted into the formula

\[ d = \frac{\lambda}{2 \sin \Theta} \]

where \( \lambda \) is the wave length of the radiation used, \( \Theta \) is the calculated angle, and \( d \) is the distance between the atomic planes in the crystal. From \( d \) the lattice parameter can be calculated.

In the investigation carried on, only the values of \( d \) needed to be found. The phases present at room temperature and their lattice parameters were known, therefore, a comparison of the parameters of the normalized and quenched samples could be made to determine if the gamma phase could be retained by rapid quenching. Samples of the normalized alloy and the as-quenched alloy were taken. Powders of each were mounted and X-rayed with no results. It was then decided to try a thin section of each. Sections were cut off each specimen and filed to a thickness of 0.006 of an inch. X-rays of these samples failed to give results that could be accurately calculated.

Further checking of previous X-ray analysis of bronzes showed that long periods of annealing after cold-working were necessary. The two previous samples were used for this treatment. One was to be
quenched and the other slowly cooled. The sample to be quenched was sealed in a fused quartz tube. This was thought to be necessary because of the thickness of the sample. Touching the sample with any implement would cause some cooling and the quenched condition could not be attained. After the samples were annealed for 28 hours at 1250°F, one was water quenched and the other allowed to cool slowly. These samples were mounted and X-rayed with the following results:

As-Quenched Sample

Only three measurable lines appeared on the film. Dist. is the measured distance from the center of the film to the center of the lines.

<table>
<thead>
<tr>
<th>Dist.</th>
<th>in rad.</th>
<th>in deg.</th>
<th>sin</th>
<th>2 sin</th>
</tr>
</thead>
<tbody>
<tr>
<td>x₁</td>
<td>3.82</td>
<td>15.58</td>
<td>.2681</td>
<td>.5362</td>
</tr>
<tr>
<td>x₂</td>
<td>4.86</td>
<td>19.89</td>
<td>.3402</td>
<td>.6804</td>
</tr>
<tr>
<td>x₃</td>
<td>7.45</td>
<td>30.48</td>
<td>.5072</td>
<td>1.0144</td>
</tr>
</tbody>
</table>

λ for copper target is 1.787

Substituting in formula, 

\[ d = \frac{\lambda}{2 \sin \theta} \]

\[ d₁ = \frac{1.787}{.5362} = 3.33 \]
\[ d₂ = \frac{1.787}{.6804} = 2.63 \]
\[ d₃ = \frac{1.787}{1.0144} = 1.76 \]

\( d₂ \) was the strongest line, with \( d₂ \) the next strongest, \( d₁ \) being the faintest.
Normalized sample

A considerable number of lines appeared on this film. Only the twelve brightest were taken.

<table>
<thead>
<tr>
<th>Dist.</th>
<th>in rad.</th>
<th>in deg.</th>
<th>sin</th>
<th>2 sin</th>
</tr>
</thead>
<tbody>
<tr>
<td>x₁</td>
<td>3.82</td>
<td>15.64</td>
<td>0.2698</td>
<td>0.5396</td>
</tr>
<tr>
<td>x₂</td>
<td>4.84</td>
<td>19.82</td>
<td>0.3398</td>
<td>0.6784</td>
</tr>
<tr>
<td>x₃</td>
<td>5.10</td>
<td>20.86</td>
<td>0.3560</td>
<td>0.7120</td>
</tr>
<tr>
<td>x₄</td>
<td>5.55</td>
<td>22.69</td>
<td>0.3857</td>
<td>0.7714</td>
</tr>
<tr>
<td>x₅</td>
<td>7.00</td>
<td>28.65</td>
<td>0.4794</td>
<td>0.9588</td>
</tr>
<tr>
<td>x₆</td>
<td>7.46</td>
<td>30.54</td>
<td>0.5082</td>
<td>1.0164</td>
</tr>
<tr>
<td>x₇</td>
<td>7.70</td>
<td>31.51</td>
<td>0.5227</td>
<td>1.0454</td>
</tr>
<tr>
<td>x₈</td>
<td>8.39</td>
<td>34.32</td>
<td>0.5638</td>
<td>1.1276</td>
</tr>
<tr>
<td>x₉</td>
<td>8.93</td>
<td>36.55</td>
<td>0.5955</td>
<td>1.1910</td>
</tr>
<tr>
<td>x₁₀</td>
<td>9.58</td>
<td>39.19</td>
<td>0.6319</td>
<td>1.2638</td>
</tr>
<tr>
<td>x₁₁</td>
<td>9.89</td>
<td>40.45</td>
<td>0.6487</td>
<td>1.2974</td>
</tr>
<tr>
<td>x₁₂</td>
<td>11.00</td>
<td>45.03</td>
<td>0.7074</td>
<td>1.4148</td>
</tr>
</tbody>
</table>

λ for copper target is 1.787

Substituting in formula \( d = \frac{\lambda}{2 \sin \theta} \)

\[
\begin{align*}
d₁ &= \frac{1.787}{0.5396} = 3.31^* \\
d₂ &= \frac{1.787}{0.6784} = 2.63^* \\
d₃ &= \frac{1.787}{0.7120} = 2.51 \\
d₄ &= \frac{1.787}{0.7714} = 2.32 \\
d₅ &= \frac{1.787}{0.9588} = 1.86 \\
d₆ &= \frac{1.787}{1.0164} = 1.76^* \\
d₇ &= \frac{1.787}{1.0454} = 1.71 \\
d₈ &= \frac{1.787}{1.1276} = 1.58 \\
d₉ &= \frac{1.787}{1.1910} = 1.50 \\
d₁₀ &= \frac{1.787}{1.2638} = 1.41 \\
d₁₁ &= \frac{1.787}{1.2974} = 1.38 \\
d₁₂ &= \frac{1.787}{1.4148} = 1.26 \\
\end{align*}
\]

The order of intensity from the strongest to the weakest are:

\( d₁, d₄, d₅, d₆, d₂, d₁, d₈, d₁₀, d₉, d₄, d₁₂ \)

The values marked with an asterisk are identical with those found in the as-quenched sample.
At first the other nine lines in the normalized sample were quite puzzling until it was found that there might be a slight amount of oxidized material present. A check soon showed that all the lines that were unaccounted for were copper oxide lines.

From this data it was concluded that the as-quenched sample, although totally dissimilar under the microscope, had the same phase present as the normalized sample.
CONCLUSIONS

"The names and designations of alloys are a problem. The problem is to remember them. Perhaps one of the Quiz Kids could do it in his unaided head, but most metallurgists and engineers require a shelf of reference books and catalogs to keep the 10,000 alloy names in order."

The above was found very true in the investigation carried on by the writer of this paper. Of the many papers consulted in research, only a few agree in the naming of the phases. Two methods or systems have been suggested to eliminate the confusion and to standardize the naming of the phases. In one the phases of the same crystal structure are denoted by the same Greek letter in all diagrams containing a phase of that structure. The other is a method based on the chemical composition. Neither of these methods are free from objections. Both have been applied to a restricted group of nonferrous alloys. Each of the methods could probably be expanded into a general system. Adoption of either of the above mentioned methods or any other suitable system would be valuable in research.
The investigation covered by this paper brought to light no new theories on the Copper-Tin system, but did clear up much of the indecision that existed. The results obtained showed that the gamma phase, immediately after cooling, decomposes and therefore cannot be maintained at room temperature. The microstructure differs radically from that of the normalized sample. It will be remembered that in quenching the samples in the molten salt the hardness increased as the time in the molten salt increased. This might be explained by saying that the complex structure is a fine mechanical mixture of the alpha and epsilon phases. As the annealing time in the pot increased, the like particles tended to coalesce. This coalescing gave larger areas of the brittle epsilon, which in turn hardened the structure.

Further investigation of the system would prove extremely interesting. Some investigators claim the break down of delta into alpha and epsilon is extremely slow. The assumptions in this paper would only hold true with a fairly rapid break down. A measurement of the speed of this break down would make an interesting problem. Much of this work would have to be carried on at relatively high temperatures, making construction of equipment another problem.
X-ray studies have brought out much in the system. Microscopic work would give pictures of what happens. The latter method is probably most accessible to the small commercial users of bronze who could use such information.
ACKNOWLEDGEMENT

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under whose direction the work was performed.


