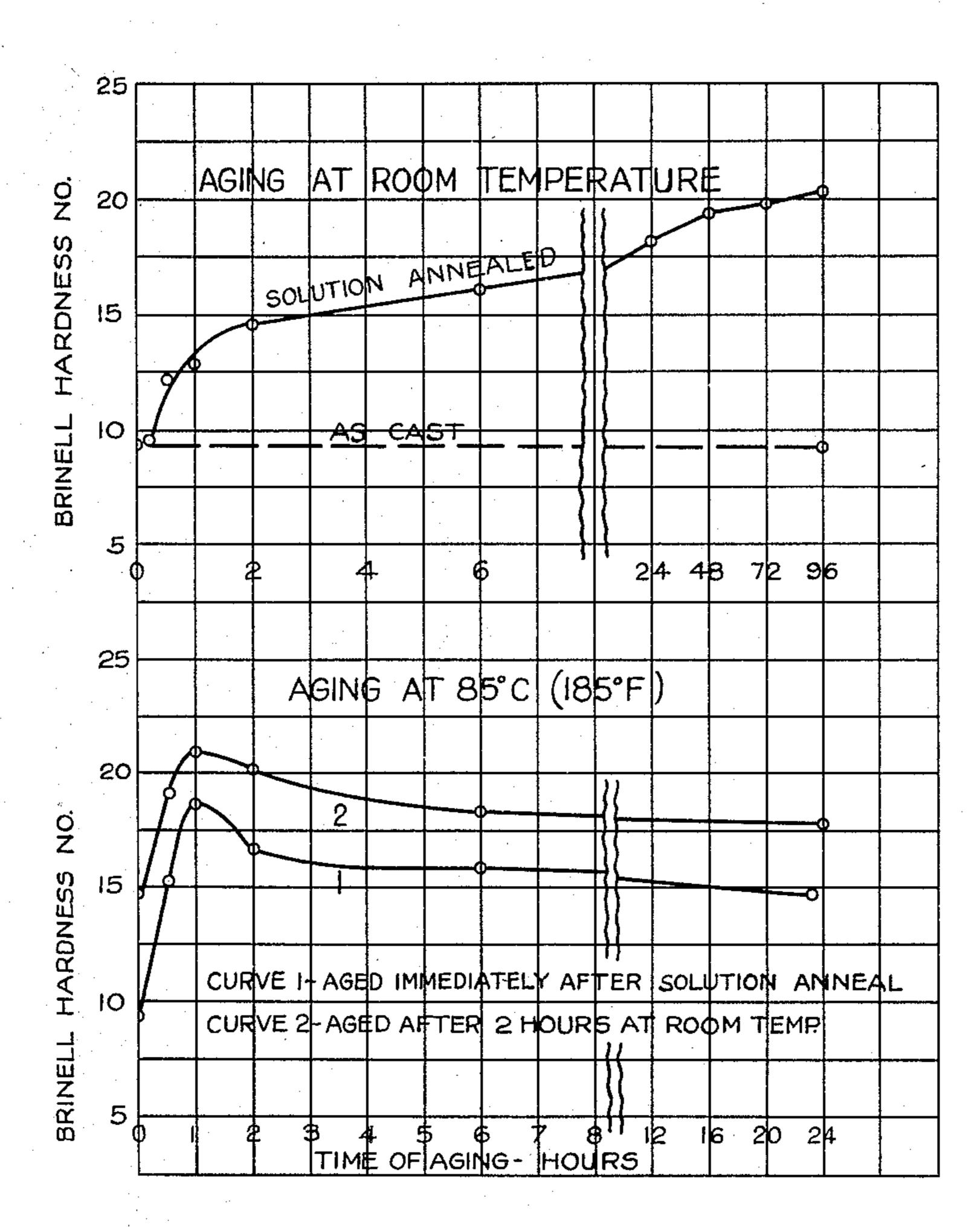
AGE-HARDENING LEAD BASE ALLOYS Filed July 22, 1937



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## AGE-HARDENING LEAD BASE ALLOYS

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8 Claims. (Cl. 148—21.4)

My invention relates to lead base alloys. It has to do particularly with lead base alloys containing either antimony or antimony and tin. It pertains, more particularly, to an alloy of this type whose properties have been improved by special heat treatments and to the method of heat-treating such alloys to bring about such improvement.

It is known that lead base alloys containing antimony or antimony and tin may be agehardened by suitable treatments. In the main, this treatment, as practiced in the prior art, has consisted in heating the alloy nearly to the melting range, then quickly cooling and thereafter holding the alloy at room temperature for a specified period of time which causes the alloy to slowly harden.

Some investigative work has been done and published on the age-hardening of antimonial 20 lead in this maner, but very little has been done on the hardening of the lead-antimony-tin alloys and this only recently. Within the last few years, the advantages of hardening electrotypes backed with lead containing a few percent of 25 antimony and tin have become known and several electrotypers have started age-hardening their products on a commercial scale, effecting the final step of hardening by holding at room temperature for 24 hours or more. However, this 30 treatment has the disadvantage of delaying production, owing to the requirement that the alloy be held at room temperature for 24 hours or more in order to attain a satisfactory gain in hardness.

One object of this invention is to provide a method of effecting age-hardening or precipitation-hardening of a metal of the type in question which will greatly reduce the time required for effecting the age-hardening process.

Another object of this invention is to provide an alloy of the type in question for use in making electrotypes which possesses a greater degree of hardness than that hitherto attainable by the known methods of age-hardening for a day or two at room temperature.

Various other objects of this invention will appear as this description progresses.

In its preferred embodiment, my alloy consists primarily of lead containing a small amount of antimony and tin. My preferred method of treatment of this alloy contemplates heating it nearly to the melting range of the alloy, rapidly cooling it, as by quenching in water, and then reheating to a much lower temperature than the initial heating temperature. It has been

found that, by this method, the attainment of the hardening effect can be greatly accelerated. Moreover, by pausing for a short time after cooling, as by holding the metal at room temperature for an hour before giving it the final heat 5 treatment to effect accelerated hardening, a degree of hardness may be attained which is markedly higher than that produced by heating immediately after quenching.

A comparison between a lead base alloy con- 10 taining antimony and tin, treated by a prior art method and treated by my methods, with particular respect to the time periods and temperatures of final aging treatment is illustrated in the graph shown in the accompanying drawing 15 which also shows the Brinell hardness of the alloys resulting from the various treatments.

To illustrate the advantages obtained by my invention, an alloy of 97% lead, 2% antimony and 1% tin was heated for a half hour at 235 20 degrees C. in an oil bath, quenched in water, and given the following treatments:

- 1. Allowed to remain at room temperature without further treatment.
- 2. Reheated immediately in a hot water bath, <sup>25</sup> held at 85 degrees C.
- 3. Allowed to remain at room temperature for 2 hours, then heated in a water bath at 85 degrees C.

Results of this series of tests are shown in the graph. By normal room temperature age-hardening, the quenched alloy remains unchanged for at least 15 minutes and then gradually hardens from about Brinell 9 to Brinell 20 in 96 hours. On accelerating this action by treatment at 85 degrees C. a hardness of about Brinell 18 is reached within an hour, or a slightly higher hardness than is attained in 24 hours at room temperature. By pausing for 2 hours before heating at 85 degrees C., however, a marked increase in hardness to over Brinell 21 is obtained.

Further investigations have shown that the pause before accelerated aging, which is necessary to give the maximum hardness, is somewhat 45 less than an hour. A longer time does no harm, but is unnecessary.

The time of accelerated aging treatment varies with the temperature used. As shown in the graph, when aging at 85 degrees C. one hour is 50 ample to reach the maximum hardness. Prolonged treatment at this temperature gives the slight softening action of over-aging which is normal in most age-hardening systems. By heating at only 65 degrees C., a longer time is 55

required to reach maximum hardness (as 2 hours for the alloy to which the graph relates), but over-aging takes place very slowly and to a very limited extent. When heating at 100 degrees C. or in boiling water, only about 10 minutes are needed to attain maximum hardness. but this hardness is not quite as great as can be obtained by a longer treatment at slightly lower temperatures and over-aging takes place 10 more rapidly. The treatment to be used can be varied, as a consequence, to meet the conditions desired.

In heating the cast metal before quenching, maximum hardness is attained by heating as close 15 to the melting range of the alloy as it is practical to get. A cast lead alloy of 2% antimony and 1% tin, for example, may be readily heated to 250 degrees C. without changing the surface of the casting, whereas a lead alloy of 3% anti-20 mony and 3% tin should not be heated over about 235 degrees C. The time of this initial heat treatment should be sufficient to permit all of the antimony and tin to go into solid solution with the lead. It will be obvious that the length 25 of this heating period will vary with the temperatures used, which may vary from 235 degrees C. to 250 degrees C., depending upon the particular alloy. A half hour is usually ample for castings such as electrotype or stereotype plates. 30 The type of heating apparatus used is of no consequence in this heat treatment as long as reasonable uniformity is attained. Oven heating is satisfactory, for example, although for rapidity and convenience water baths for temperatures 35 below 100 degrees C. and oil baths for higher temperatures are preferable.

The lead-base alloys capable of receiving this improved hardening treatment are, in general, any alloy of lead and antimony, or lead, anti-40 mony and tin, which will age harden, as the term is broadly used in metallurgy. Any leadbase alloy containing from 1 to 10 per cent antimony or containing from 1 to 10 per cent antimony and 0.5 to 10 per cent tin may be hardened according to my method. A large excess of antimony or antimony and tin over that which will go into solid solution at the solidus line diminishes the hardening effect of the heat treatment. Thus, a greater increase in hardness is attained 50 by this improved heat treatment on a 97% lead, 2% antimony, 1% tin alloy than on a 94% lead, 3% antimony, 3% tin alloy, but as long as some increased hardness is obtainable by an aging treatment the improved treatment as outlined 55 can be advantageously used. The added hardness effect of a pause at room temperature of an hour or two before aging at an elevated temperature is present with an alloy of lead and antimony only as well as with a lead-antimony-tin 60 alloy.

As is well known, very small amounts of impurities in the lead-base alloys may have a marked effect on the hardness and other properties. Such impurities as alkali or alkaline earth 65 metals, arsenic, copper, selenium, tellurium, silver and nickel may be present as impurities in the raw materials used in making the alloy or may be added purposely to effect a desired change in the properties of the alloy. The presence of 70 any one or more of such impurities up to 0.5 per cent may affect the age-hardening properties of the lead-antimony or lead-antimony-tin alloys without detracting from the application of this invention.

Certain physical properties other than hard-

ness are changed by my age-hardening processes, as for example, tensile strength and elongation. The hardness change is taken as typical, however, and exemplifies the changes that take place in the alloy by my methods of heat treatment.

As a particularly practical application of my improved methods of heat treatment, electrotype plates may be markedly improved in their ability to stand up under severe service, by hardening in accordance with my methods. The electrotype 10 backing metal is normally a lead-base alloy containing 2% to 5% antimony and 1% to 4% tin. After casting this metal as a backing for the electro-deposited electrotype shell, the entire plate may be heated from 15 minutes to a half 15 hour at a temperature of 235 degrees to 250 degrees C., the time, periods, and temperatures depending upon the composition of the backing metal. It may then be quenched in water and finished. Then, it may be hardened by immersing 20 it in boiling water for a period of ten minutes. Thus, it will be seen that the prior art step of maintaining the backing metal at room temperature for 24 hours or more to effect age-hardening thereof is dispensed with and, by my methods, 25 the plates may be hardened in an extremely short time.

When that form of my method which involves the pausing for about an hour or more before final heating to effect age-hardening is used, 30 this period of pause may be substantially consumed by the usual finishing operations, such as shaving the plate to size and correcting imperfections. As a matter of fact, these finishing operations frequently consume an hour or more, 35 so that the period of pause which occurs at this time does not detract from the economic advantages of my methods.

Where several hours may elapse before finishing, the plate may age-harden at room tempera- 40 ture sufficiently to hinder some of the correcting operations. In this case, the entire heat treatment may be given after the plates have been finished, or the hardening tendency of the plate may be largely prevented until desired by keep- 45 ing the plate cold by refrigeration or immersion in dry ice, or by similar means.

This method of hardening electrotype plates may be applied to stereotype plates also, in those cases where the stereotype metal used is amenable 50 to age hardening.

Though I have indicated that during the pause of approximately one hour or more which precedes the final heat treatment, the alloy should be held at room temperature, it will be under- 55 stood that similar results may be obtained while varying this temperature somewhat. Thus, the temperature at which the alloy is held may be anything less than 50 degrees C. without departing from the scope of my invention.

It will be seen from the above that I have produced a novel lead base alloy, that is, a lead base alloy containing antimony or antimony and tin which, because of the particular methods of treatment thereof, has an enhanced degree of hardness 65 so that it is rendered more suitable for use in electrotype plates and in stereotype plates. It will also be seen that I have devised novel methods of treatment which not only greatly shorten the process of age-hardening the alloy but which 70 makes possible the attainment of a greater degree of hardness than that attainable by prior art methods. Numerous other advantages will appear from the preceding description and the appended claims.

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Having thus described my invention, what I claim is:

1. A method of age-hardening a lead-base alloy containing antimony and tin which comprises heating such alloy to a temperature to bring the antimony and tin into solid solution in the lead, cooling with sufficient rapidity to retain the said solid solution state, holding the alloy thus cooled at room temperature for at least an hour, and then reheating it to effect age-hardening of the alloy.

2. A method of age-hardening a lead-base alloy containing from 1% to 10% antimony and from 0.5% to 10% tin which comprises heating such alloy to a temperature to bring the antimony and tin into solid solution in the lead, cooling with sufficient rapidity to retain the said solid solution state, holding the alloy thus cooled at room temperature for at least an hour, and then reheating it to effect age-hardening of the alloy.

3. A method of age-hardening lead-base alloys containing antimony and tin and containing up to 0.5% of an element from the group consisting of copper, nickel, arsenic, alkali, or alkaline earth metals, selenium, tellurium, and silver which comprises heating such an alloy to a temperature to bring the antimony and tin both into solid solution in the lead, cooling with sufficient rapidity to retain the said solid solution state, holding the alloy thus cooled at room temperature for at least an hour, and then reheating it to effect agehardening of the alloy.

4. A method of age-hardening a lead-base alloy containing antimony and tin which comprises heating such alloy to a temperature to bring the antimony and tin into solid solution in the lead, cooling with sufficient rapidity to retain the said solid solution state, holding the alloy thus cooled at room temperature for at least an hour, and then reheating at temperatures between 50 and 100 degrees C. to effect age-hardening of the alloy.

5. A method of age-hardening a lead-base alloy containing from 1% to 10% antimony and from

0.5% to 10% tin which comprises heating such alloy to a temperature to bring the antimony and tin into solid solution in the lead, cooling with sufficient rapidity to retain the said solid solution state, holding the alloy thus cooled at room temperature for at least an hour, and then reheating at temperatures between 50 and 100 degrees C. to effect age-hardening of the alloy.

6. A method of age-hardening lead-base alloys containing antimony and tin and containing up to 10 0.5% of an element from the group consisting of copper, nickel, arsenic, alkali, or alkaline earth metals, selenium, tellurium, and silver which comprises heating such an alloy to a temperature to bring the antimony and tin into solid solution in 15 the lead, cooling with sufficient rapidity to retain the said solid solution state, holding the alloy thus cooled at room temperature for at least an hour, and then reheating at temperatures between 50 and 100 degrees C. to effect age-harden-20 ing of the alloy.

7. A method of age-hardening a lead-base alloy containing from 1 per cent to 10 per cent antimony which comprises heating such alloy to a temperature to bring part or all of the antimony 25 to solid solution in the lead, cooling with sufficient rapidity to retain the said solid solution state, holding the alloy thus cooled at room temperature for at least an hour, and then reheating to effect the age-hardening of the alloy.

8. A method of age-hardening a lead-base alloy containing from 1 per cent to 10 per cent antimony and containing up to 0.5 per cent of an element from the group consisting of copper, nickel, arsenic, alkali or alkaline earth metals, selenium, tellurium and silver which comprises heating such an alloy to a temperature to bring a part or all of the antimony into solid solution in the lead, cooling with sufficient rapidity to retain the said solid solution state, holding the alloy thus cooled at room temperature for at least an hour, and then reheating it at temperatures between 50 and 100 degrees C. to effect age-hardening of the alloy.

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