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Tilman et al.

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[54] **METHOD OF PRODUCING CREEP RESISTANCE OF PB-SB ALLOYS**

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[52] **U.S. Cl.** .... **148/11.5 R**

[51] **Int. Cl.** **C22c 11/00**

[58] **Field of Search** .... 148/11.5 R; 75/166 B

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[57] **ABSTRACT**

Lead-antimony alloys of improved creep resistance are prepared by means of a process comprising finish deformation at elevated temperature with light, i.e., less than 10 percent reductions.

5 Claims, No Drawings

METHOD OF PRODUCING CREEP RESISTANCE OF PB-SB ALLOYS

Lead-antimony alloys containing about 1 to 9 percent antimony are in common usage for a variety of applications such as chemical processing equipment, sound barriers, radiation shielding, roofing and flashing. Conventional processing procedures commonly produce usable, although not high, tensile strength in these alloys. Such procedures generally do not produce desirably high creep resistance. It has now been found, according to the invention, that creep resistance of Pb-Sb alloys containing up to about 5 percent Sb may be substantially improved by the use of a processing procedure comprising breakdown deformation, cold or hot, with greater than about 10 percent reduction per pass for about 67 percent, or greater, reduction of original thickness, followed by finish deformation of less than 10 percent per pass, and preferably about 5 percent per pass or less, at a temperature of about 100° to 200°C.

The alloys of the invention consist essentially of lead and up to about 5 percent of antimony. The process of the invention has been found to be particularly beneficial in treatment of alloys containing about 0.25 to 2.0 percent antimony. Deformation will generally be by means of rolling, but other means such as press forming may also be used. These procedures, and the equipment employed, are conventional in the art of metal working. Rolling speed and lubricants are also conventional, and are not critical. Reheating between passes need be only long enough to restore the elevated temperature, and the workpiece is normally air cooled after the last rolling pass.

The following examples will serve to more particularly illustrate the invention and the advantages achieved thereby.

EXAMPLES

Samples were initially prepared by means of the following procedure:

Sixteen pound heats of lead-antimony alloys consisting of corroding grade lead and antimony of 99.99 percent purity were melted in an induction furnace using MgO crucibles. The lead was brought to the molten condition and a $ZnCl_2$ flux cover added. The required amount of antimony was added through the flux cover at approximately 420°C. After cooling to approximately 400°C, NaCl was added to the flux cover to thicken the flux and facilitate removal. The flux was skimmed and the molten alloy was cast at approximately 400°C into two preheated (about 300°C) steel slab molds. Upon removal from the molds, the slabs measured 0.69 inch × 4 inches × approximately 7 inches and weighed approximately 8 lbs. each. A ¼ inch slice was cut from the riser end of each slab and discarded. Samples for chemical analysis were then filed from both riser and butt ends of each slab and each sample analyzed separately for antimony content.

By rolling, slabs were cut in half, each half measuring 0.69 inch × 4 inches × 3 inches to 3.5 inches. The slabs were preheated at either 125° or 175°C for 1 hour. Reduction per pass was 20 percent based on the thickness obtained after the preceding pass. Kerosene was used as a lubricant. The slabs were reheated for 10 to 15 minutes between each pass. Five passes at 125° or 175°C were sufficient to reduce the slabs to 0.225 inch to 0.227 inch × 4 inches × 9 inches to 9.5 inches. These slabs were cut in half to 0.225 inch to 0.227 inch × 4 inches × 4.5 inches to 4.75 inches long and used as starting stock for finish rolling.

The final 26 percent reduction (based on original thickness of 0.69 inch) was made at either room temperature, 125° or 175°C at consecutive reductions of either 5 percent per pass or 20 percent per pass based on the thickness obtained after each preceding pass. When rolling at 125° or 175°C, the plate was preheated for 1 hour at the selected temperature and reheated for 10 to 15 minutes between each pass. Final dimensions were approximately 4.125 inches × 18 inches × 0.0625 inch. Standard 8 inches sheet tensile and creep speciments were cut longitudinally from the finished sheet.

Table 1 gives creep data for Pb-Sb alloys that were finish rolled at 5 to 20 percent reduction per pass at 125°C. The marked improvement in creep rates for the 5 percent reductions is apparent. Furthermore, the improvement is greatest for the alloy having the lower antimony content.

TABLE 1

Sb, wt. pct.	Reduction per pass, pct.	Creep rate, pct/yr[1]
0.25	5	9
.25	20	321
2.0	5	9
2.0	20	25

[1] 700-psi stress, tests at 90±3°F

Table 2 lists creep data, in percent per year, for Pb-Sb alloys containing up to 5 percent Sb, that were processed in various ways including the process of the invention, after breakdown rolling in the range of 125° to 175°C.

Column II of the table gives data for alloys finish rolled cold, i.e., near room temperature, for 26 percent reduction based on starting thickness. This procedure is similar to commercial practice, which is done at various reductions per pass.

Columns III and VI give data for alloys finish rolled at 175°C for 26 percent reduction based on starting thickness, for 5 and 20 percent reduction per pass, respectively.

Column IV gives data for alloys finish rolled at room temperature at 20 percent reduction per pass prior to annealing at 220°C, and with a final pass at 175°C and 5 percent reduction.

Column V gives data for alloys finish rolled at room temperature at 20 percent reduction per pass prior to solution treatment at 235°C and aging 10 to 12 days at 90°F.

As seen from the data of table 2, there is a vast difference in creep rates between alloys finish rolled cold and those finish rolled hot with light passes. The data of column III, where repeated light passes were given at elevated temperature, shows very low creep rates, i.e., high creep resistance, and creep rates about the same for alloys containing 0.5 to 5.0 percent Sb. In contrast, rolling at 175°C with 20 percent reduction per pass does not result in low creep rate, as shown in column VI.

The data in column IV show that a single pass at 175°C and 5 percent reduction does not provide maximum benefits, although the Pb-1.0 Sb alloy is approaching the rate for repeated passes (column III).

In table 3 are compared some tensile properties to further illustrate advantages to processing according to the invention. The tensile and yield strengths are substantially superior to those for material finish rolled cold, while maintaining good ductility (elongation), and are especially good in conjunction with the excellent creep properties.

TABLE 2

I Sb, wt pct	II Finish rolled cold, Reduction per pass 5 pct	II Finish rolled cold, Reduction per pass 20 pct	III Finish rolled at 175°C, 5 pct reduction per pass	IV Annealed 220°C, last pass at 175°C, 5 pct reduction	V Solution treated at 235°C, aged 10 to 12 days 90°F	VI Finish rolled at 175°C, 20 pct reduction per pass
0	126	253	71	104	—	266
0.25	71	151	17	32	158	279
.50	77	118	4	21	45	255
1.0	94	241	3	6	11	136
2.0	149	160	4	12	7	94
5.0	456	1054[1]	3	151	5	3.6[1]

[1]Rolled at 10 pct reduction per pass to avoid cracking.

TABLE 3

Property	1.0 pct Sb Finish rolled cold[1]	1.0 pct Sb Invention[2]	2.0 pct Sb Finish rolled cold[1]	2.0 pct Sb Invention[2]	5.0 pct Sb Finish rolled cold[1]	5.0 pct Sb Invention[2]
Tensile strength, psi	2890	3310	3180	3940	3630	4930
Yield strength, psi (0.2% offset)	1470	2160	1920	2920	2320	4080
Elongation in 2 inches, pct	47	44	47	37	62	34

[1]At room temperature. Last 26 pct total reduction at 5 pct per pass.
[2]Finish rolled at 175°C. Last 26 pct total reduction at 5 pct per pass.

We claim:

1. A method for treatment of lead-antimony alloys, containing up to about 5 percent antimony, comprising (1) breakdown deformation of the alloy with reductions per pass greater than about 10 percent for a total reduction of at least about 67 percent of the original thickness and (2) finish deformation of the alloy at a temperature of about 100° to 200°C and with reduction per pass less than 10 percent.

2. The method of claim **1** in which the alloy contains about 0.5 to 2.0 percent antimony.

3. The method of claim **1** in which the deformation is accomplished by rolling.

4. The method of claim **1** in which the finish deformation is accomplished at a temperature of about 125° to 175°C.

5. The method of claim **1** in which the finish deformation is accomplished at a reductions per pass of about 5 percent.

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