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**THE EFFECT OF CASTING VARIABLES AND HEAT TREATMENT
ON THE TENSILE PROPERTIES OF LEAD-0.02 TO 0.10 WEIGHT
PER CENT CALCIUM ALLOY**

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UNION CARBIDE NUCLEAR COMPANY

DIVISION OF UNION CARBIDE CORPORATION

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- OAK RIDGE GASEOUS DIFFUSION PLANT • OAK RIDGE Y-12 PLANT
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ABSTRACT

Some of the controllable variables have been investigated in casting of lead alloys having 0.06 to 0.09 weight per cent calcium. The alloy has been found to respond to solution heat treatment, room temperature precipitation hardening, and accelerated hardening at 100°C. Tensile strengths to 7,000 psi have been obtained.

TABLE OF CONTENTS

I. INTRODUCTION	7
II. SUMMARY	9
III. MATERIALS	11
IV. THE EFFECT OF CALCIUM CONTENT ON PHYSICAL PROPERTIES	11
V. HEAT TREATMENT OF THE ALLOY	12
A. COOLING RATE	12
B. SOLUTION HEAT TREATMENT	13
C. ROOM TEMPERATURE AGING FOLLOWING SOLUTION HEAT TREATMENT	14
D. AGE HARDENING AT 100° FOLLOWING SOLUTION HEAT TREATMENT	15
E. DOUBLE AGING	16
VI. TENSILE STRENGTH AND CALCIUM ANALYSIS VARIATIONS WITHIN A CHILL CAST ALLOY SLAB	17
VII. TEST METHODS	20
VIII. CONCLUSIONS	22
IX. ACKNOWLEDGMENTS	25

1

2

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I. INTRODUCTION

Alloys of calcium and lead having a nominal calcium content of 0.02 to 0.10 weight per cent have been prepared. A series of experiments have been made to determine over what limits a number of the controllable casting conditions can be varied and still produce castings with adequate physical properties.

Since the addition of calcium to the lead makes it age-hardenable, the variables examined were those expected to be most critical - calcium content and heat treatment.

The exact mechanism of age hardening has not yet been completely defined; but empirically, the process has been well examined. The hardening mechanism in the lead-calcium alloy depends upon a decrease in solid solubility of calcium in the lead as the temperature is lowered. Such a condition is shown by the boundary line AB in Figure 1, a portion of the lead-calcium system investigated by Schumacher and Bouton.* Secondly, the alloy must be cooled through the temperature range over which this decrease in solubility takes place at a rate faster than the kinetics of the system will allow equilibrium conditions to be maintained. A supersaturated solution must be developed. Once the nonequilibrium conditions have been established, the material will harden with time. The rate at which the hardening takes place and the extent is dependent, among other things, upon the aging temperature. If the aging temperature is too high and the time too long, the alloy will over-age or again become soft. This represents a nearly complete return to equilibrium conditions by reduction of the supersaturation initially developed within the alloy.

Figure 1. Constitution Diagram of Lead-Calcium Age-Hardening Area

*Bouton, E. E., and Schumacher, G. M., Metals and Alloys, Volume 1, pp. 405-9 (1930).

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II. SUMMARY

Castings with section thicknesses up to 4 inches have been made from a nominal 0.07 weight per cent calcium-lead alloy. It has been found that when the alloy has as much as 0.05 weight per cent calcium in it, it easily develops strengths in excess of 4,000 psi. As the nominal calcium content drops to the 0.03-0.02 weight per cent level, the resulting alloy tensile values drop rapidly.

An alloy part was cast in such a way as to develop a variety of freezing and cooling rates within the casting. The resulting varying tensile strengths were then examined with respect to the cooling rates. Tensile strengths in excess of 4,000 psi were found to develop after 8 hours at 100°C when the alloy was cooled through the temperature range 600°F to 300°F in 30 minutes or less. Strengths up to 6,000 psi were obtained when the cooling time for this range was dropped to 2.6 minutes.

The alloy was found to respond to solution heat treatment. Times from 2 to 16 hours were found adequate for treatment at 300°C. When water quenched, the solution-treated alloy hardened at room temperature in about 24 hours. Accelerated aging at 100°C for 8 hours gave tensile strengths superior to those developed at room temperature. Preliminary room temperature aging followed by accelerated aging was not demonstrated to be better than immediate accelerated aging.

A slab cast from the alloy was cut into tensile samples and analyzed for calcium content. It was found that the tensile strength varied within the casting in a manner reflecting the rate of chill of the metal after casting. The calcium content was found to be uniform throughout the part.

Some tensile determinations were made at +165°F and -65°F, with strength of about 4,000 psi and 6,000 psi, respectively, being obtained.

Hardness was found to give a regular but not too precise a correlation with the tensile strength.

III. MATERIALS

The alloys in this work were prepared from Doe Run Lead, a premium grade of corroding lead produced by the St. Joseph Lead Company. The calcium was added as a 3 weight per cent calcium-lead master alloy prepared by the National Lead Company.

IV. THE EFFECT OF CALCIUM CONTENT ON PHYSICAL PROPERTIES

Figure 1 shows that at 300°C about 0.07 weight per cent calcium is soluble in lead. At room temperature the solubility has dropped to about 0.01 weight per cent. This is the range of decreasing solubility which gives the lead-calcium alloy its age-hardening properties. The diagram will show that at a calcium content of 0.02 weight per cent, very little supersaturation can be developed. At the 0.07 weight per cent level considerable amounts of calcium will remain in solution if the alloy is rapidly cooled.

An examination of the available tensile strength data that can be correlated with analytical values is shown in Figure 2. The analytical data are derived from spectrographic methods and have a precision of about ± 100 per cent. The tensile values are usually good to ± 100 psi. It can be seen from Figure 2 that the tensile strength increases with increasing calcium content through the range of calcium percentages examined. The recent development of a simple, more precise calcium analysis will enable more quantitative information to be gained on the effect of calcium content on the physical properties of the alloy.

Figure 2. Tensile Strength Versus Calcium Content

V. HEAT TREATMENT OF THE ALLOY

A. COOLING RATE

Since the degree of supersaturation of the alloy (and thus the hardenability) is a function of the cooling rate through the range of decreasing solid solubility, an experiment was conducted to determine how supersaturation and resulting tensile strength of the alloy was affected by the cooling rates met within normal casting practice.

A casting - about 37 inches tall, and with a wall thickness of 3 inches - was made from an alloy having a nominal composition of 0.08 weight per cent calcium. The alloy was prepared and poured at 524°C into a heated steel mold. The mold was then quenched with water from the lower end in such a manner as to develop a series of different cooling rates within the casting. The time-temperature curves were obtained from thermocouples imbedded in the casting. After freezing, the casting was artificially aged by heating at 100°C for 8 hours, a procedure found to develop maximum hardness. The part was then sectioned and tensile samples taken from heights within the part corresponding to the zones whose temperatures were monitored by the thermocouples.

Figure 3 is a plot of the tensile strength versus time required to cool the alloy between 316°C and 149°C. This plot shows the dependence of the tensile strength upon the rate of cooling through the region of decreasing solid solubility. The first three sets of data in Figure 3 represent the strength obtained in the region where the mold was rapidly flooded with water.

The fourth set of data is from samples taken from the casting at the upper level of water cooling. The fifth set of data represents a slower solidification and cooling from a region above the water-cooled portion of the mold.

Any reasonable casting procedure employing controlled directional freezing should

Figure 3. Tensile Strength of Lead Alloy Samples Cooled at Various Rates Through Temperature Range 600°F to 300°F (316°C to 149°C) and Then Artificially Aged at 100°C for 8 Hours

permit cooling at a rate faster than that rate found in the fifth zone, and thus should yield parts with tensile strengths in excess of 4,700 psi. Considerably slower cooling rates (sixth set of data) should still yield tensile strengths in excess of 4,250 psi. Care must be taken, however, to assure that the sample blank is taken from sound metal. Table I gives the complete listing of tensile data on the samples taken in this experiment. Samples H-1 and H-2, while appearing to be sound metal, gave low tensile values and zero elongation. This stresses the necessity for adequate excess stock at the casting head.

TABLE I
TENSILE STRENGTH OF CALCIUM-LEAD ALLOY SAMPLES FROM A LARGE CASTING

Sample Number	Ultimate Tensile Strength (psi)	Yield Strength 0.2% Offset (psi)	Elongation 2-Inch Gauge Length (%)	Time to Cool Through 600°F to 300°F (minutes)
A-1	6,150	4,700	32	2.6
A-2	5,950	--	29	2.6
A-3	6,000	--	33	2.6
B-1	5,500	4,300	39	3.0
B-2	5,350	--	40	3.0
B-3	5,550	--	38	3.0
C-1	5,350	4,000	43	3.1
C-2	5,350	--	34	3.1
C-3	5,300	--	34	3.1
D-1	5,100	3,800	39	4.0
D-2	5,150	--	38	4.0
D-3	5,300	--	35	4.0
E-1	4,700	3,200	39	11.0
E-2	4,750	--	38	11.0
E-3	4,650	--	35	11.0
F-1	4,400	2,950	42	29.0
F-2	4,250	--	37	29.0
F-3	4,350	--	40	29.0
G-1	4,400	2,750	48	39.0
G-2	4,050	--	35	39.0
G-3	4,150	--	45	39.0
H-1	1,800	--	0	39.0
H-2	1,650	--	0	39.0

B. SOLUTION HEAT TREATMENT

If the necessary degree of supersaturation of the calcium has not been maintained at the time of casting by an adequate cooling rate through the critical solubility range, this condition can be established by solution heat treatment. This procedure consists of holding the alloy

at a temperature corresponding to the degree of increased solubility desired, until such time as the precipitated calcium compound has gone into solid solution in the lead. When near equilibrium conditions have been established at this elevated temperature, the alloy is quenched at a rate adequate to develop the required supersaturation. To determine the time necessary to effect a solution of the precipitated compound, a series of experiments were run using the subsequent hardening of the alloy as a measure of the completeness of the treatment.

A casting was made from a nominal 0.09 weight per cent Ca-Pb alloy in such a manner as to approach equilibrium cooling. This casting was allowed to cool slowly along with the mold, crucible, and furnace in which it was made. No direct measurement of the cooling rate was made, but several hours were probably required to solidify the 8-inch diameter by 18-inch long ingot and 10 to 15 hours to cool it below 150°C .

Figure 4 shows how four samples of this casting hardened following solution heat treatment at 300°C for 1/2, 1, 2, and 16 hours, and then water quenching. In a second test, tensile bar blanks were solution heat treated for 1, 2, 4, 8, and 16 hours. Table II gives the data, and Figure 5 gives a plot of that data relating the tensile strength at 30 days to the solution treatment time. The time-tensile strength correlation is not good but indicates that 16 hours at 300°C is most certainly adequate.

C. ROOM TEMPERATURE AGING FOLLOWING SOLUTION HEAT TREATMENT

Tensile bar blanks were water-quenched after solution treatment at 300°C for 16 hours. Curve A of Figure 6 gives the increase in hardness as a function of time at room temperature. It appears that the hardness obtained at 32 days is Brinell 14 ($\sim 5,700$ psi), and 95 per cent of that strength was reached in 4 to 8 hours.

Figure 4. Hardness vs Time at Room Temperature Following Water Quench After Solution Treatment at 300°C for Indicated Length of Time

TABLE II
EFFECT OF SOLUTION HEAT-TREAT TIME ON TENSILE STRENGTH

Sample Number	Time at 300°C (hours)	Ultimate Tensile Strength (psi)	Elongation 2-Inch Gage Length (%)
1-4	0	3,719	42.0
1-15	0	3,617	36.0
1-14	0	3,781	39.5
1-2	0	3,709	35.5
1-12	1	4,166	23.5
1-8	1	4,406	22.0
1-6	1	4,344	27.5
1-11	2	5,759	32.0
1-16	2	5,557	28.0
1-9	2	5,451	35.0
1-1	2	5,451	26.0
2-11	4	5,000	40.7
2-12	4	5,500	42.5
2-13	4	5,500	43.0
2-14	8	5,600	44.5
2-15	8	5,200	41.8
2-16	8	5,800	48.0
2-1	16	5,900	37.0
2-2	16	5,600	42.0

D. AGE HARDENING AT 100°C FOLLOWING SOLUTION HEAT TREATMENT

Samples were solution heat treated at 300°C for 16 hours and water quenched. They were then aged at 100°C. Samples were removed after various times at temperature, cooled, and tested for hardness. A new sample was used for each different time at temperature. The hardness of the samples at the time of withdrawal from the 100°C aging are plotted as Curve B, Figure 6. The hardness of the samples with further aging at room temperature did not change significantly. The maximum hardness reached in this test was BHN 18.5 (~7,500 psi).

E. DOUBLE AGING

Earlier experiments* on lead-calcium alloys found that preliminary room temperature aging for 24 hours followed by accelerated aging at 100°C produced a hardness greater than that

*Bouton, E. E., and Schumacher, G. M., Metals and Alloys, Volume 1, pp. 405-9 (1930).

Figure 5. Solution Heat Treat Time vs Tensile Strength for Lead-0.08 Weight Per Cent Calcium

Figure 6. Change of Hardness with Time Following Different Heat Treatments

obtained by immediate treatment at 100°C. To determine if this applied to the present alloy, a series of tensile bar blanks were solution heat treated for 16 hours at 300°C, water quenched, aged 24 hours at room temperature, and then aged at 100°C for varying lengths of time. After the test time at 100°C, the samples were cooled and tested for hardness. Curve C of Figure 6 is a composite curve showing the average hardness of the tensile bar blanks during the 24-hour room temperature aging, and then the hardness of the individual samples at the end of the indicated length of time at treatment.

The hardness values of the samples did not change significantly with additional aging at room temperature. It would seem that within the precision afforded by the hardness determination, that there was no advantage in the double aging.

Curves B and C indicate that after two days at 100°C, the alloy begins to over-age or soften again.

Table III is a listing of tensile values obtained from the bars used to derive Curves A and C.

TABLE III
TENSILE STRENGTH OF LEAD-0.8 WEIGHT PER CENT CALCIUM AFTER SOLUTION TREATMENT
AT 300°C FOR 16 HOURS, WATER QUENCHED, AND VARIOUS AGING TREATMENTS

Sample No.	Age Treatment	Tensile Strength (psi) ^a	Elongation (%)	Brinell Hardness No.
1 }	Aged at room temperature	5,900	37	14.1
2 }		5,600	42	14.3
3 }	Aged at room temperature 24 hours, 24 hours at 100°C, balance at room temperature	6,600	27	16.2
4 }		6,800	29	17.3
5 }	Aged at room temperature 24 hours, aged 48 hours at 100°C, balance room temperature	6,550	29	16.7
6 }		7,050	28	18.0
7 }	Aged at room temperature 24 hours, aged 120 hours at 100°C, balance at room temperature	6,400	28	16.7
8 }		6,600	33	16.9
9 }	Aged at room temperature 24 hours, aged 192 hours at 100°C, balance at room temperature	6,250	31	15.5
10 }		6,200	33	16.5

^a Tensile values determined at 17 days total time after solution heat treatment.

VI. TENSILE STRENGTH AND CALCIUM ANALYSIS VARIATIONS WITHIN A CHILL CAST ALLOY SLAB

A casting was made to determine how the tensile strength and calcium content varied at different locations within the casting when that casting was chilled at a rate fast enough to give adequate strength (as determined in Section V-A) yet a slow enough rate to allow progressive, directional solidification. The controlled directional solidification is necessary to produce a casting with high radiographic soundness and free from extensive pipes or shrinks.

The melted 0.06 weight per cent calcium alloy was poured into a preheated, steel slab mold 24 inches wide, 4 inches thick, and 25 inches high. The mold and its contents were then chilled by gradually flooding the mold with water from below. The time required to raise the chilling water from the bottom to the top of the mold was about one minute. The slab was stripped from the mold and sawed vertically to give four similar sections 5 inches wide, 4 inches thick, and 24 inches tall. The ends of the billet were discarded. Each section was then partially sawed into 1-inch-square by 5-inch-long tensile bar blanks. Each blank was numbered as shown in Figure 7. The section from which the bar was taken was indicated by the addition of the initial number 1, 2, 3, or 4 to the location number.

Corresponding samples were taken from seven positions in each section. The blanks were machined into 0.505 tensile bars and tested for ultimate tensile strength and per cent elongation.

Table IV gives the results of these tests.

Figure 8 shows a chart of the tensile values obtained in each of the four sections plotted against their probably increasing strength, estimated from their position in the part and the relative chill rates that existed during the cooling. Also shown on Figure 8 are the results of six tests made at 165°F and five tests made at -65°F. Table V lists the results of these 11 tests.

Analytical samples were taken from some of the same specimens reported for tensile values. These were analyzed for calcium

Figure 7. Side View of Slab Section Showing Ends of 1-Inch-Square by 5-Inch Long Tensile Bar Blanks

TABLE IV
PHYSICAL TESTS OF Pb-0.06 WEIGHT PER CENT CALCIUM ALLOY TEST SLAB

Location Number	Section Number							
	1		2		3		4	
	Tensile Strength (psi)	Elongation (%)	Tensile Strength (psi)	Elongation (%)	Tensile Strength (psi)	Elongation (%)	Tensile Strength (psi)	Elongation (%)
06	4,550	35	4,600	38	4,650	36	4,350	33
12	5,200	25	5,700	28	5,500	28	5,100	35
15	5,200	27	5,750	27	5,500	32	5,200	29
23	5,400	32	5,550	28	5,700	28	6,000	30
24	5,800	25	5,750	28	5,800	29	6,200	24
26	4,400	21	4,650	30	4,950	23	4,450	26
27	4,000	40	4,450	33	4,600	30	4,700	31

content by a flame photometric method. The analytical results of 18 samples indicated that the calcium content of the part was virtually homogeneous.

Table VI summarizes the analytical results, giving the averages for the determinations made on the positions shown.

The alloy slab sectioned and tested in this strength distribution study had not been given the heat treatment at 100°C, but had only been room temperature age hardened. Since Figure 6 shows that superior strength can be obtained by this accelerated aging, some additional specimens of the slab were heat treated to see how the strength of the casting may have been increased by heat treatment.

Figure 8. Tensile Strength vs Position Arranged According to Estimated Increasing Strength Based on Probable Chill Rate

TABLE V
TENSILE TESTS OF Pb-Ca ALLOY MADE AT +165°F AND -65°F

Sample Number	Test Temperature	Ultimate Tensile Strength (psi)	Yield Strength 0.2% (psi)	Elongation - 2 Inches (%)
102	+165°F	3,959	3,061	--
114	+165°F	4,327	3,449	29
120	+165°F	3,801	2,551	32
302	+165°F	3,750	2,802	44
314	+165°F	4,168	3,188	34
320	+165°F	4,477	3,502	29
202	-65°F	5,492	3,477	28
214	-65°F	6,224	4,056	25
220	-65°F	6,224	4,133	23
402	-65°F	5,469	3,443	30
420	-65°F	6,454	4,286	28

The strength increases expected were of the same general magnitude as the variations within the part, so 12 samples were chosen in such a way as to give estimates of the tensile strength before heat treatment to compare with the values found after. Hardness measurements were made, before and after heat treatment, and are shown in Figure 9. Four samples were given no heat treatment. Four were solution treated 16 hours at 300°C and aged 8 hours at 100°C. The third set was simply aged 8 hours at 100°C. Each of the three

groups of specimens had two samples chosen from the lower strength areas of the casting and two from the higher strength areas. It would appear that the best results were obtained by the simple accelerated aging, and further that, the weaker areas of the casting were strengthened most by this treatment.

Tensile bars were machined from the heat-treated blanks and tested. These data are shown in Table VII. In Table VII an estimate of the original tensile strength has been made. This estimate is based upon the strength pattern revealed by the 28 samples taken in the initial examination. The tensile results support the hardness indications that the simple 100°C aging is the better of the two treatments and most effective on the weaker portions of the casting.

VII. TEST METHODS

The Brinell hardness numbers used in this work were obtained using a Rockwell hardness tester with a 1/2-inch steel ball for a penetrator, and a 60-kilogram load applied for 45 seconds. The depth of penetration as indicated by the Rockwell dial was used as a measure of the impression diameter. The hardness number obtained correlated generally with the corresponding tensile values where they have been obtained. Figure 10 shows this correlation.

TABLE VI
ANALYSIS OF TEST SLAB FOR CALCIUM

Sample Location	Average Per Cent Calcium $\pm$ LE of the Mean
26 and 27	0.0610 $\pm$ 0.0006
12 and 15	0.0594 $\pm$ 0.0010
23 and 24	0.0610 $\pm$ 0.0013
06, 24, and 27	0.0609 $\pm$ 0.0010
23 and 26	0.0611 $\pm$ 0.0006
All samples	0.0604 $\pm$ 0.0006

Figure 9. Hardness of Calcium-Lead Alloy as a Function of Time and Treatment

TABLE VII
TENSILE STRENGTH OF HEAT-TREATED SAMPLES FROM TEST SLAB

Specimen Number	Heat Treatment	Tensile Strength Before Treatment Estimated (psi)	Tensile Strength After Treatment (psi)
105	No heat treatment	4,750	4,900
303		4,750	4,450
221		5,800	4,950
418		5,250	5,400
205	Solution treated 16 hours at 300°C, water quenched, aged 8 hours at 100°C	4,750	5,150
405		4,500	5,400
118		5,250	5,350
318		5,800	5,120
103	Aged 8 hours at 100°C	4,750	6,200
403		4,750	6,250
121		5,800	6,300
421		6,000	6,300

The tensile tests were made on conventional 0.505 test specimens with shoulder grips. The tests at low and elevated temperatures were made in a controlled temperature chamber and were tested after 30 minutes time in the chamber.

VIII. CONCLUSIONS

It has been found that the alloy will strengthen best when heat treated at 100°C immediately following the casting and cooling (or after solution treatment). Alloys of the nominal composition of 0.06 to 0.09 have been hardened thus to 7,000 psi with modest heat treatment. Chill rates of the castings need not be so rapid as to preclude good casting technique. A cooling rate of 11 minutes across the range of 600°F to 300°F still gave an alloy with strengths of 4,600 to 4,800 psi.

Figure 10. One-Half Inch Steel Ball, 60 Kilogram Load, 45 Seconds

Per cent elongation of the material is uniformly good. Where low tensile strength values are accompanied by low elongation values, the indications are that the sample has not been taken from a region of sound metal.

IX. ACKNOWLEDGMENTS

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