SHORT-TIME HEAT TREATING CHARACTERISTICS OF A BINARY COPPER-BERYLLIUM ALLOY

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James Robert Burnett
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THESIS

This is to certify that the

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SHORT-TIME HEAT TREATING CHARACTERISTICS OF A BINARY COPPER-BERYLLIUM ALLOY

by

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TABLE OF CONTENTS

INTRODUCTION

Within the last forty years (13), a new field has opened to the metallurgist and the engineer by the discovery of a treatment which can be applied to a large number of alloys and by which their properties can be changed radically. This treatment has been used most effectively with the light alloys of aluminum and the heavy alloys of copper, iron, nickel, and gold. Since it is dependent upon the time as well as temperature, the term age hardening is commonly used. However, this gives no indication to the actual phenomenon - that of precipitating a constituent from a supersaturated solid solution. It is a well known fact that this precipitation is hastened in many alloys by an increase in temperature, sometimes from days or weeks at room temperature to minutes or hours at an elevated temperature. An application of this as applied to the copper-beryllium system lead to the current investigation.

Beryllium-copper alloys may be classified on the basis of high strength or high conductivity. Since the high conductivity alloys are usually supplied by the mill in the fully hardened condition (11), the following discussion will be limited to the high strength alloys. At the present time most manufacturers of these alloys are supplying the heat treating procedures. While the equilibrium diagrams show quite a wide variation in allowable temperature, most commercial recommendations are 1400°F - 1500°F for solution heat treatment (1, 2, 4, 7, 8, 10, 12) and 525°F - 700°F for precipitation hardening (1, 2, 3, 6, 7, 8, 10, 12). However, Hildebrandt (9) found that in low beryllium (2.4% or less) alloys the rate of precipitation of the gamma phase was much more rapid at increased temperatures giving rise to the possibility of using considerably higher temperatures when aging the low beryllium alloys. Since hardness is a function of time and temperature, the time required for precipitation should be reduced by such an increase in temperature.

Starting from this, it was decided to investigate the short-time-high-temperature heat treating characteristics of a binary copper-beryllium alloy. A 2% beryllium alloy was selected as one representative of about the center of the useful beryllium range. Starting at the highest point of the usual temperature range, samples were aged at 700°F, 750°F, 800°F, 850°F, 900°F. The time of aging was 2 to 64 minutes for those samples at 700°F and 750°F and 1 to 32 minutes for those at 800°F, 850°F, and 900°F. Following the usual heat treating procedures, the allowance in temperature variation was set at ±5°F (11). In order to get maximum rate of heat transfer, a salt bath was used.

In his investigation, Hildebrandt also found that variations in solution heat treating temperature had no noticeable effect on the time-hardness curve so all samples were solution heat treated at 1450°F which is the center of the usual solution heat treating range. A salt bath was also used in this operation to get maximum rate of heat transfer as some of the samples were heated for as little as 2 minutes.

DISCUSSION

In attempting to show similarities between the precious stones, emerald and beryl, the French investigator Vauquelin (3) found an unknown constituent, common to both stones, that was believed to be one of the rare earth elements. Because of its property of forming sweetish-tasting salts, it was called glucinum, a name still retained in French scientific circles. However, in most countries it has given was to the name beryllium.

It was not until 1828 that the German scientist Wöhler (3) first isolated beryllium from its salts by heating its chloride with flat pressed balls of calcium. Then very little work was done until Le Beau (3) instigated new methods of producing beryllium by electrolysis. In addition to his research on the production of beryllium Le Beau investigated several beryllium alloys including that of copper along with those of chromium, tungsten, molybdenum, and carbon.

However, it did not get beyond the laboratory stage until about 1925 (5). Masing in Germany studied the phenomena of precipitation hardening of copper-aluminum alloys and concluded that it was a function of solubility varying widely with temperature. Since a phase diagram of copper-beryllium published about 10 years earlier indicated this alloy had the right type of solubility-temperature relationship for his investigation, he began working with it. Thus Masing proved his theory was valid and as a by-product discovered the age-hardening properties of copper-beryllium.

About two years prior to this, work was done in this country with copper-beryllium as a possible substitute for tin-copper or zinc-copper. However, the high cost of extracting pure beryllium stood in the way of commercial production.

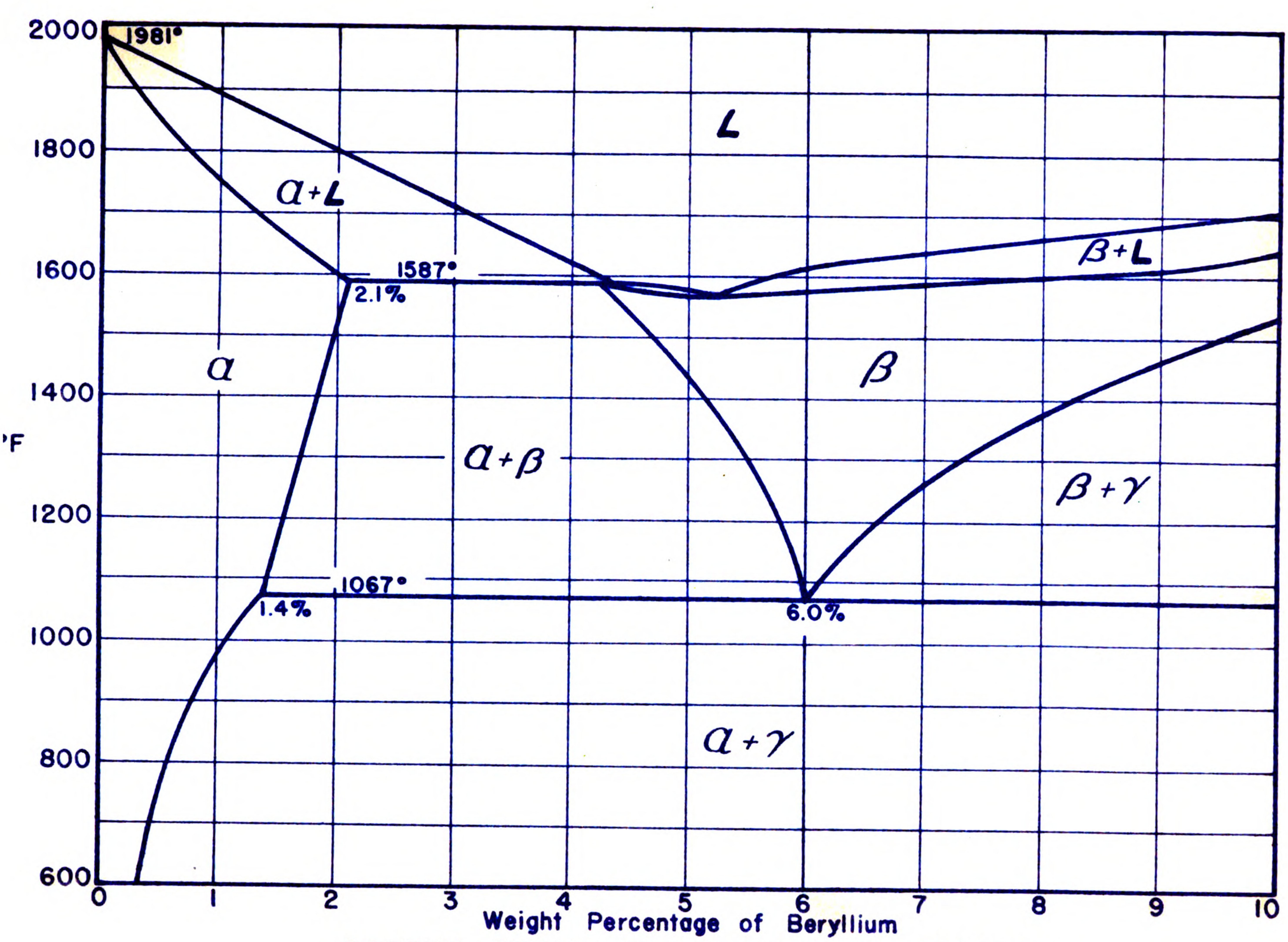
With the news of Masing's discovery, the picture was changed. Copper-beryllium was no longer a substitute alloy. During the following few years, the technical and popular press gave copper-beryllium quite a play. It became a "wonder metal" and a great and glowing future was freely prophesied for this alloy.

About this time a much lower cost process was developed for getting the beryllium into copper by converting beryllium oxide, powdered carbon, and powdered copper into a "master alloy" of about 4% beryllium. This could then be remelted with additional copper to get the composition desired.

Laboratory research by J.K. Smith (5) in 1932 on an alloy containing about 2.50% beryllium confirmed the unusual properties obtained by precipitation hardening and established initial mill procedure. However, most of the laboratory work had been done on standard test bars of relatively large section and the effect of size, mass, and cold work was not given proper weight.

Since the announcement of Smith's work, the properties of copper-beryllium have led to a number of applications, many successful and some disappointing. Overenthusiasm and insufficient data in some instances have led to failure. Today, however, the available data and information on physical properties and heat treatment are more complete, and successful applications are found in a variety of products. Copper-beryllium is not only as corrosion-resistant as brass and phosphor bronze, but also is nearly twice as strong. It is non-magnetic. It has endurance strength nearly equal to steel; higher under corrosive conditions.. Its electrical conductivity is nearly twice that of bronze and it will maintain spring properties under temperatures 100°F higher than bronze. In addition, it has less tendency to drift or take a set than almost any other spring material. Since it is hardened by heat-treatment after forming, copper-beryllium parts can be made in forms and shapes impossible to duplicate in many other alloys.

The copper rich section of the presently accepted copper-beryllium equilibrium diagram is given on the following page (14). Considering this diagram for the case of small cast parts (which would cool rapidly), it may be assumed that the structure would



COPPER-BERYLLIUM EQUILIBRIUM DIAGRAM

be of alpha dentrites with the harder beta constituent at the boundaries. Such a partially dispersed condition increases the ease with which a satisfactory solution heat treatment can be made and also prevents the formation of undesirable beta segregations (3).

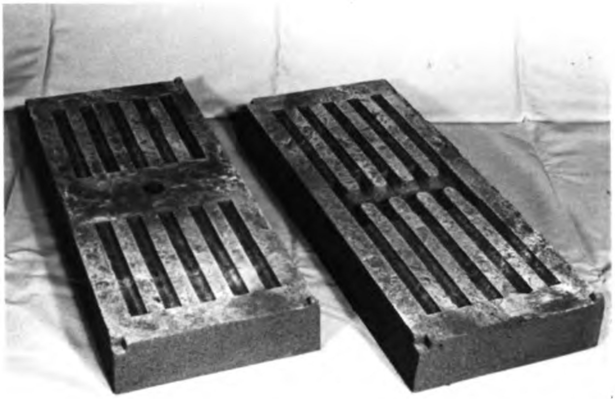
After a super-saturated solid solution is formed by quenching from the alpha solid solution range, comes the problem of precipitation hardening the alloy. Another look at the equilibrium diagram shows that this temperature could be as high as 1067°F. However, since time is of critical importance, the hardening temperature is somewhat lower, the requirement being that the time and temperature must be so regulated as to give an optimum precipitation of the gamma phase. In accordance with the accepted theory of hardening, there is an optimum particle size which causes the maximum amount of keying action on the potential slip planes with a resulting maximum increase in hardness. The time and temperature must be such that there will be just sufficient atomic mobility to produce this size.

PROCEDURE

The 2% copper-beryllium alloy was made from electrolytic copper punchings and a master copper-beryllium alloy of 3.98% beryllium. These were melted together in an electric induction furnace and poured in core sand molds made by mixing 100 parts of lake sand, 10 parts of cereal binder, 5 parts of water, and 5 parts of oil binder. These green molds were baked in a gas furnace at approximately 500°F for 24 hours and, when cool, pasted together and baked again for about 8 hours. A test mold from a mixture containing smaller quantities of cereal binder and oil binder was found to give very rough surfaces on the cast bars.

The mold box,

and thus each mold



was made so as to yield ten bars approximately 6 1/2 inches long and 5/8 inches in diameter. Since each mold required about 9 3/4 pounds of metal and the capacity of the furnace was only 12 pounds, it was necessary to make a separate heat for each mold. It was decided that 30 bars would be necessary, requiring three heats, the calculations for which follow:

Heat #1

weight of master alloy 2.823 kg.

$2.823 \times 0.0398 = 0.11236$ kg. weight of Be

$0.11236 \div 0.02 = 5.618$ kg. total heat

$5.618 \times 0.98 = 5.506$ kg. weight of Cu

$5.506 - (2.823 - 0.112) = 2.795$ kg. weight of Cu required

Heat #2

weight of master alloy 2.241 kg.

2.241 x 0.0398 = 0.08919 kg. weight of Be

0.08919 ÷ 0.02 = 4.460 kg. total heat

4.460 x 0.98 = 4.372 kg. weight of Cu

4.372 - (2.241 - 0.089) = 2.220 kg. weight of Cu required

Heat #3

weight of master alloy 2.044 kg.

2.044 x 0.0398 = 0.08135 kg. weight of Be

0.08135 ÷ 0.02 = 4.068 kg. total heat

4.068 x 0.98 = 3.987 kg. weight of Cu

3.987 - (2.044 - 0.081) = 2.024 kg. weight of Cu required

It was found by Hildebrandt that the calculated composition of such castings agreed with the actual composition within the normal accuracy of analytical methods of analysis. Therefore, no such quantitative analysis was made.

The castings were allowed to cool in the mold, were then broken out, and the test bars machined to $0.50^{+0.01}_{-0.00}$ inches in diameter. In order to have a sample that would come to temperature quickly, they were cut into wafers of about 1/8 inch thickness on a water-cooled abrasive cut-off wheel and drilled with a #54 drill near one edge in order to accept a 18 gauge Nichrome wire for handling in the salt bath and quench.

The solution heat treating was done in a salt bath at 1450°F (± 10°F) for 2, 4, 7, 11, 16, and 22 minutes and the quenching in the tap water at about 60°F. The aging was done at 700°F and 750°F for 2, 4, 8, 16, and 32 minutes and at 800°F, 850°F, and 900°F (all ± 5°F) for 1, 2, 4, 8, 16, and 32 minutes. The aging was arrested by quenching in water. During both the solution heat treating and the aging operations, the samples were agitated for one minute after insertion into the bath, then for about 5 seconds at 2 minute intervals, and for 20 seconds immediately prior to quenching.

A clean surface on each sample was obtained by rubbing on wet #180 emery paper and the penetration hardness determined on the B and/or C Rockwell scale. In order to make a graph on one scale, both B and C hardness values were taken on samples of 90-100 R_B and the average of the differences used as the conversion factor. This gave $R_B = R_C + 77$.

Photomicrographs (p. 26) were taken at 100X of the following samples: as cast, solution heat treated for 4 minutes, solution heat treated for 16 minutes, and several representative aged samples. The etchant used was 5 parts $FeCl_3$, 10 parts HCl, 100 parts H_2O (by volume).

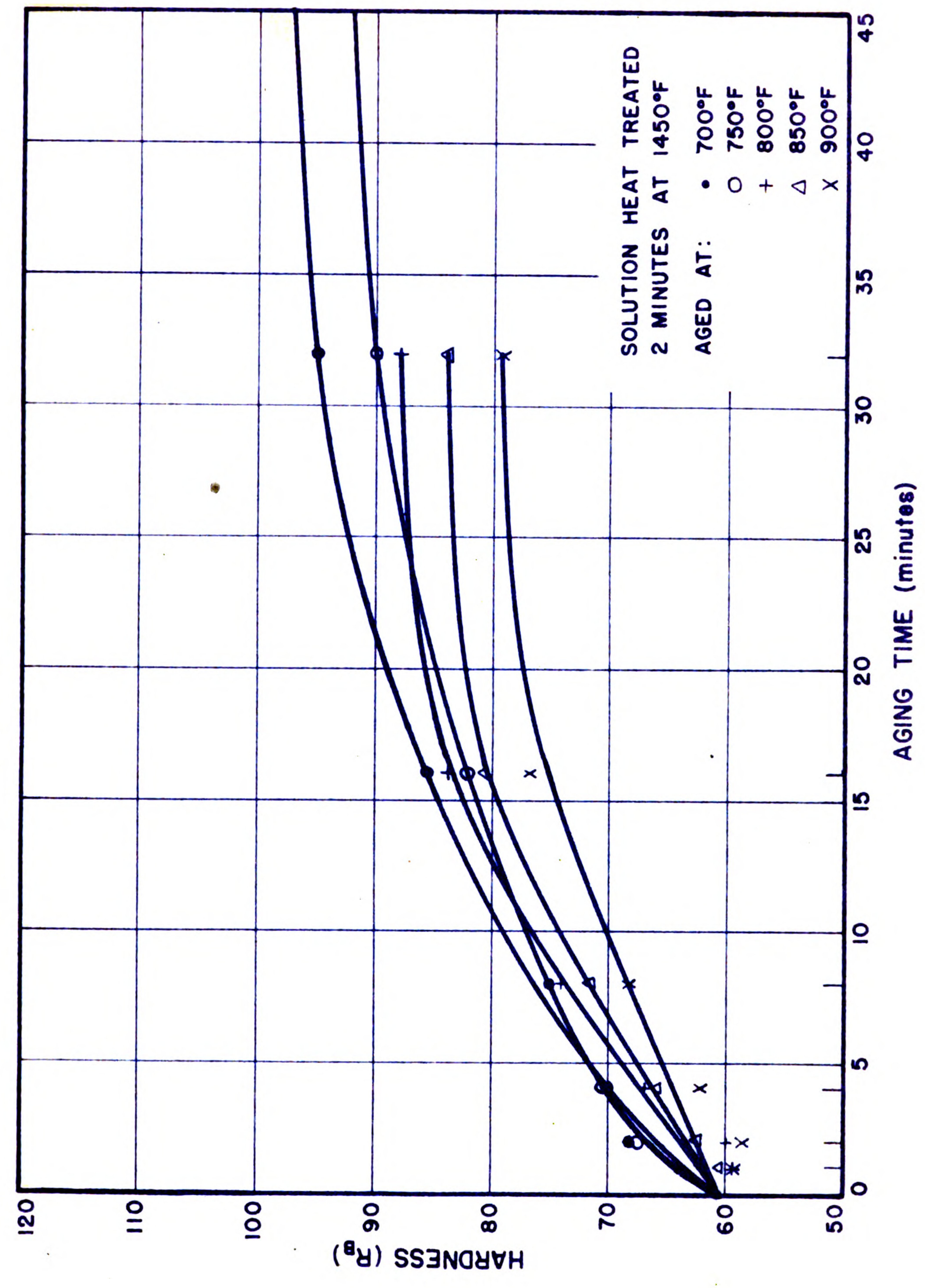
Rockwell$_B$ hardness values of a 2% copper-beryllium binary alloy solution heat treated for 2 minutes at 1450°F and aged as indicated.

All values are the averages of eight readings, four being taken on each of two samples.

Time of Aging (minutes)

Temp. of Aging (°F)	0	1	2	4	8	16	32	64
700	61		68	70	75	86	95	25*
750	61		67	71	75	82	90	96
800	61	59	60	67	74	84	88	
850	61	61	63	66	72	81	84	
900	61	59	58	63	68	77	79	

* Rockwell$_C$

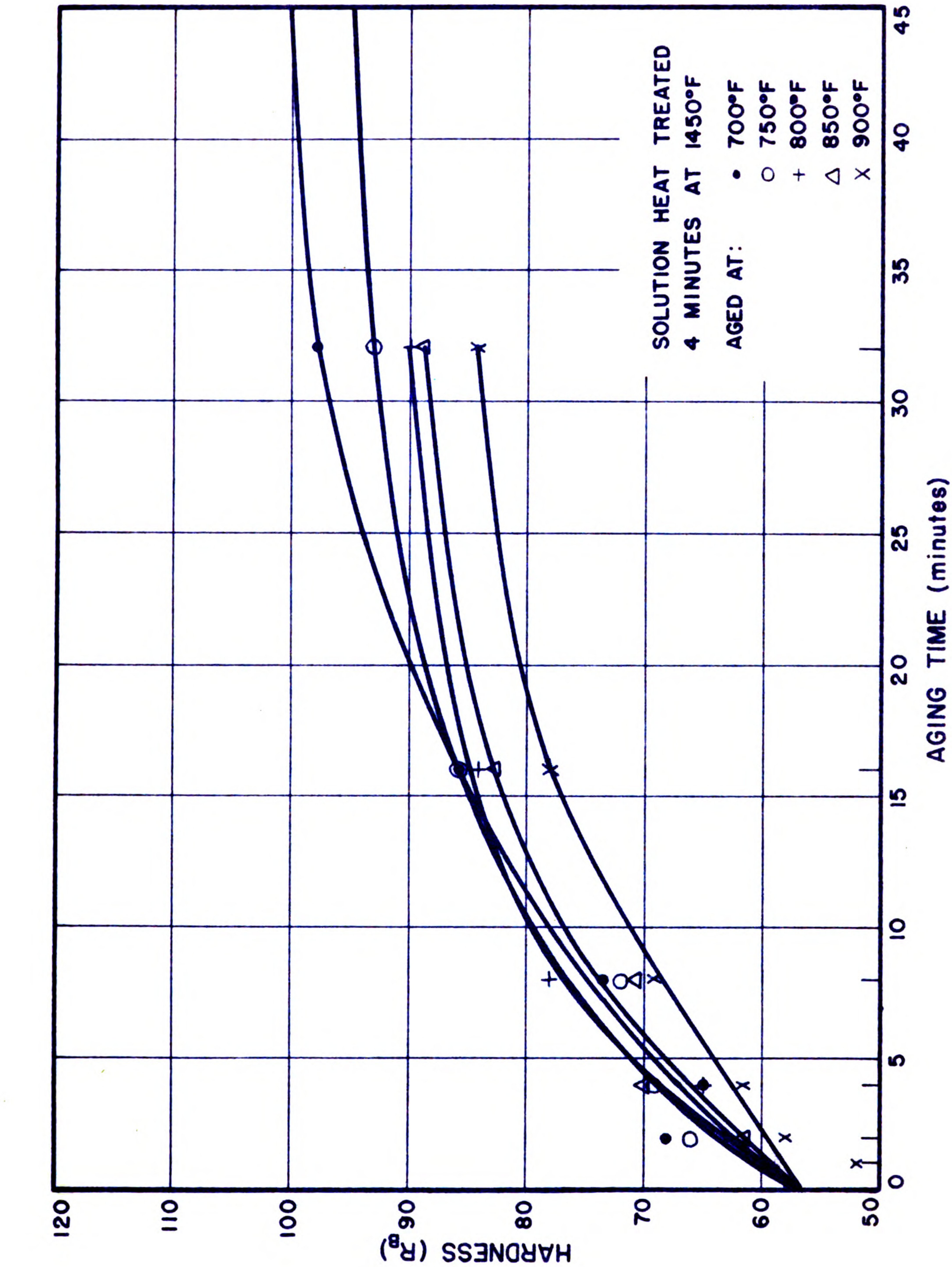

HARDNESS (R$_B$)
120
110
100
90
80
70
60
50
AGING TIME (minutes)
0
5
10
15
20
25
30
35
40
45
SOLUTION HEAT TREATED
2 MINUTES AT 1450°F
AGED AT:
● 700°F
○ 750°F
+ 800°F
△ 850°F
X 900°F

Rockwell$_B$ hardness values of a 2% copper-beryllium binary alloy solution heat treated for 4 minutes at 1450°F and aged as indicated.

All values are the average of eight readings, four being taken on each of two samples.

Temp. of Aging (°F)	Time of Aging (minutes)							
	0	1	2	4	8	16	32	64
700	57		68	65	73	86	96	31*
750	57		66	69	72	86	93	98
800	57	60	63	65	78	84	90	
850	57	60	62	70	71	83	89	
900	57	52	58	62	69	78	84	

* Rockwell$_C$


SOLUTION HEAT TREATED
4 MINUTES AT 1450°F
AGED AT:
● 700°F
○ 750°F
+ 800°F
△ 850°F
× 900°F
HARDNESS (R_B)
120
110
100
90
80
70
60
50
AGING TIME (minutes)
0
5
10
15
20
25
30
35
40
45

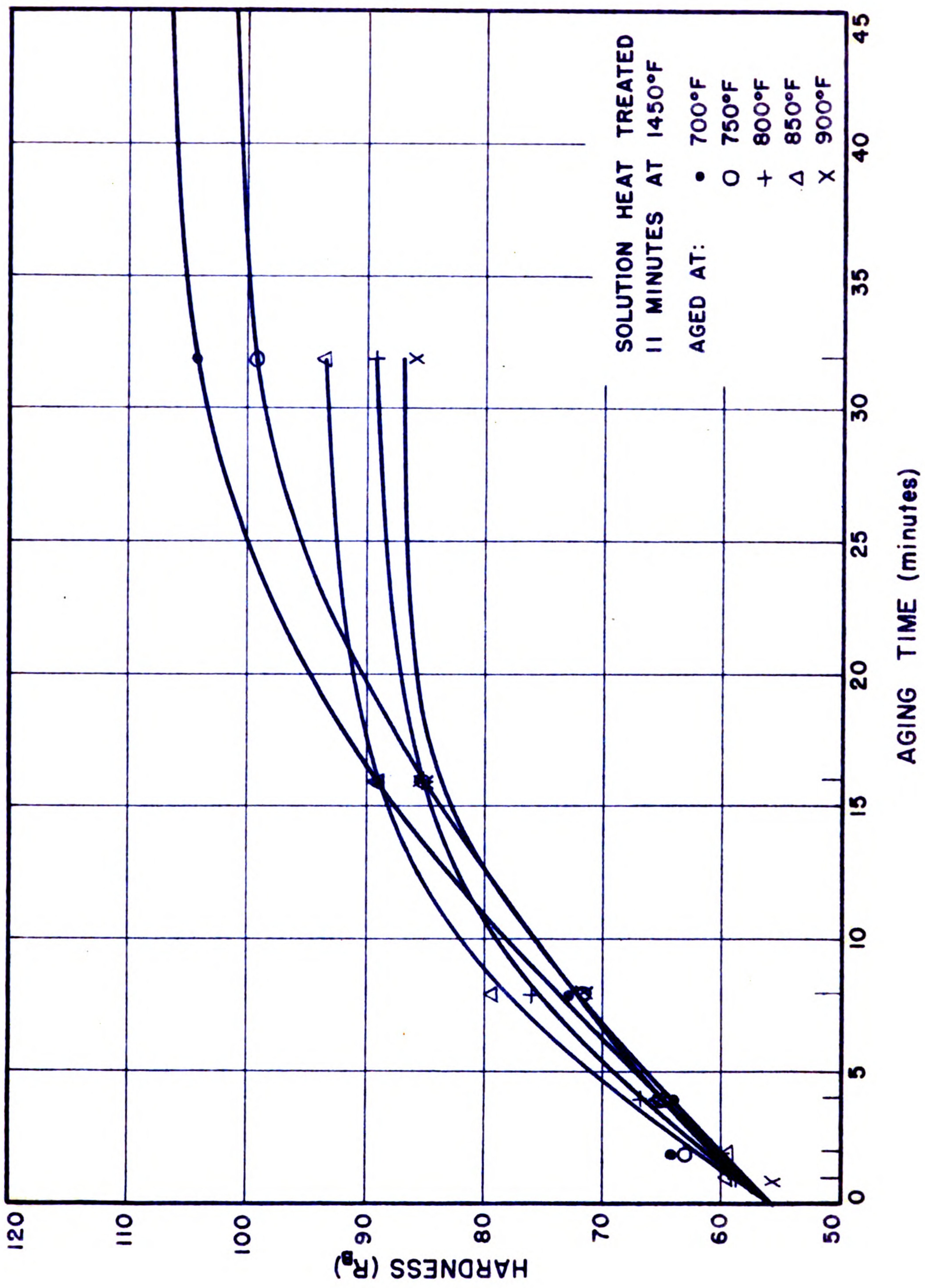
Rockwell$_B$ hardness values of a 2% copper-beryllium binary alloy solution heat treated for 11 minutes at 1450°F and aged as indicated.

All values are the averages of eight readings, four being taken on each of two samples.

Time of Aging (minutes)

Temp. of Aging (°F)	0	1	2	4	8	16	32	64
700	56		64	64	73	89	30*	35*
750	56		63	65	72	85	99	28*
800	56	58	60	67	76	85	89	
850	56	59	59	65	79	89	93	
900	56	56	61	65	72	85	86	

* Rockwell$_C$


SOLUTION HEAT TREATED
11 MINUTES AT 1450°F
AGED AT:
• 700°F
O 750°F
+ 800°F
Δ 850°F
X 900°F
HARDNESS (R_B)
120
110
100
90
80
70
60
50
AGING TIME (minutes)
0
5
10
15
20
25
30
35
40
45

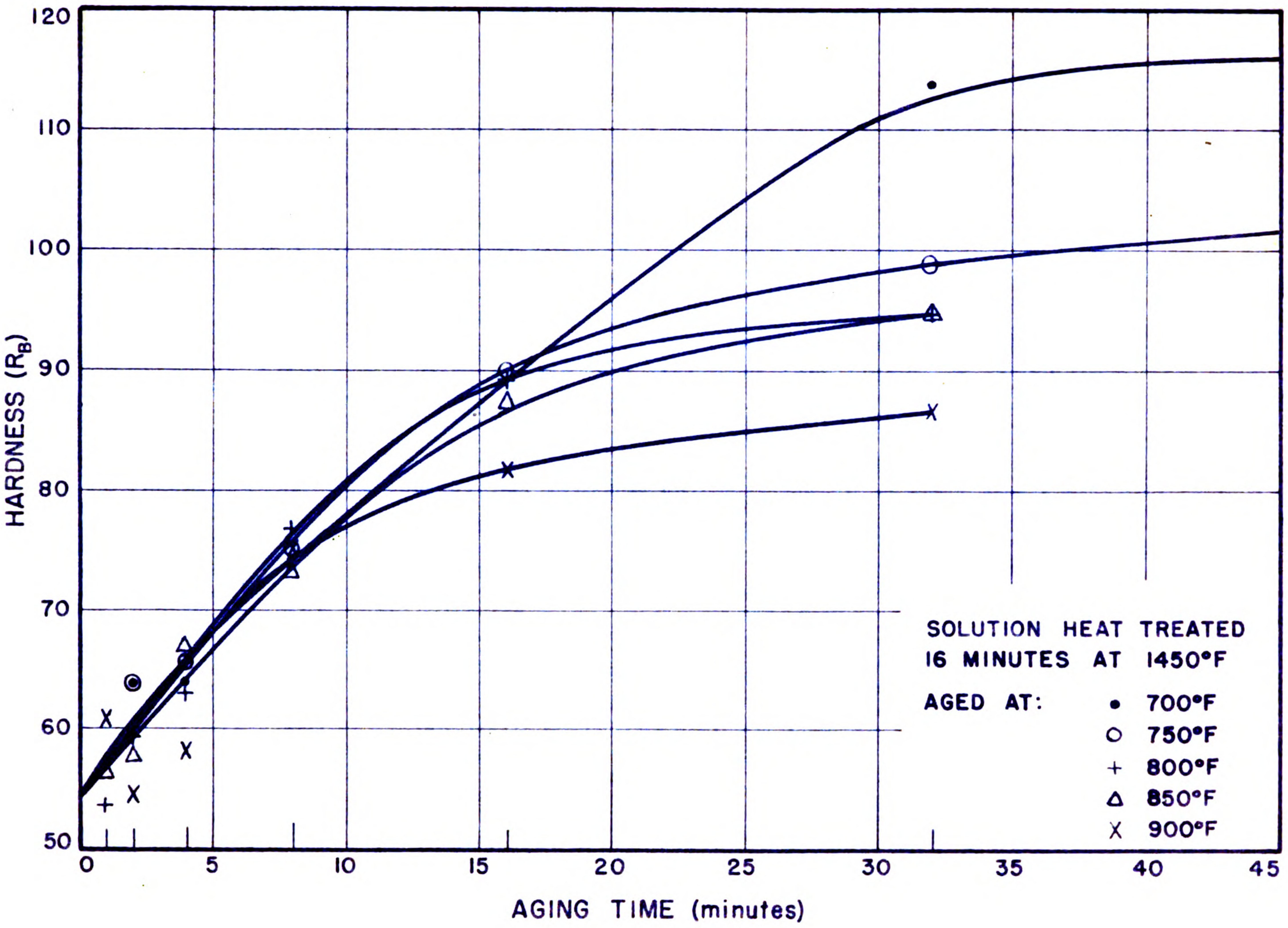
Rockwell$_B$ hardness values of a 2% copper-beryllium binary alloy solution heat treated for 16 minutes at 1450°F and aged as indicated.

All values are the averages of eight readings, four being taken on each of two samples.

Time of Aging (Minutes)

Temp. of Aging (°F)	0	1	2	4	8	16	32	64
700	54		63	63	74	89	37*	38*
750	54		63	65	75	90	98	30*
800	54	54	59	63	77	89	95	
850	54	57	58	67	73	88	95	
900	54	61	55	58	75	82	87	

* Rockwell$_C$


120
110
100
90
80
70
60
50
HARDNESS (R_B)
0
5
10
15
20
25
30
35
40
45
AGING TIME (minutes)
SOLUTION HEAT TREATED
16 MINUTES AT 1450°F
AGED AT:
● 700°F
○ 750°F
+ 800°F
△ 850°F
X 900°F

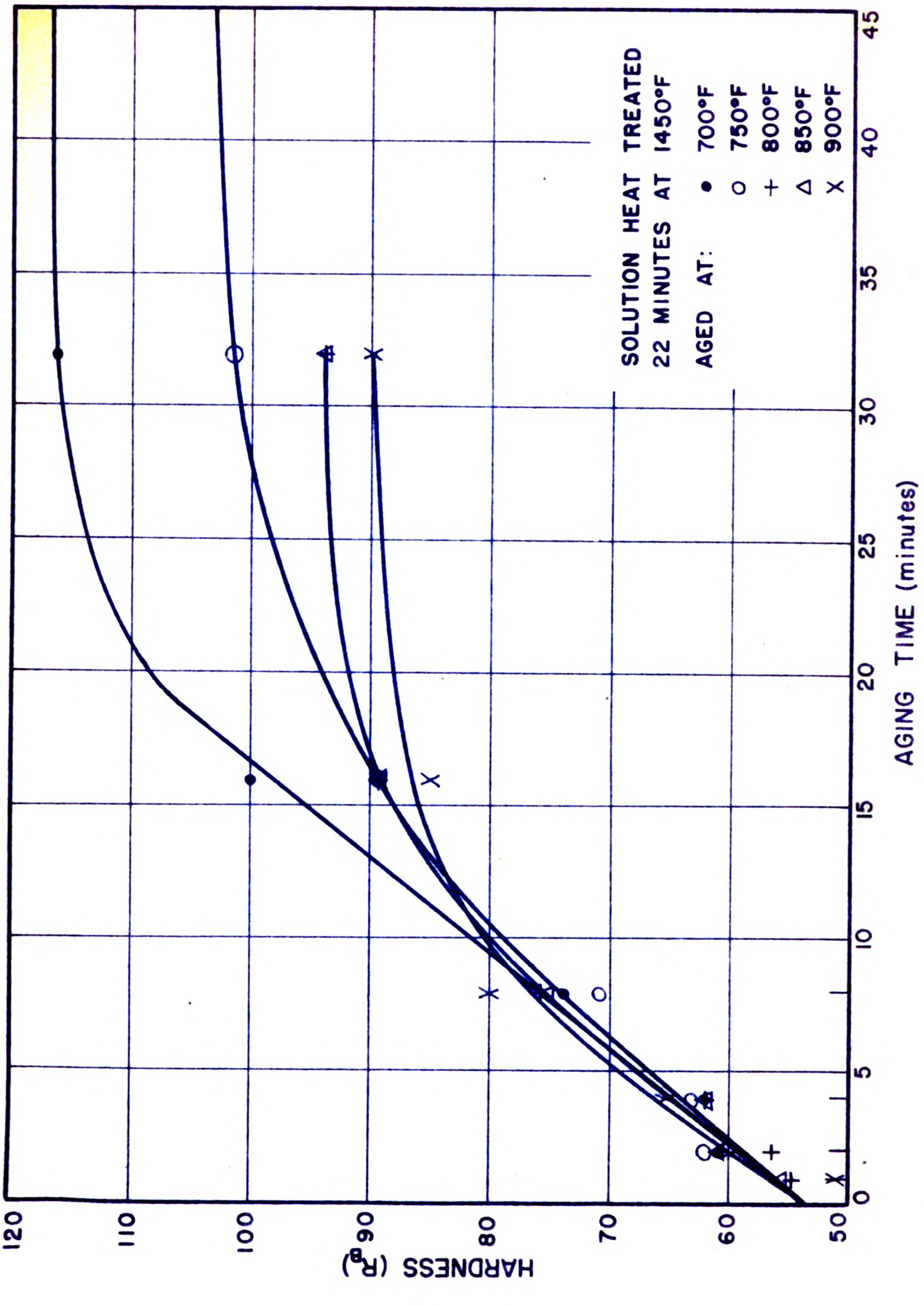
Rockwell$_B$ hardness values of a 2% copper-beryllium binary alloy solution heat treated for 22 minutes at 1450°F and aged as indicated.

All values are the averages of eight readings, four being taken on each of two samples.

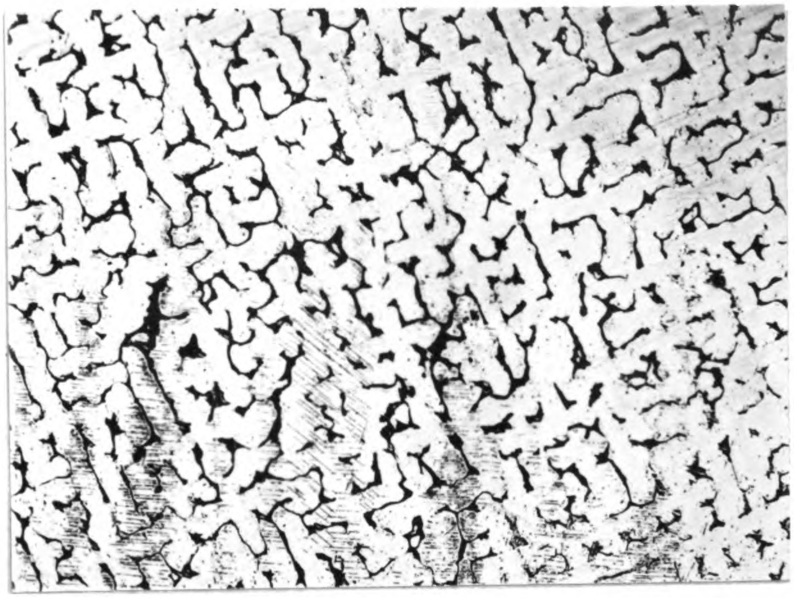
	Time of Aging (Minutes)							
Temp. of Aging (°F)	0	1	2	4	8	16	32	64
700	53		61	62	74	23*	40*	39*
750	53		62	63	71	89	25*	30*
800	53	55	57	62	76	89	94	
850	53	56	61	62	75	89	94	
900	53	51	60	65	80	85	90	

* Rockwell$_C$

The hardness of the 2% alloy in the "as cast" condition was 60 R$_B$.

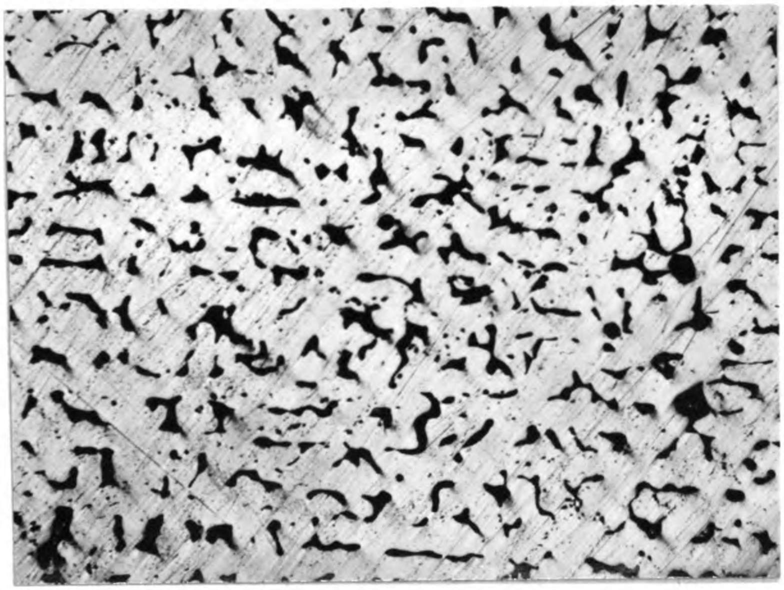

SOLUTION HEAT TREATED
22 MINUTES AT 1450°F
AGED AT:
● 700°F
○ 750°F
+ 800°F
△ 850°F
X 900°F
HARDNESS (R_B)
120
110
100
90
80
70
60
50
AGING TIME (minutes)
0
5
10
15
20
25
30
35
40
45

PHOTOMICROGRAPHS

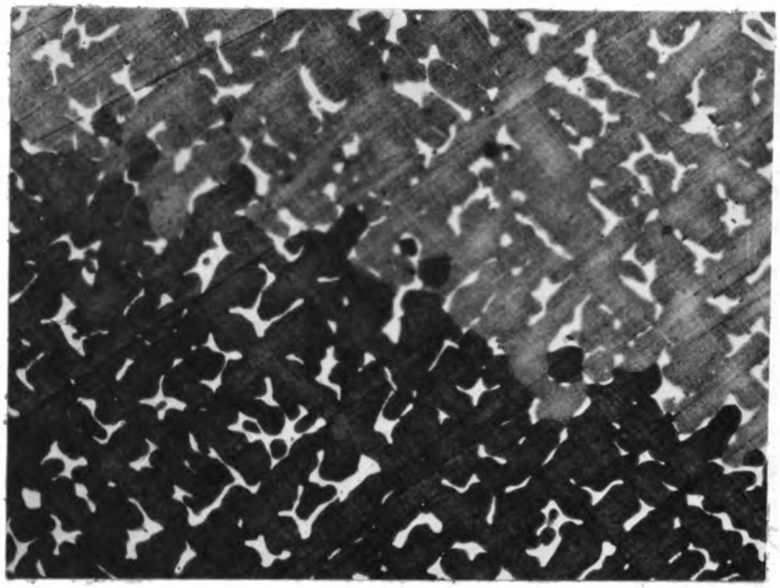


"as cast"
100X

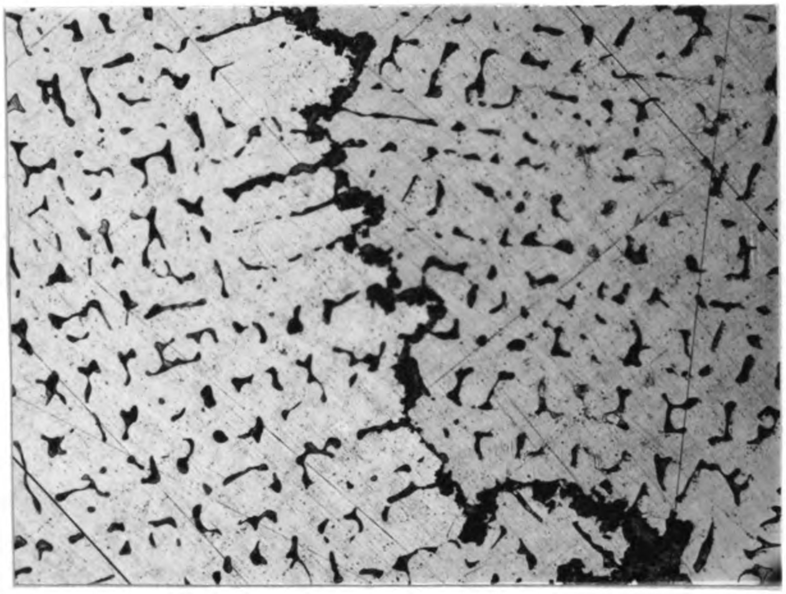
Solution heat treated at 1450°F for 4 minutes
100X



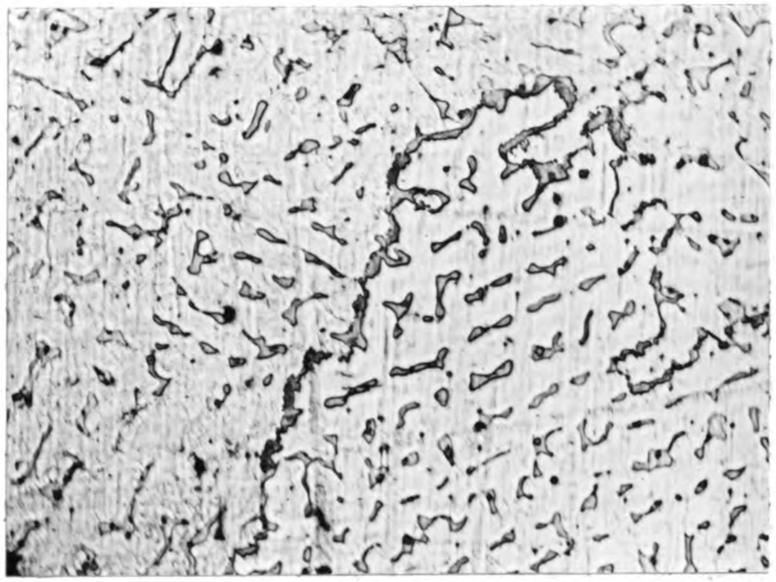
Solution heat treated at 1450°F for 16 minutes
100X



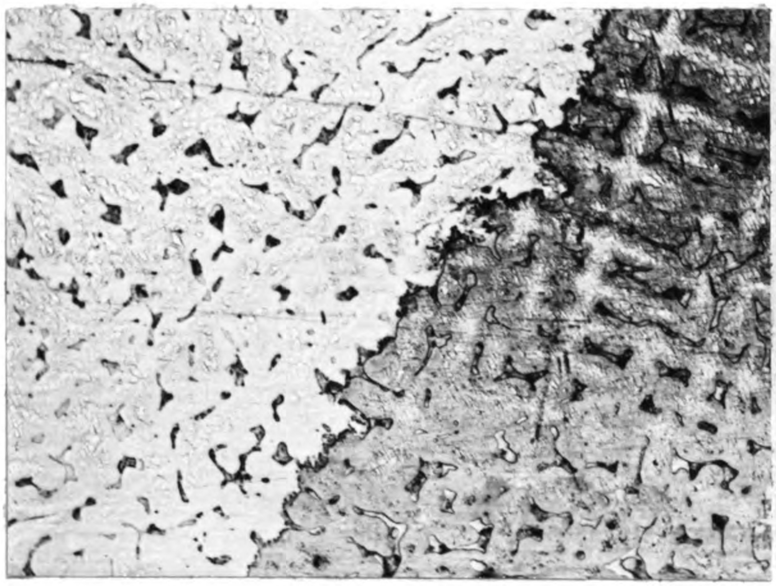
Solution heat treated at 1450°F for 16 minutes
and aged at 700°F for 4 minutes
100X



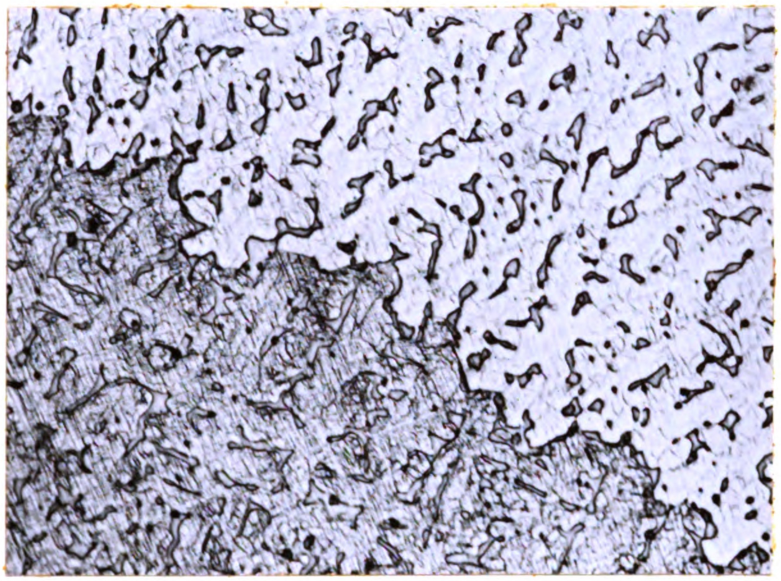
Solution heat treated at 1450°F for 16 minutes
and aged at 700°F for 64 minutes
100X



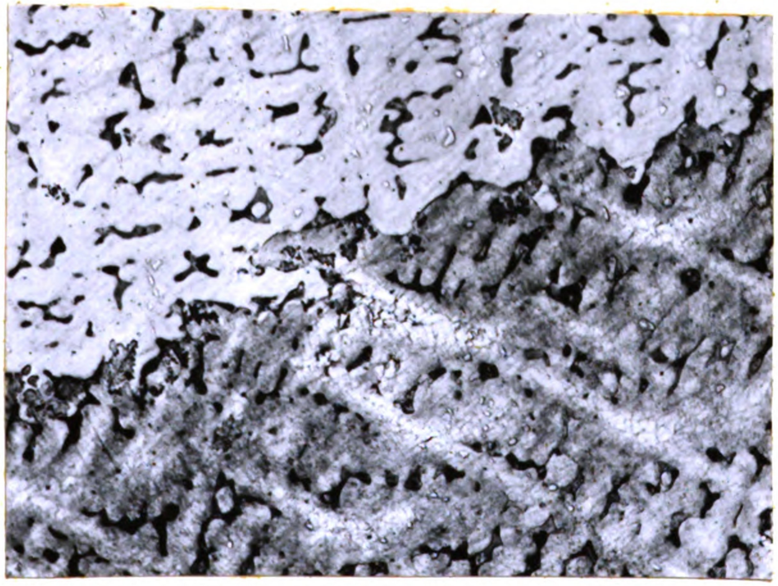
Solution heat treated at 1450°F for 16 minutes
and aged at 800°F for 4 minutes
100X



Solution heat treated at 1450°F for 16 minutes
and aged at 800°F for 32 minutes
100X



Solution heat treated at 1450°F for 16 minutes
and aged at 900°F for 4 minutes
100X



Solution heat treated at 1450°F for 16 minutes
and aged at 900°F for 32 minutes
100X

SUMMARY

1. The results of this investigation clearly show that there is a rapid decrease in the rate of precipitation hardening after about 15 minutes of heating except for those heated at 700°F where the decrease came at about 25 minutes. However, there seems to be little advantage in using temperatures toward the maximum of the alpha-gamma range as the increased rate of hardening hardly seems sufficient to compensate for the lower final hardness unless high hardnesses are not desired.

2. Even though the cast samples were cooled in the mold, the rate of cooling was apparently too rapid to maintain equilibrium conditions. This is brought out by the slight increase in hardness in the short-time solution heat treatment.

3. There seems to be a drop in hardness in hardening for short times at 900°F. Whether or not this is significant is not obvious.

4. It seems that the data would be much more conclusive if the procedure was such as to insure uniform and homogenous samples. In cases it was necessary to take as many as eight hardness readings in order to find four that were representative of the correct

hardness of the sample. It is believed that an increase in casting temperature might be of some help.

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