

# Experiments on the Brittleness of the Cupro-Nickel Alloy for Coinage.

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## INTRODUCTORY.

In 1892, the coinage of cupro-nickel, which is an alloy of 75% copper and 25% nickel, was first adopted in this country, and since then it has been manufactured to a large amount in the Imperial Mint.

It was long ago that we first encountered a great difficulty in the manufacture on account of the occasional brittleness of the coins produced.

Investigations were made whenever the brittleness made its appearance.

The results of the earlier researches, however, were not such as to enable us to avoid the trouble altogether; they only served to give a partial remedy. Hence, the same difficulty arose from time to time.

In Autumn, 1919, there were discovered some exceedingly brittle nickel coins which had been crushed into pieces during transportation in a sealed wooden box from the Mint to the Korean banks; they were as brittle as glass or porcelain and could be easily broken up with one's fingers.

It was necessary that the repetition of such an occurrence should be prevented at the earliest possible moment.

The present paper is the outcome of the investigations into the cause of the trouble with suggestions for the remedy of the brittleness, undertaken by the author with the collaboration of Mr. S. Yamada, engineer, and Mr. T. Aboshi, metallurgist, of the Mint.

PART I. THE PROPER TEMPERATURE FOR ANNEALING  
COINAGE CUPRO-NICKEL.

1) Annealing and bending tests.

The difficulties of reducing a brittle-disposed-bar into a good one, fit for further rolling and coining processes, have long been known in practical operations. Several observers in the Mint agreed in declaring that the blanks cut out from a brittle-disposed-bar would be always brittle, and also that the higher the temperature as well as the longer the time of annealing the better blanks would be obtained.

For these reasons, when we encountered the brittle coins frequently in the autumn of 1919, the temperature of annealing was, first, raised and the time of annealing prolonged, but all in vain. The effect was found to be worse.

We had, therefore, to find out what was the proper temperature for annealing the cupro-nickel blanks.

Several annealing tests were carried out at different temperatures from  $550^{\circ}C.$  to  $900^{\circ}C.$ , at different intervals of time from 15 to 90 min., with enclosed or direct fired blanks, and rapid or slow cooling, their hardness was examined with Shore's scleroscope. All the blanks were bent up  $90^{\circ}$  or sometimes  $180^{\circ}$  to test toughness and divided into 4 classes according to the degree of brittleness.

The results were as follows:—

Annealing Test (1).

Ingot bar.	Annealing.		No. of blanks bent $90^{\circ}$ .				Hardness by Shore's Scl.		
	Heating.	Cooling.	Tough.	Torn.	Broken.	Crushed.	High.	Low.	Average.
good	enclosed.	in water.	30	0	0	0	21	17	18.9
" @	"	"	18	12	0	0			
"	"	in atmos.	30	0	0	0	23	21	22.3
" @	"	"	24	6	0	0			
"	direct.	in water.	30	0	0	0	23	21	21.7
" @	"	"	29	1	0	0			
"	"	in atmos.	30	0	0	0	23	21	22.2
" @	"	"	18	12	0	0			

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Ingot bar.	Annealing.		No. of blanks bent 90°				Hardness by Shore's Scl.		
	Heating.	Cooling.	Tough.	Torn.	Broken.	Crushed.	High.	Low.	Average.
bad	enclosed.	in water.	30	0	0	0	21	18	19.4
" @	"	"	29	1	0	0			
"	"	in atmos.	30	0	0	0	20	19	19.4
" @	"	"	14	16	0	0			
"	direct.	in water.	30	0	0	0	21	19	20.2
" @	"	"	30	0	0	0			
"	"	in atmos.	30	0	0	0	23	20	21.3
"	"	"	30	0	0	0			

N. B. Blanks marked @ were bent 180°.

"Tough" means a blank exhibiting no crack when bent.

"Torn" means a blank cracked slightly on the back of the blank.

"Broken" means a blank separated into 2 pieces due to a deep crack.

"Crushed" means a blank which flew into pieces at a single blow of a hammer.

"Hardness" means a number obtained by the use of the soft hammer of Shore's Scleroscope.

The temperature of cooling water was 100°C.

The annealing temperature was 750°C.

Annealing Test (2).

Test No.	Annealing temp. C.	Heating.		No. of blanks bent 90°					Remarks.
		Hour.	Min.	Tough.	Torn.	Broken.	Crushed.	Total.	
1	350°	1	25	0	22	3	0	25	direct fired.
2	400°	1	25	0	23	2	0	25	
3	450°	1	30	2	23	0	0	25	
4	500°	1	25	7	18	0	0	25	
5	550°	1	25	18	7	0	0	25	
@	"	1	0	10	39	1	0	50	best. weakly torn.
6	600°	1	25	25	0	0	0	25	
@	"	1	0	43	7	0	0	50	
7	650°	0	15	23	2	0	0	25	weakly torn.
@	"	1	0	46	4	0	0	50	

Test No.	Annealing temp. C.	Heating.		No. of blanks bent 90°.					Remarks.
		Hour.	Min.	Tough.	Torn.	Broken.	Crushed.	Total.	
8	650°	0	30	25	0	0	0	25	best.
9	"	1	10	25	0	0	0	25	best.
10	700°	1	0	22	3	0	0	25	
@	"	1	0	49	1	0	0	50	slightly torn.
11	750°	1	0	23	2	0	0	25	
@	"	1	0	41	9	0	0	50	weakly torn.
12	800°	1	0	22	3	0	0	25	
@	"	1	0	22	27	1	0	50	strongly torn.
13	850°	0	40	20	2	3	0	25	
14	900°	0	40	10	5	10	0	25	

N. B. Blanks marked @ are comparatively good blanks in Test (1).

All the blanks were rapidly cooled in water at 100°C.

### Annealing Test (3).

Test No.	Annealing temp. C.	Heating.		No. of blanks bent 90°.					Remarks.
		Hour.	Min.	Tough.	Torn.	Broken.	Crushed.	Total.	
1	600°	0	15	27	23	0	0	50	
2	"	0	30	35	15	0	0	50	
3	"	0	45	43	7	0	0	50	
4	"	1	0	43	7	0	0	50	
5	"	1	15	41	9	0	0	50	
6	650°	0	15	48	2	0	0	50	
7	"	0	30	49	1	0	0	50	best
8	"	0	45	45	5	0	0	50	
9	"	1	0	46	4	0	0	50	
10	"	1	15	48	2	0	0	50	
11	700°	0	15	48	2	0	0	50	
12	"	0	30	49	1	0	0	50	best
13	"	0	45	48	2	0	0	50	best
14	"	1	0	49	1	0	0	50	
15	"	1	15	46	4	0	0	50	
16	750°	0	15	45	5	0	0	50	
17	"	0	30	41	9	0	0	50	
18	"	0	45	37	13	0	0	50	
19	"	1	0	41	0	0	0	50	
20	"	1	15	38	12	0	0	50	



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Annealing Test (4).

Test No.	Annealing temp. C.	Heating.		No. of blanks bent 90°.					Remarks.
		Hour.	Min.	Tough.	Torn.	Broken.	Crushed.	Total	
1	650°	0	30	50	0	0	0	50	best
2	"	0	45	50	0	0	0	50	"
3	"	1	0	50	0	0	0	50	"
4	"	1	15	50	0	0	0	50	"
5	"	1	30	50	0	0	0	50	"
6	700°	0	30	50	0	0	0	50	"
7	"	0	45	49	1	0	0	50	"
8	"	1	0	49	1	0	0	50	"
9	"	1	15	43	7	0	0	50	"
10	"	1	30	39	11	0	0	50	"
11	750°	0	30	43	7	0	0	50	"
12	"	0	45	36	14	0	0	50	"
13	"	1	0	30	20	0	0	50	"
14	"	1	15	32	18	0	0	50	"
15	"	1	30	20	29	1	0	50	"

Annealing Test (5).

Test No.	Annealing temp. C.	Heating.		No. of blanks bent 90°.					Remarks.
		Hour.	Min.	Tough.	Torn.	Broken.	Crushed.	Total.	
1	900°	1	10	0	0	0	10	10	
2	850°	1	10	0	0	0	10	10	
3	840°	1	10	0	3	0	7	10	
4	840°	1	10	0	9	0	1	10	
5	830°	1	10	0	3	0	7	10	
6	820°	1	10	0	9	0	1	10	
7	820°	1	10	0	2	0	8	10	
8	810°	1	10	0	5	0	5	10	
9	800°	1	10	0	5	0	5	10	
10	800°	1	10	0	10	0	0	10	best
11	790°	1	10	0	9	0	1	10	
12	790°	1	10	0	10	0	0	10	best
13	790°	1	10	0	10	0	0	10	"
14	780°	1	10	0	10	0	0	10	"
15	770°	1	10	0	10	0	0	10	"

*N. B.* All the blanks used in the Test were torn weakly.

Temperature of the cooling water was 100° C.

## Annealing Test (6).

Test No.	Annealing temp. C.	Heating.		No. of blanks bent 90°.					Remarks.
		Hour.	Min.	Tough.	Torn.	Broken.	Crushed.	Total.	
1	820°	1	10	0	4	6	0	10	
2	810°	1	10	0	6	4	0	10	
3	800°	1	10	0	7	3	0	10	
4	790°	1	10	0	6	4	0	10	
5	780°	1	10	0	10	0	0	10	best
6	770°	1	10	0	10	0	0	10	"
7	760°	1	10	0	10	0	0	10	"
8	750°	1	10	0	10	0	0	10	"

N. B. All the blanks had a reticular or retiform appearance.  
After heating cooled in the atmosphere.

In the above two Tests we see that the blanks with more or less of a net-work appearance cut out from the so called "squamous bar" indicate the possibility of being made into perfect coins, since we can easily bend them 90° by tapping with a hammer when they are annealed at a temperature below 780°C. or 790°C., otherwise they are mostly "broken."

In practice, therefore, it is very much to be desired that they should be annealed at a far lower temperature than 780°C., because it is difficult to heat up all the blanks perfectly uniform, so as to leave absolutely no brittle blank.

## Annealing Test (7).

All the blanks had a squamous appearance.

Test No.	Annealing temp. C.	Heating.		No. of blanks bent 90°.					Remarks.
		Hour.	Min.	Tough.	Torn.	Broken.	Crushed.	Total.	
1	700°	1	30	0	10	0	0	10	
2	850°	1	10	0	0	0	10	10	
3	900°	1	10	0	0	0	10	10	
4	770°	1	10	3	7	0	0	10	
5	775°	1	10	2	8	0	0	10	
6	780°	1	10	3	7	0	0	10	
7	785°	1	10	0	10	0	0	10	

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Test No.	Annealing temp. C.	Heating.		No. of blanks bent 90°.					Remarks.
		Hour.	Min.	Tough.	Torn.	Broken	Crushed.	Total.	
8	790°	1	10	2	8	0	0	10	
9	795°	1	10	2	7	0	1	10	
10	800°	1	10	1	7	1	1	10	
11	805°	1	10	0	7	1	2	10	
12	810°	1	10	0	5	2	3	10	
13	815°	1	10	0	6	2	2	10	
14	820°	1	10	0	4	2	4	10	
15	825°	1	10	0	3	5	2	10	
16	830°	1	10	0	0	5	5	10	
17	835°	1	10	0	1	6	3	10	
18	840°	1	10	0	0	3	7	10	
19	845°	1	10	0	0	1	9	10	
20	850°	1	10	0	0	1	9	10	
21	860°	1	10	0	0	2	8	10	
22	870°	1	10	0	0	0	10	10	
23	880°	1	10	0	0	0	10	10	
24	890°	1	10	0	0	0	10	10	
25	900°	1	10	0	0	0	10	10	

Annealing Test (8).

All the blanks had a squamous appearance as in Fig. No. 53.

Cooling in hot water.

Test No.	Annealing temp. C.	Heating.		No. of blanks bent 90°					Remarks.
		Hour.	Min.	Tough.	Torn.	Broken.	Crushed.	Total.	
1	650°	1	10	0	10	0	0	10	Fig. No. 54
2	700°	1	10	0	10	0	0	10	
3	770°	1	10	0	10	0	0	10	„ No. 55
4	780°	1	10	0	10	0	0	10	
5	790°	1	10	0	9	1	0	10	„ No. 56
6	800°	1	10	0	5	5	0	10	
7	810°	1	10	0	5	5	0	10	
8	820°	1	10	0	2	8	0	10	
9	830°	1	10	0	3	7	0	10	
10	840°	1	10	0	3	7	0	10	„ No. 57
11	850°	1	10	0	0	10	0	10	„ No. 58
12	900°	1	10	0	0	10	0	10	

## Annealing Test (9).

Common blanks in routine work.

Test No.	Annealing temp. C.	Heating.		No. of blanks bent 90°.					Remarks.
		Hour.	Min.	Tough.	Torn.	Broken.	Crushed.	Total.	
1	650°	0	30	2	437	11	0	450	best
2	"	0	45	411	39	0	0	450	
3	"	1	0	445	5	0	0	450	
4	"	1	15	420	5	0	0	425	
5	"	1	30	434	16	0	0	450	

The above Test (9) was conducted under conditions nearest the practical working i. e., blanks were packed in an iron box containing 18 piles, 25 blanks each, and filled up with charcoal powder. The box was sealed with clay on an iron cover and heated in an electric furnace to the temperature of 650°C. for 30 min., 45 min., 60 min., 75 min., and 90 min. After annealing each blank was tested by bending 90° with the results shown in Fig. 1 to Fig. 5.

From the annealing Test (9) and the Hardness Tests mentioned below, we see that it is best to anneal the Cupro-nickel in ordinary working conditions at a temperature of 650°C. for 1 hour or 1 hour & a quarter.

We, next, examined the relations between the hardness of blanks and the annealing temperature as well as the duration of heating.

In these tests 10 blanks were piled up with a rod passing through the central holes to prevent them from falling down, and imbedded in charcoal powder in an iron pipe in the upright position with one end closed. The pipe was heated in an electric furnace of the Hoskin's crucible type. After annealing, the hardness of each blank was tested by Shore's scleroscope, the average results are shown in the following tables.

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2) Hardness Tests from 550°C. to 900°C.

Hardness Test (1).

At 550°C.

Test No.	Annealing.		Average hardness (Shore).			No. of blanks.	Test bent 90°.	Remarks.
	Hour.	Min.	Before.	After.	Difference.			
1	0	15	62.1	53.4	8.7	10	good	
2	0	20	63.2	42.0	21.2	10	"	
3	0	20	62.9	50.1	12.8	10	1-torn	
4	0	25	60.2	43.9	16.3	10	good	
5	0	25	61.2	45.2	16.0	10	"	
6	0	30	63.0	42.7	20.3	10	"	
7	0	30	61.4	46.5	14.9	10	"	
8	0	30	60.8	37.5	23.5	10	"	
9	0	35	61.3	34.7	26.6	10	"	
10	0	35	61.5	39.3	22.2	10	"	
11	0	40	61.7	33.9	27.8	10	"	best
12	0	45	61.5	34.3	27.2	10	"	"
13	0	50	61.6	33.8	27.8	10	"	"
14	0	55	61.2	31.8	29.4	10	"	"
15	0	55	61.5	38.0	23.5	10	"	
16	0	55	61.8	40.3	21.5	10	"	
17	1	0	61.0	37.7	23.3	10	"	
18	1	5	61.5	37.3	24.2	10	"	
19	1	10	61.7	40.1	21.6	10	"	
20	1	15	64.3	38.9	25.4	10	"	
21	1	20	62.7	37.8	24.9	10	"	
32	1	25	61.6	38.3	23.3	10	"	
23	1	30	61.6	38.3	23.3	10	"	

Hardness Test (2).

At 600°C.

Test No.	Annealing.		Average hardness (Shore).			No. of blanks.	Test bent 90°.	Remarks.
	Hour.	Min.	Before.	After.	Difference.			
1	0	15	62.2	35.7	26.5	10	good	
2	0	20	63.8	36.1	27.7	10	"	
3	0	25	62.4	35.7	26.7	10	"	
4	0	30	60.7	33.2	27.5	10	"	

Test No.	Annealing.		Average hardness (Shore).			No. of blanks.	Test bent 90°.	Remarks.
	Hour.	Min.	Before.	After.	Difference.			
5	0	35	63.0	33.0	30.0	10	good	exception
6	0	40	61.0	32.1	27.9	10	"	
7	0	45	62.3	33.0	29.3	10	"	
8	0	50	62.8	34.9	27.9	10	"	
9	0	55	62.9	34.0	28.9	10	"	
10	1	0	65.4	33.8	31.6	10	"	
11	1	5	61.8	34.4	27.4	10	"	
12	1	10	60.4	33.4	27.0	10	"	
13	1	15	60.9	33.0	27.9	10	"	
14	1	20	61.9	33.5	28.4	10	"	
15	1	25	62.7	34.9	27.8	10	"	
16	1	30	60.4	31.1	29.3	10	"	

## Hardness Test (3).

At 650°C.

Test No.	Annealing.		Average hardness (Shore).			No. of blanks.	Test bent 90°.	Remarks.
	Hour.	Min.	Before.	After.	Difference.			
1	0	15	59.6	37.0	22.6	10	good	
2	0	20	62.1	35.1	27.0	10	"	
3	0	25	64.6	36.7	27.9	10	"	
4	0	30	63.0	35.2	27.8	10	"	
5	0	35	64.1	35.2	28.9	10	"	
6	0	40	61.7	34.6	27.1	10	"	
7	0	45	62.9	35.7	27.2	10	"	
8	0	50	63.6	35.4	28.2	10	"	
9	0	55	64.3	35.2	29.1	10	"	
10	1	0	65.5	33.7	31.8	10	"	
11	1	5	61.8	34.4	27.4	10	"	
12	1	10	64.4	34.9	29.5	10	"	
13	1	15	62.8	33.9	28.9	10	"	
14	1	20	60.0	34.9	25.1	10	"	
15	1	25	66.3	34.4	31.9	10	"	
16	1	30	65.2	35.2	30.0	10	"	

Hardness Test (4).

At 700°C.

Test No.	Annealing.		Average hardness (Shore).			No. of blanks.	Test bent 90°.	Remarks.
	Hour.	Min.	Before.	After.	Difference.			
1	0	15	64.0	34.2	29.8	10	good	
2	0	20	65.0	34.3	30.7	10	"	
3	0	25	64.1	33.2	30.9	10	"	
4	0	30	62.3	31.9	30.4	10	"	
5	0	35	65.7	31.8	33.9	10	"	
6	0	40	65.3	32.2	33.1	10	"	
7	0	45	65.2	32.8	32.4	10	"	
8	0	50	65.3	31.3	34.0	10	"	
9	0	55	63.5	32.3	31.2	10	"	
10	1	0	63.5	31.1	32.4	10	"	
11	1	5	63.9	30.6	33.3	10	"	
12	1	10	64.3	32.1	32.2	10	"	
13	1	15	63.8	30.1	33.7	10	"	
14	1	20	64.5	30.2	34.3	10	"	
15	1	25	61.7	29.7	32.0	10	"	
16	1	30	62.3	29.5	32.8	10	"	

Hardness Test (5).

At 750°C.

Test No.	Annealing.		Average hardness (Shore).			No. of blanks.	Test bent 90°.	Remarks.
	Hour.	Min.	Before.	After.	Difference.			
1	0	15	66.2	32.9	33.3	10	good	
2	0	20	62.9	31.5	31.4	10	"	
3	0	25	64.5	28.6	35.9	10	"	
4	0	30	64.3	29.5	34.8	10	"	
5	0	35	63.1	27.6	35.5	10	"	
3	0	40	64.4	30.5	33.9	10	"	
7	0	45	64.0	29.9	34.1	10	"	
8	0	50	63.3	29.6	33.7	10	"	
9	0	55	64.1	28.7	35.4	10	1-torn	
10	1	0	61.9	27.8	34.1	10	good	
11	1	5	61.3	27.5	33.8	10	"	
12	1	10	64.0	28.6	35.4	10	"	
13	1	15	64.5	28.3	36.2	10	"	
14	1	20	63.0	28.4	34.6	10	"	
15	1	25	63.5	26.9	36.6	10	"	
16	1	30	63.4	27.6	35.8	10	"	

## Hardness Test (6).

At 800°C.

Test No.	Annealing.		Average hardness (Shore).			No. of blanks.	Test bent 90°.	Remarks.
	Hour.	Min.	Before.	After.	Difference.			
1	0	15	64.4	28.6	35.8	10	good	exception
2	0	20	62.3	28.7	33.6	10	"	
3	0	25	59.7	27.2	32.5	10	"	
4	0	30	67.6	27.3	40.3	10	"	
5	0	35	64.5	27.1	37.4	10	"	
6	0	40	61.6	26.5	35.1	10	"	
7	0	45	63.6	26.7	36.9	10	"	
8	0	50	60.0	27.7	32.3	10	"	
9	0	55	60.3	25.7	34.6	10	"	
10	1	0	64.6	28.1	36.5	10	"	
11	1	5	61.8	27.3	34.5	10	"	
12	1	10	62.0	28.7	33.3	10	"	
13	1	15	62.4	28.3	34.1	10	"	
14	1	20	62.8	28.0	34.8	10	1-broken	
15	1	25	62.1	28.0	34.1	10	"	
16	1	30	59.9	28.1	31.8	10	good	exception

## Hardness Test (7).

At 850°C.

Test No.	Annealing.		Average hardness (Shore).			No. of blanks.	Test bent 90°.	Remarks.
	Hour.	Min.	Before.	After.	Difference.			
1	0	15	62.3	29.2	33.1	10	good	exception
2	0	20	61.1	28.9	32.2	10	"	
3	0	25	60.5	28.6	31.9	10	"	
4	0	30	67.0	29.5	37.5	10	"	
5	0	35	64.1	28.7	35.4	10	"	
6	0	40	62.6	29.9	32.7	10	"	
7	0	45	64.5	29.4	35.1	10	"	
8	0	50	64.6	28.3	36.3	10	"	
9	0	55	65.0	27.2	37.8	10	"	
10	1	0	64.5	27.7	36.8	10	"	
11	1	5	65.8	29.0	36.8	10	"	
12	1	10	62.0	27.4	34.6	10	"	
13	1	15	62.7	28.1	34.6	10	1-torn	
14	1	20	66.3	28.2	38.1	10	good	exception
15	1	25	64.0	26.9	37.1	10	"	
16	1	30	62.5	26.7	35.8	10	"	



Hardness Test (8).

At 900°C.

Test No.	Annealing.		Average hardness (Shore).			No. of blanks.	Test bent 90°.	Remarks.
	Hour.	Min.	Before.	After.	Difference.			
1	0	15	65.1	27.8	37.3	10	good	
2	0	20	62.9	28.0	34.9	10	"	
3	0	25	65.0	27.7	37.3	10	"	
4	0	30	65.8	27.1	38.7	10	"	
5	0	35	66.0	26.7	39.3	10	"	
6	0	40	63.8	26.6	37.2	10	"	
7	0	45	64.9	26.0	38.9	10	"	
8	0	50	62.6	26.2	36.4	10	"	
9	0	55	68.1	25.5	42.6	10	"	exception
10	1	0	65.2	24.7	40.5	10	"	
11	1	5	66.4	25.5	40.9	10	"	
12	1	10	66.0	25.3	40.7	10	"	
13	1	15	65.5	25.0	40.5	10	"	
14	1	20	63.0	26.0	37.0	10	"	
15	1	25	67.2	23.5	43.7	10	"	exception
16	1	30	66.2	23.9	42.3	10	"	

From the above 8 Tests at temperatures ranging from 550°C. to 900°C. we see that in general the higher the annealing temperatures as well as the longer the time of annealing the softer can blanks be obtained. It is well in accord with the opinions heretofore accepted, but as a matter of fact, we found some brittle blanks among them; i. e., 1 in Test (1) at 550°C., 1 in Test (5) at 750°C., 2 in Test (6) at 800°C. and 1 in Test (7) at 850°C. so that there must be some important exceptions to the Rule.

It is obvious that it is much safer to anneal at a temperature of from 650°C. to 700°C. if there are some chances of internally defective blanks being mixed, that is to say, some scale-like blanks being found, as will be the case in the usual practice; in fact we rather prefer 650°C. as the standard temperature of annealing cupro-nickel alloy in such cases, because some blanks might be more inclined to be overheated and in consequence be brittle at 700°C. than would be the case at 650°C. by a local overheating in the furnace.

To anneal blanks at  $650^{\circ}\text{C}$ . is, of course, a tedious process, but it is very necessary at present to prevent accidental occurrences of brittle coins, which can not be easily found out by inspection.

The difficulties encountered since November, 1919, have been entirely overcome in this way.

#### Conclusions for Part I.

- a) The conclusions we have arrived at are as follows:—
- 1) The higher the temperature of annealing the lower is the hardness of blanks obtained.
  - 2) It is better to cool rapidly the cupro-nickel after annealing than to cool slowly.
  - 3) A long duration of time is required to anneal the alloy thoroughly when the temperature is low.
  - 4) A good bar never turns brittle even when annealed at a very high temperature.
  - 5) We can make a perfect coin that will never be brittle, if it is annealed at about  $650^{\circ}\text{C}$ . for about 1 hour, though it may be brittle-disposed at the start.
  - 6) We can never turn a brittle bar into a malleable one however long we may reanneal it at the highest possible temperature below the melting point. Remelting is the only way to make it malleable.
- b) The remedy of the troubles in the Mint practice would be as follows:—
- 1) To improve the annealing furnaces both for bars and blanks so that they can be heated up more uniformly and regulated more surely; we can then bring up the standard annealing temperature of  $650^{\circ}\text{C}$ . to about  $750^{\circ}\text{C}$ . or higher and reduce the annealing time.
  - 2) To improve the original bars during melting, by adding some deoxidizing agent, so as to prevent the tendency toward a

squamiform appearance. If only we could get rid of all the injurious substances from the bars the annealing temperature can be brought up as high as may be desired and the period of annealing be thereby markedly diminished.

### 3) Present Appliances in the Mint.

The annealing furnaces now in operation in the Mint are shown in Fig. (6), Fig. (7) and Fig. (8); the former is fired with town gas using air blast and the latter two with coal. Fig. (9) shows an annealing box for coinage nickel blanks.

Recently, we have set up a rotary furnace, fired with town gas, for annealing. The blanks are fed into a hopper at one end of the furnace from which they are fed into the spiral retort. The retort is uniformly heated and very slowly rotated; the inside being comparatively air-tight a perfect non-oxidizing annealing is obtained. The result of the trial was excellent and it may be possible to improve the annealing furnaces in the near future in accordance with Remedy 1) above mentioned.

As to Remedy 2), we are going to describe it more particularly in Part III.

## PART II. INJURIOUS SUBSTANCES CAUSING THE BRITTLENESS OF CUPRO-NICKEL.

We have endeavoured to find out what is the injurious matter which produces a defective bar, the so called "Scale-like bar."

For this purpose we have examined both a malleable and a defective bar inside and outside, at top, middle and bottom, before and after annealing and also the blanks cut from a good bar or bad one in a similar way by means of a microscope.

It may perhaps be necessary to give a brief account of the manufacturing process for the nickel coinage here.

### 4). A brief account of the manufacturing process for coinage nickel in the Mint.

#### a) Casting.

The nickel, used chiefly as material, is obtained from The International

Nickel Co. of America; the composition is about 98.4%—99.0% nickel, and 0.52%—0.56% iron. But, sometimes, the Mond nickel from England is used; its composition is 99.3–99.6% nickel and 0.14–0.38% iron. The copper added to the nickel is electrolytic copper of 99.92–99.98% copper content, with no bismuth, nor antimony nor arsenic.

The materials are exactly weighed in fixed proportions, i. e., 25% of nickel and 75% of copper. About 60% of new materials, and 40% of scissels from rolled fillets, and also some imperfect bars, blanks, and coins, are melted together in a plumbago crucible in a coke furnace. An electric furnace by GREAVES ETCHELS of 1000 pounds capacity has lately been built and it is just now under trial, for the purpose of melting cupro-nickel alloy.

The melted alloy in the crucible after thorough stirring up is raised to the working floor and poured into cast iron moulds,  $\frac{5}{8}$ " thick  $3\frac{1}{4}$ – $3\frac{1}{2}$ " wide and 22" long, in upright position.

One or two inches of the head of cast bars are cut off to fit them for rolling. A sample piece for assay is taken from a bar, one for each pot.

b) Bar Annealing and Rolling.

We anneal the bars from the Melting Room, at first, in a reverberatory furnace fired by coal. This is done by packing them, side by side, with the edge of bar downward upon a heavy cast iron truck which is pushed into a hot zone of the furnace, where the bars are directly heated by flame, being oxidized more or less on the surface.

As a rule, when they have been heated up to  $650^{\circ}C$ . for about 1 hour they are thrown into a water tank, where they are rapidly cooled.

Then they are passed through between chilled rolls of cast iron, 14" dia. to break them down. After several passings in the break-down rolls the bars undergo intermediate rollings in 10" dia. chilled rolls, and then final rollings in  $6\frac{1}{2}$ " dia. hardened steel rolls.

The number of passings in each roll and the thickness of the fillets in 1/1000 of an inch after the respective passings are shown in the following table.

		Rolling Mill.						Total no. of passings.
		Break-down.		Middling.			Finish.	
		I	II	III	IV	V	VI	
10-sen fillet.	No. of passings.	7	6	4	3	3	2	25
	Thickness in mil.	245.	120.	88.	65.	47.	43.	
5-sen fillet.	No. of passings.	7	5	3	3	2	2	22
	Thickness in mil.	245.	110.	88.	65.	47.	41.	

c) Cutting, Milling and Centering.

When the above stage of rolling is finished, the fillets are punched at random in the middle part by means of a trier's cutter, the blank thus obtained from each fillet is weighed exactly by a skilful workman, and the fillets are arranged into 6 different groups according to the result of the weighing. If there is a heavier blank than No. 1, the particular fillet is passed, once more, through the finishing rolls. The fillets which give blanks lighter than No. 6, are melted again.

The weights of the trial blanks corresponding to the several groups are as shown in the following list.

Exact weight of blanks, not perforated, for tests in 1/1000 Momme.

	Order of groups of fillets tested.						Standard blank in Momme.
	I	II	III	IV	V	VI	
For 10-sen blank.	+7,+6	+5,+4	+3,+2	+1,0	-1,-2	-3,-4	1.052
For 5-sen blank.	+7,+6	+5,+4	+3,+2	+1,0	-1,-2	-3,-4	0.740

*N. B.* The figures are the weights of respective blanks heavier than or lighter than the standard blank, and "+" means "heavy" & "-" means "light."

1 Momme =  $3\frac{3}{4}$  Grammes.

Approximate thickness of fillets of each group in 1/1000 of an inch.

	Order of groups of fillets tested.					
	I	II	III	IV	V	VI
For 10-Sen blank.	43.8-43.7	43.7-43.6	43.6-43.5	43.5-43.4	43.4-43.3	43.3-43.2
For 5-Sen blank.	41.9-41.8	41.8-41.7	41.7-41.6	41.6-41.5	41.5-41.4	41.4-41.3

A number of cutting presses (automatic punching machines) are provided, each having a mark from I to VI, corresponding to the order of the fillets determined by the trier, so that all the fillets are cut out automatically into blanks by the cutting presses having the same mark as the fillets.

The idea of this system is to minimize the inequalities of rolling.

It is one of the most important operation in minting.

All the blanks cut out from the fillets are now assembled and sorted by workmen for "milling," i. e., compressing on the edge by a radial inward pressure, each blank being passed through in succession between a curved passage of rounded grooves, so that the periphery of the blank is a little swollen up.

The blanks are next operated in centre-punching presses where they are perforated in the exact center one after another.

d) Annealing and Cleaning of the Blanks.

The annealing is done by heating in iron boxes, shown in Fig. (9), sealed with clay, in an annealing furnace, gas or coal fired, as in Fig. (6) & (7).

It will take about 1 hour to anneal the blanks at the working temperature of  $650^{\circ}C$ .

After annealing, the blanks are tumbled in a wooden barrel containing dilute sulphuric acid mixed with rice hulls. Then, they are washed with fresh water over and over again to take off all trace of the acid, and thoroughly dried upon a steam drying table. They are now ready for being put in the Coining Presses.

#### 5) Equilibrium diagram for Cupro-nickel alloys.

According to the researches of B. Roozeboom and others the cooling phenomena of Cupro-nickel alloy are shown in the following Solidification Diagram.

In Fig. (10), the abscissae denote the percentage contents of the nickel and copper, and the ordinates temperature (Cuertler & Tamman).

There is now for each melt a temperature at which mixed crystals begin to separate. The locus of these points is marked Cl; or the line CpnD.

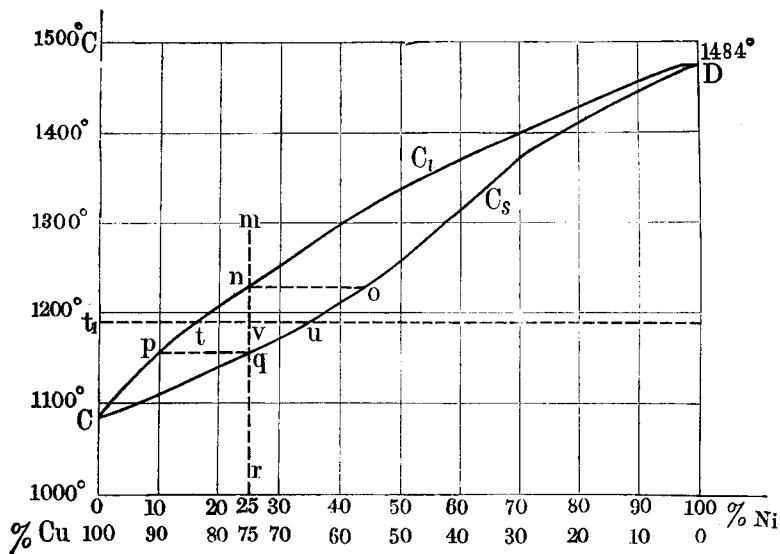


Fig. 10.  
Solidification diagram of Cupro-nickel alloy.

Further there exists a second point at which freezing is at an end ; these points joined by a line give C<sub>s</sub> ; or C<sub>q</sub>oD. The former is called the liquidus curve and the latter the solidus curve.

The melt of composition mr, if cooled to point n, deposits mixed crystals of composition corresponding to the point o, and thereby becomes richer in the component of copper. Consequently from the same melt there will separate, along the curve oC, a series of mixed crystals increasingly richer in copper. The curve nC gives the composition of the corresponding melts.

If it be desired to know the relative amount of mixed crystals formed from a mixture of the composition (75% copper, 25% nickel) at a temperature t<sub>1</sub>, it may be readily found from the diagram. If a line t<sub>1</sub>tvu be drawn horizontally through t<sub>1</sub>, then the abscissa of t will indicate the composition of the fused liquid and the abscissa of u the composition of the mixed crystals.

But the average composition will be represented by v, and therefore

$$\frac{\text{Weight of the mixed crystals}}{\text{Weight of the remaining liquid}} = \frac{vt}{vu}.$$

The above relation holds good for melts and mixed crystals following the lines np and oq respectively. In the vicinity of the line pq the residual liquid is about nil and the alloy consists almost entirely of mixed crystals.

The crystals separating last of all of the composition q were crystallized from a mother liquor of the composition p.

The originally separated crystals which were poorer in copper equalize their composition little by little as they absorb from the mother-liquor a corresponding amount of copper, so that finally the whole mass consists of homogeneous mixed crystals of the composition mr. To this end it is of course necessary that the cooling proceeds so slowly that this enrichment of the already solid mass by diffusion of the constituent of copper is possible. If this does not take place, the ensuing aggregate of mixed crystals is heterogeneous and the freezing temperature of the latter falls lower than expected from the concentration.

#### 6) Liquation of Cupro-nickel alloy.

##### a) Cooling phenomena of the ingot bar.

We may consider the cooling phenomena of the coinage cupro-nickel bar in the practical operation, according to the general rule described above.

The cast iron moulds in use at the Mint are shown in Fig. (11),

The moulds are put in upright position and the melted alloy is poured in. Therefore, we can suppose that the liquid metal will at first be solidified from the very bottom of the mould which is the coldest part of it, comparatively speaking. While the cooling proceeds, the heat emitted by the metal is given to the mould, heating it up to a higher temperature, and retarding the cooling action of the melted metal.

Further, we can suppose that the central part of the bar is cooled more slowly than the surface.

The annexed illustrations show these relations very distinctly; viz.,

1) The surface of the bottom is most finely crystallized due to the rapid cooling.



- 2) The surface of the middle part is next in fineness.
- 3) The surface of the top is coarser than the middle.
- 4) The central part of the bar is less heterogeneous than the surface, showing the effect of diffusion due to the comparatively slow cooling.
- 5) The central parts of the middle and the top show the coarsest structures of all.

Fig. 12 to Fig. 17 illustrate the appearances of the alloy, etched with alcoholic solution of ferric chloride ( $\text{FeCl}_3$ ).

Hence we know that to diminish heterogeneity it is better to heat the mould beforehand as high as possible before pouring in the melt.

- b) Effect of annealing on the internal structure of the bars.

Next, we consider the effect of the annealing of the bars. Fig. 18 to Fig. 23 show the internal change of structure of ingot-bars by annealing at different temperatures for 1 hour.

- c) Effect of annealing on the internal structure of the fillets.

Fig. 24 to Fig. 33 are the illustrations of fillets, reduced by rolling, to  $\frac{1}{3}$  thickness of the original bars. It is obvious that the structure becomes more homogeneous by annealing and that a high heating is much more efficient than a low.

- d) Effect of annealing on the internal structure of the blanks.

We have tried to investigate the hardness of the new type 5-Sen blanks annealed at different temperatures; the result is shown in the next table.

Here, the blanks were heated in a wrought iron pipe, containing sand mixed with carbon powder, and after heating for 1 hour & 10 min. they were cooled in hot water. The furnace used was of a Tube Chamber Type made by the Hoskins Manufacturing Co., Detroit, Mich., U. S. A. The temperature was measured with a pyrometer of Platinum and Platinum-rhodium thermo-couple made by Siemens Halske, Berlin, Germany.

The hardness of blanks after annealing in this test is generally lower than in the case of the Hardness Tests on page 7-9.

The reason why is as follows:—

In this experiment only one blank was taken in each test and each blank was cut into two pieces diametrically. The one half was examined with the microscope and the other half was annealed at 500°C., 600°C., 650°C., 700°C., 750°C., 800°C., 850°C. and 900°C. respectively and examined as before; In this case the individual half blank was packed in a wrought iron pipe as stated before. Therefore, the heating effect was more efficient than in the case of the Hardness Tests on pages 7-9. It is another reason that the thickness of the new type 5-Sen blank is much less than that of the old type.

Test No.	Annealing temp. C.	Hardness by Shore's Scleroscope.			Remarks.
		Before annealing.	After annealing.	Difference.	
1	500°	65.0	30.0	35.0	
2	600°	65.0	21.5	43.5	
3	650°	61.0	21.5	39.5	
4	700°	63.0	19.5	43.5	
5	750°	65.0	21.5	43.5	
6	800°	65.0	19.0	46.0	
7	850°	60.0	18.0	42.0	
8	900°	60.0	17.0	43.0	

Fig. 34 to Fig. 47 are the illustrations of the microscopic structure of the above new type 5-Sen blanks, before and after annealing.

The even number of the figures represents the upper half of a blank and the next number the lower half of the same blank annealed respectively.

We find:—

1) A part of blank in Fig. 34 slightly changed into white in Fig. 35, indicating that a diffusion between different phases begins, but the strata due to rolling effect are clearly visible.

2) In Fig. 36 & 37, the above change is more clearly visible, and strata decrease gradually.

3) In Fig. 38 & 39, the above phenomenon becomes severely marked.

4) In Fig. 40 & 41, the change is more severe, but the strata yet remain.

5) In Fig. 42 & 43, the diffusion is very much advanced and the strata are scarcely seen.

6) In Fig. 44 & 45, the diffusion is almost completed and the strata have almost vanished.

7) In Fig. 46 & 47, the phase being uniform, Fig. 47 shows a perfectly homogeneous solid solution.

The above figures illustrate the effects of annealing for the same duration of time, i. e., 70 min.; if we prolong the time of annealing similar effects can be seen though the temperature of annealing be lower.

Fig. 48 to Fig. 51 are a few examples of these.

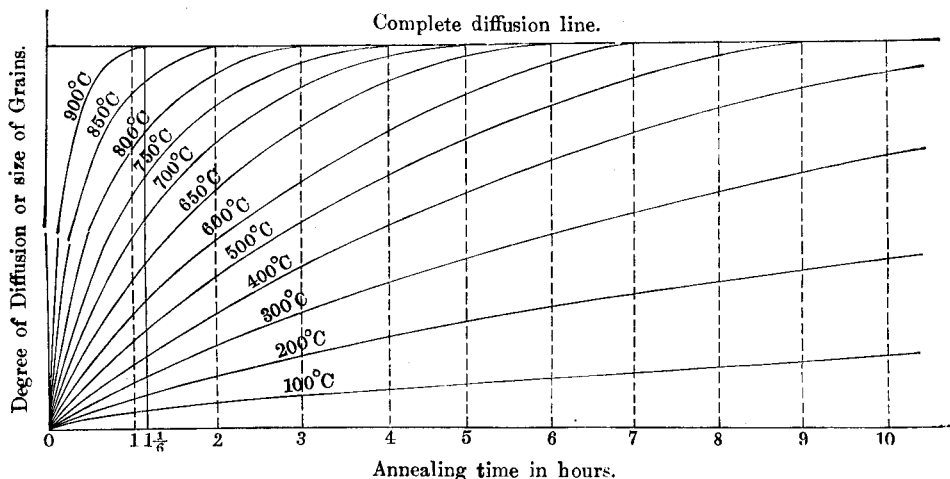


Fig. 52.

Supposed-diffusion diagram of cupro-nickel in annealing.

In Fig. 52, we see that we can get an almost homogeneous solid solution after a prolonged annealing of 8 hours at a comparatively low temperature of  $600^{\circ}\text{C}$ . and also the same effect after 5 hours' annealing at  $800^{\circ}\text{C}$ . Moreover, we can clearly see in Fig. 51, a fine net-work forming the boundaries of the intercrystalline structure of the alloy; it is not brittle in spite of the net form, because there is contained therein no foreign substance between the boundaries of the crystals.

## e) Internal structure of the brittle blanks.

In practical operations, the blanks which have been annealed at a high temperature are soft and show a homogeneous structure, as shown in Fig. 47.

But, if there are contained in the metal some impurities, for instance, oxide, the structure is broken by overheating; in other words, a heavy network extending over the whole structure is developed, as shown in Fig. 55 & Fig. 56.

The illustrations of brittle blanks presumably caused by some impurity are shown in Fig. 53 to Fig. 56. They were found in the actual operation of the old type 5-Sen blanks.

## f) Annealing of the scale-like blanks.

We have made experiments in annealing, at different temperatures using the scale-like blanks found till June, 1920, in the daily operation; the result is shown in the next table.

Test No.	Annealing temp. in C.	Annealing time.		No. of blanks bent 90°.			Remarks.
		Hour.	Min.	Torn.	Broken.	Total.	Cooling.
1	650°	1	10	10	0	10	In hot water.
2	700°	1	10	10	0	10	„
3	770°	1	10	10	0	10	„
4	780°	1	10	10	0	10	„
5	790°	1	10	9	1	10	„
6	800°	1	10	5	5	10	„
7	810°	1	10	5	5	10	„
8	820°	1	10	2	8	10	„
9	830°	1	10	3	7	10	„
10	840°	1	10	3	7	10	„
11	850°	1	10	0	10	10	„
12	900°	1	10	0	10	10	„

The micro-structure of the above is shown in Fig. 57 to Fig. 62.

## g) Influence of oxygen on the internal structure of the Cu-Ni alloy.

We can clearly perceive the heavy black net-work on the white ground

of Fig. 60 to Fig. 62. It is supposed to be an injurious matter introduced into the metal during the operations, but it is found only in the blanks heated higher than  $790^{\circ}C$ . The intercrystalline appearance in the embrittled blank is rather similar to that which occurs on "burning" nickel and Monel metal. It has been said, by some writers<sup>1</sup> that "this brittleness is considered as being associated with the precipitation of graphitic carbon and on this account manufacturers of this metal aim to keep the percentage of total carbon below about 0.04"

We have executed the carbon analysis of an exceedingly brittle blank of a 5-Sen nickel and found that it contains only 0.077% of carbon; and also that a tough blank of a 10-Sen nickel annealed at  $900^{\circ}C$ . contains 0.072% of carbon. Both are higher than 0.04%, and the one is exceedingly brittle while the other is very tough.

From the above we can say that carbon is not an important element but oxygen in the form of an oxide is one of the most injurious substances in the working of the coinage nickel.

Fig. 63 to Fig. 69 are examples showing the effect of oxygen of the different percentages.

### PART III. CHEMICAL INVESTIGATION OF THE BRITTLE CUPRO-NICKEL BLANKS.

#### 7) Tests for injurious matter causing brittleness.

We collected the malleable blanks only which, after annealing at high temperature, were bent  $90^{\circ}$  by hammer test, and added cupric oxide (CuO) in different amounts by melting together in a plumbago crucible, and then blanks were made as usual from the bar.

These blanks were annealed at different temperatures and subjected to bending tests; the record is as follows:—

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1) *Chemical & Metallurgical Engineering*, March 30, 1921. Vol. XXIV, No. 13, pp. 559.

Test No.	Percentage added.		Annealing temperature in C.				Remarks.
	CuO.	Oxygen (approx).	600°	700°	800°	850°	
1	2.000	0.400	bent	bent	crushed	broken	
2	1.000	0.200	"	"	"	crushed	
3	0.500	0.100	"	<u>crushed</u>	<u>"</u>	<u>"</u>	
4	0.250	0.050	"	bent	"	"	
5	0.125	0.025	<u>"</u>	<u>"</u>	bent	broken	
6	0.065	0.013	"	"	"	"	
7	0.033	0.007	"	"	broken	crushed	

N.B. The bars from which the blanks were cut out were annealed at 600°C. for 70 min. The original blanks taken as material may be considered to have already contained some oxide, therefore the actual percentage of oxygen in the above list would be somewhat higher.

The micro-structures of the underlined blanks in the above list are shown in Figs. 63 to Fig. 67.

Here we see that the blanks intentionally mixed with cupric oxide have the similar intercrystalline appearances as the brittle blanks which appeared in the course of the daily working in the Mint. We also see that the blanks annealed at from 600°C. to 700°C., though they contain a sufficient amount of the injurious matter, do not show a net-work as in Fig. 56, or do not form an injurious structure as can be seen in the above list, except No. 3 which was annealed at 700°C.

#### 8) Chemical composition of the brittle blanks.

The analysis was executed by Mr. Aboshi, under the supervision of Dr. Koga, the superintendent of the Assay Department, and Mr. Shibata, chief assayer, with very great care.

##### a) Composition of old type 5-Sen nickel blank crushed.

	First analysis.	Second analysis.	Average.	Remarks.
	%	%	%	
Cu	74.480	74.700	74.590	
Ni	25.020	24.920	24.970	
Fe	0.250	0.200	0.225	
S	0.056	0.056	0.056	
Mg	trace	trace	trace	

	First analysis.	Second analysis.	Average.	Remarks.
	%	%	%	
Pb	trace	trace	trace	Carbon was estimated later.
O	0.0667-0.0645	0.0697-0.0645	0.06635	
C	0.077			
Total	99.9486	99.9431		

b) Composition of new type 10-Sen malleable blank annealed at 900°C.

	First analysis.	Second analysis.	Average.	Remarks.
	%	%	%	
S	0.0623	0.0590	0.0606	Rather greater than in a).
Fe	0.1900	0.2000	0.1950	
O	0.0239	0.0289	0.0264	Exceedingly less than in a).
C	0.0720	—	—	Carbon was estimated latter.

9) Influence of oxygen added intentionally.

Both the nickel and copper used for the tests were new materials, the former being shot nickel from The International Nickel Co. and the latter electrolytic copper.

At first, a certain amount of cupric oxide was added into the above, and bars containing theoretically 0.05% of oxygen were cast with careful precaution to prevent oxidation. The cast bars had a very good appearance, but after annealing and rolling, the blanks cut out from the bars were found to be brittle.

Next, we made blanks according to the same process except that there was added no cupric oxide.

Finally, we made blanks containing 0.025% of oxygen with the same process as above.

The 3 Tests were as follows:—

Annealing temp. in C.	First test	Second test.	Third test.	Annealing period.	Remarks.
	0.05%O.	0.025%O.	No. oxygen.		
685°	all bent	all bent	all bent	90 min	good
785°	broken or crushed	"	"	"	
885°	all crushed	"	"	"	
blanks tested	30 pcs	15 pcs	15 pcs	"	

In the above list, we see that those containing 0.05% of oxygen shew an inferior quality, when annealed at a higher temperature than 785°C. and also that when annealed at 685°C. they did not show any bad effect.

All the blanks containing 0.025% of oxygen or less were safe at all temperatures.

b) Lastly, we made tests to find out the critical point at which oxygen does harm, by adding cupric oxide to fresh materials of copper and nickel.

The results are as follows:—

Percent oxygen.	Annealing temp. in C.	Cooling temp. in C.	Annealing period.	Test bent 90°.				
				Tough.	Torn.	Brkn.	Crshed.	Total.
0.020%	850°	100°	1 hour	20	0	0	0	20
"	800°	"	"	20	0	0	0	20
"	750°	"	"	20	0	0	0	20
"	700°	"	"	20	0	0	0	20
"	650°	"	"	20	0	0	0	20
0.025%	850°	"	"	20	0	0	0	20
"	800°	"	"	20	0	0	0	20
"	750°	"	"	20	0	0	0	20
"	700°	"	"	20	0	0	0	20
"	650°	"	"	20	0	0	0	20
0.026%	850°	"	"	1	5	8	6	20
"	800°	"	"	15	5	0	0	20
"	750°	"	"	20	0	0	0	20
"	700°	"	"	20	0	0	0	20
"	650°	"	"	20	0	0	0	20
0.027%	850°	"	"	2	6	1	1	10
"	800°	"	"	0	1	1	8	10
"	750°	"	"	6	4	0	0	10
"	700°	"	"	0	9	1	0	10
"	650°	"	"	10	0	0	0	10
0.028%	850°	"	"	0	0	0	20	20
"	800°	"	"	0	8	1	11	20
"	750°	"	"	7	11	0	2	20
"	700°	"	"	4	2	0	14	20
"	650°	"	"	20	0	0	0	20
0.029%	850°	"	"	0	0	0	10	10
"	800°	"	"	0	0	0	10	10
"	750°	"	"	0	0	0	10	10
"	700°	"	"	0	6	4	0	10



Percent oxygen.	Annealing temp. in C.	Cooling temp. in C.	Annealing period.	Test bent 90°.				
				Tough.	Torn.	Brkn.	Crshed.	Total.
0.029%	650°	100°	1 hour	8	2	0	0	10
0.030%	850°	"	"	0	0	0	20	20
"	800°	"	"	0	0	0	20	20
"	750°	"	"	0	2	0	18	20
"	700°	"	"	0	2	9	9	20
"	650°	"	"	19	1	0	0	20
0.035%	850°	"	"	0	0	0	20	20
"	800°	"	"	0	0	0	20	20
"	750°	"	"	0	0	0	20	20
"	700°	"	"	0	0	0	20	20
"	650°	"	"	4	16	0	0	20

*N. B.* The percentage of oxygen in the above list is the amount of oxygen included in the cupric oxide added.

Therefore, we can say that oxygen of less than 0.025% in oxide form does not practically destroy the intercrystalline structure of the alloy, and that even with more than 0.05% of oxygen when annealed at a temperature of less than 685°C. for 1 hour a similar good effect can be obtained. As to the critical point for oxygen doing harm we may say that it is that which corresponds to 0.026% of oxygen in the alloy, according to the above tests.

#### 10) Oxidation of Cupro-nickel and its reduction.

The oxidation commonly occurs during melting, pouring and annealing; but the last is the most important. The amount of increase of weight by oxidation at the annealing of cast bars at about 650°C. during 70 min. under direct fire was ascertained to be about 0.0132% by our experiments, the determination being made as follows:

A small test piece similar to the ingot-bar cut out from a bar was carefully finished at the surface, weighed precisely before and after the annealing, which was done in the usual manner. The average increase of weight was found to be 0.0132% which is the weight of oxygen combined with the constituents. Besides the test piece, the increase of weight of the

actual bars by annealing was ascertained by weighing the bars as well as the detached coatings of oxide which were carefully collected.

The average figures were found to be not very far removed from the result obtained with the test piece.

We may therefore suppose that an annealed bar contains at least 0.0132% of oxygen. About 40% of these bars in the form of scissels or imperfect blanks are usually mixed with new materials which contain practically no oxygen, and when cast in bars, they are annealed again, thereby being made to contain at least 0.01848% of oxygen.

$$0.0132 + 0.0132 \times .4 = 0.01848\% \text{ of oxygen.}$$

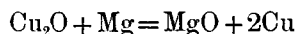
This process of remelting old material is repeated over and over again, thus gradually increasing the amount of oxygen; therefore we have to reduce the oxide by the use of some deoxidizing agent.

#### 11) Deoxidation of cupro-nickel.

a) We took new materials of nickel and copper as mentioned in 4) and added, such an amount of cupric oxide as to contain 0.05% of oxygen, by melting together as usual, taking good care to prevent further oxidation.

Reducing agent, i. e., magnesium in the form of magnesium copper, in amount of 50% more than that theoretically required to reduce all oxide, was mixed into the molten metal which was well stirred and poured into a mould and so on.

According to the equation



the amount mixed was 0.116% of Mg or 0.232% of magnesium copper of 50%.

The cast bars had a very bad appearance, but after annealing and rolling, the blanks cut out from them were found to be all malleable as shown in the following table:

Annealing temp. in C.	No. of pieces.	Annealing period, in min.	Bending 90°.			Remarks.
			Magnesium added.			
			0.116%	0.0766%	0.010%	
685°	15	90	a) 15 bent	b) 15 bent	c) 15 bent	The underlined bent 180°
785°	15	90	15 bent	15 bent	{ 4 torn 5 broken 6 crushed	
885°	15	90	<u>15 bent</u>	15 bent	{ 4 torn 11 crushed	
900°	470	100	470 bent			
Oxygen content			trace	trace	0.04%	By analysis

b) The same as a) except that the amount of magnesium added was 0.0766% instead of 0.116%.

c) The same as a) except that the amount of magnesium added was 0.01% instead of 0.116%.

From the above list, we see that tests a) and b) show good results, but in the test c) the de-oxidation was insufficient.

As already stated, the increase of weight by oxidation in the annealing of a bar under direct fire at about 650°C. during 70 min. is 0.0132% on the average; therefore about 2 momme of Mg. is required theoretically for 10 Kwan of the scissels from the annealed bar as above mentioned.

$$10 \times \frac{0.0132}{100} = 0.00132 \text{ Kwan of oxygen.}$$

$$0.00132 \times \frac{24.3}{16} = 0.002 \text{ Kwan of magnesium.}$$

or 2 momme of pure magnesium or 4 momme of 50% magnesium-copper.

In practice, however, more than 4 momme of magnesium-copper is required for every 10 Kwan of scissels, due to oxidation during melting and pouring.

### 12) Influence of sulphur upon brittleness.

To investigate the injurious effect of sulphur contained in the cupro-nickel alloy we made several experiments by adding intentionally various amounts of sulphur in the form of copper sulphide into new materials of copper and nickel. The results being not satisfactory we repeated the same

tests 3 times and found, after analyses, that the sulphur content of each sample was not more than 0.048%, notwithstanding the fact that we added at most 0.20% sulphur as copper sulphide. It is supposed that a large portion of the sulphur was burnt away on the surface of the molten alloy so that only a part remained in the alloy.

The result of bending tests made after annealing at 5 different temperatures, 650°, 700°, 750°, 800°, & 850°, for 1 hour, was all good.

The percentages of sulphur added and analysed were as follows:—

Table A. List of sulphur contents.

Case No.	Sulphur contents in percent.		Case No.	Sulphur contents in percent.	
	Added.	Analysed.		Added.	Analysed.
1	0.070	0.033	6	0.120	0.030
2	0.080	0.033	7	0.140	0.041
3	0.090	0.047	8	0.160	0.037
4	0.100	0.048	9	0.180	0.044
5	0.110	0.030	10	0.200	0.036

Now we had to add more sulphur to investigate the full influence of this element upon the brittleness of the alloy; so we made further additions of copper sulphide to the ingot-bars of from Case No. 5 to Case No. 10 for the purpose of increasing the sulphur content. The result of the bending tests of these samples is shown in Table B.

We see from Table B, that the percentage of oxygen in each case was not more than 0.0192%; we can say therefore that the influence of oxygen on the brittleness in these cases was negligible, but in all cases except Case No. 1 the metal showed an inferior quality which may be considered as due to the sulphur contained in it.

The critical point of harmfulness of sulphur upon brittleness is from 0.072% to 0.076%. The ordinary coin blanks containing about 0.056% of sulphur are not thereby made brittle, therefore we may consider the sulphur as not being so dreadful as oxygen in practical operations.

The materials used for the above tests were as follows:—

Electrolytic copper	{	Copper	99.98%
		Bismuth	nil.
		Sulphur	nil.
Shot nickel	{	Nickel	98.99%
		Iron	0.52%
		Copper	0.13%
		Sulphur	trace.

Table B. List of bending tests relating to the influence of sulphur.

Case No.	Total contents in percentage.		Annealing temp. C.	No. of blanks bent 90°.			
	Sulphur.	Oxygen.		Tough.	Torn.	Broken.	Crushed.
1	0.0714	0.0186	650°	20	0	0	0
			700°	20	0	0	0
			750°	20	0	0	0
			800°	20	0	0	0
			850°	5	15	0	0
2	0.0762	0.0138	650°	20	0	0	0
			700°	9	4	7	0
			750°	0	3	0	17
			800°	2	3	2	13
			850°	0	0	0	20
3	0.0804	0.0096	650°	20	0	0	0
			700°	17	3	0	0
			750°	7	2	0	11
			800°	0	0	0	20
			850°	0	0	0	20
4	0.0908	0.0192	650°	0	4	0	16
			700°	0	0	0	20
			750°	0	0	0	20
			800°	0	0	0	20
			850°	0	0	0	20
5	0.0962	0.0188	650°	20	0	0	0
			700°	17	3	0	0
			750°	0	1	0	19
			800°	0	0	0	20
			850°	0	0	0	20
6	0.1237	0.0013	650°	20	0	0	0
			700°	1	13	6	0
			750°	0	0	0	20
			800°	0	0	0	20
			850°	0	0	0	20

## 13) Influence of carbon upon brittleness.

We have made careful analyses of the carbon content of cupro-nickel blanks; the apparatus and the method used for the purpose were as follows:—

The sample containing carbon was heated in a combustion boat put in an electric tube furnace made by Hoskins Mfg. Co., Detroit, U. S. A.

The temperature of the furnace was regulated to be about 1000°C.

Pure oxygen compressed in a steel container was passed gradually through the furnace to burn off the carbon contained in the sample. The CO<sub>2</sub> gas, the product of combustion, was absorbed by KOH solution and weighed.

From this weight was calculated the amount of carbon contained in the sample. Utmost care was taken to have the apparatus air-tight.

The result of analyses was as follows:—

Samples.	No. 1.	No. 2.
	Old type 5-Sen blank.	New type 10-Sen blank.
Property after annealing.	Brittle, easily crushed by fingers.	Malleable.
Annealing temperature.	—	900°C
Carbon content.	0.077%	0.072%

We see from the above that the effect of carbon upon brittleness is not so important as that of oxygen in the Mint operations. The practice of placing a large amount of charcoal powder upon the melted alloy to prevent oxidation, as well as the direct contact with the graphite crucible of the molten alloy does no harm.

PART IV. GENERAL CONSIDERATIONS AS SET FORTH  
BY DIFFERENT WRITERS.

## 14) Further structural investigations of the brittle blanks.

1) As shown in the foregoing pages, the structure of a brittle blank always presents a heavy dark net-work. The injurious substance which gives brittleness to the blank may be supposed to be included in the boundaries of the crystalline polyhedra of the alloy.

The substance which acts most powerfully injurious to the blank in practical working, is found to be oxygen in the form of oxide which is formed in the metal chiefly during the annealing of the bar from which it was produced. The oxide does not much spoil the malleability of the alloy if it is annealed at a comparatively low temperature, but if the temperature is so high as  $800^{\circ}C.$  or more the metal seems to be burnt by overheating.

This fact is also observed in the case of iron or soft steel as well as brass or German silver.

According to G. H. Gulliver (*Metallic Alloys*, pp. 229 & 393), the meaning of the term "burnt" differs somewhat according to the metal or alloy to which it is applied. In general, there are two cases of it; one is oxidation and the other volatilisation. Both of these actions cause an alteration which is greatest in the outer layer of the piece.

In iron or soft steel above a certain temperature atmospheric oxygen can penetrate along the crystal boundaries, giving rise to the formation of a layer of oxide; this is sufficient to destroy, almost entirely, the cohesion of a piece of moderate dimensions, and the defect can not be cured by any subsequent treatment without remelting.

When an alloy contains a volatile metal like zinc, antimony, or arsenic, the burning temperature, if it lies below the melting point, is that at which vapour begins to form within the mass; the internal pressure set up is sufficient to cause more or less complete disintegration. If the annealing temperature is very high, gases which were unable to escape during the relatively rapid solidification of the metal may be evolved within the mass; these gasses collect at the granular boundaries, where there are probably minute voids at most times, and have a disintegrating tendency, which becomes very marked when the temperature is so high that the metal had begun to soften appreciably. The grains are then forced apart and atmospheric oxygen soon penetrates the cracks and effectually completes the work of destruction.

The metal in these conditions is said to be burnt, and must be remelted to remove the defect.

Annealing at too high a temperature may result, not only in the production of an excessive coarse structure, but in the separation of the crystal grains, the metal or alloy being transversed by numerous fissures following the boundaries of the crystalline polyhedra. Such a coarse-fissured structure is characteristic of burnt steel or brass. The effect is due to the production of gas at the boundaries, which in forcing its way out of the metal or alloy, separates the grains.

2) In the Mint practice, however, the oxide is formed on the surface of the bar, which is directly fired for annealing. This oxide remains entangled in the metal by rolling. When the metal, now in form of blanks containing oxide in more than a fixed rate, is subjected to a higher temperature than about  $800^{\circ}\text{C}$ . to be annealed, the oxide seems to collect at the granular boundaries separating the crystal grains as shown in Figs. 60, 61, 62, 67 & 75.

A. Sauveur states in his book (*The Metallography and Heat Treatment of Iron and Steel*, pp. 97 & 98).

“By impurity we mean a very small proportion of some foreign substance which may be any other metal, a metalloid, or a definite compound.

The metal or metalloid contaminating the metal may (1) remain uncombined or (2) it may combine with some (generally a small amount) of the metal to form a definite chemical compound. The uncombined contaminating metal or metalloid or resulting chemical compound may then

(a) be soluble in the solid metal forming with it a ‘solid solution’ or

(b) be insoluble in the solid metal in which case it is rejected by the crystalline grains, in the form of an eutectic alloy.”

In case of coinage cupro-nickel containing oxide, if the alloy is annealed at too high a temperature, the diffusion between two phases of nickel-rich and copper-rich crystals are rapidly carried out forming solid solution and rejecting the eutectic of copper and cuprous oxide which is insoluble in the solid solution. The eutectic collects at the boundaries of the crystals and reduce cohesion between crystals.

Too prolonged annealing even at a moderate temperature may cause the same result as shown in Figs. 49, 51 & 69.



If the alloy is annealed at a moderate temperature as about  $650^{\circ}C$ . for a comparatively short period, the oxide seems to be embedded in the copper-rich part; of course in this case, only a partial diffusion between the crystals taking place, the alloy is not sufficiently soft although possible to undergo the coining process.

In Figs. 58 & 59, we can never find a polyhedral grain and the diffusion being insufficient, the copper-rich part (black) is clearly distinctive with nickel-rich part (white).

The correctness of the supposition seems to be sustained from another point of view:—namely, formerly bars were annealed after the first rolling by which the thickness of the bars was reduced to about  $\frac{1}{3}$  of the original; accordingly the length of the bar or the area of the surface of bar was increased about 3 times. When the trouble of the brittle coins arose last year, we altered the process so as to anneal the bars before rolling instead of after the first rolling. Then we found that the trouble of brittleness was decreased appreciably, that is because the reduced surface of bar induced less oxidation. This is one of the most important improvements in the Mint operations.

3) We got a new phenomenon from the brittle blanks which were annealed at a very high temperature as shown in Figs. 76 & 77. They were very tenacious instead of being brittle. All the injurious substances of the oxide or sulphide, in this case, are embedded in the black part of the alloy and do not affect the malleability.

The explanation for the phenomenon may be obtained from the following consideration:—

a) According to G. H. Gulliver (*Metallic Alloy*, pp. 230).

“A burnt brass and German silver generally show signs of incipient melting, as in Fig. 165” which is similar to Figs. 76 & 77.

“This is revealed by a thickening of the granular boundaries and the appearance of rounded spots within the grains, corresponding with the positions of the most fusible portions of the imperfectly uniform solid solution.”

b) C. H. Desch says (*Metallography*, pp. 194 & 195).

“Copper eagerly absorbs oxygen when fused, the cuprous oxide produced dissolving in the metal to form a homogeneous solution.”

“Nickel behaves in an exactly similar manner.”

“Sulphur dioxide is insoluble in solid, but very soluble in molten copper.

On freezing, a large part of the dissolved gas is retained mechanically.”

The typical solidification diagram for copper and cuprous oxide is shown in Fig. 78.

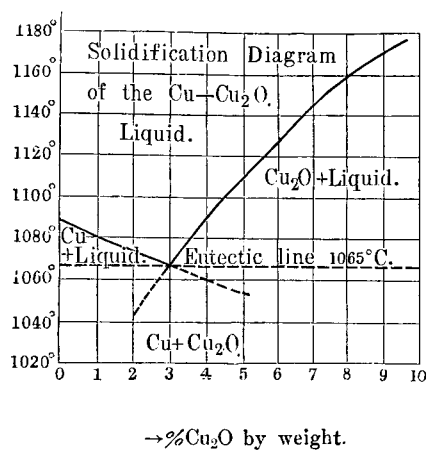


Fig. 78.

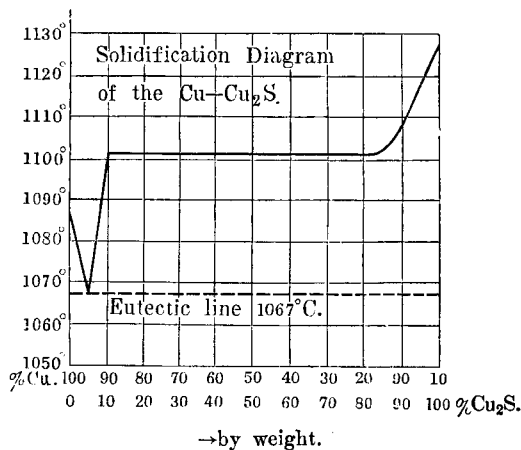


Fig. 79.

c) According to R. Schenck (Physical Chemistry of the Metals, pp. 117, 202 & 204).

“The crystallization curve for copper and copper-sulphide arrives at the eutectic point where it meets the precipitation curve for the metallic copper.”

Fig. 79 shows the equilibrium diagram.

d) According to M. Giua (Chemical Combination among Metals, pp. 296).

“The compounds  $\text{Ni}_3\text{S}_2$ ,  $\text{Ni}_6\text{S}_5$  and probably  $\text{NiS}$  occur. The system has been investigated by Bornemann,” and the diagram is shown in Fig. 80.

“From the melting point of nickel the curve falls to an eutectic point at  $644^\circ\text{C}$ . This point occurs at 33.3% sulphur, but no compound occurs.

At about  $785^{\circ}\text{C}$ . and 40% of sulphur  $\text{Ni}_3\text{S}_2$  separates; it decomposes at  $532^{\circ}\text{C}$ ., giving another crystallization species; this in turn undergoes a decomposition at  $520^{\circ}\text{C}$ ., giving a compound with the probable formulae  $\text{Ni}_6\text{S}_5$ .

In all probability the compound  $\text{NiS}$  is formed at about  $900^{\circ}\text{C}$ . and 50% of sulphur.

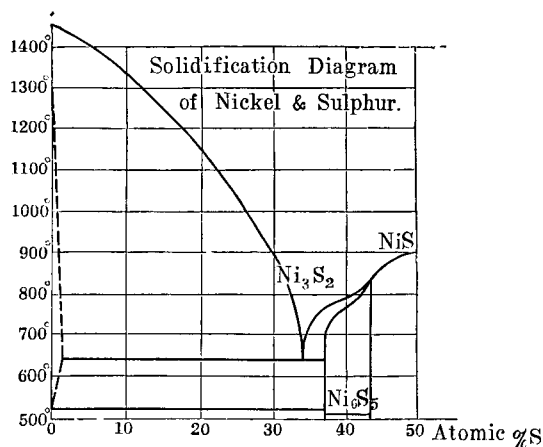


Fig. 80.

e) According to R. Schenck (*Physical Chemistry of Metals*, pp. 202).

“Nickel carbonyl  $\text{Ni}(\text{CO})_4$  is a water clear, very volatile liquid which boils at  $40^{\circ}$  and at higher temperature decomposes easily into the components from which it was formed. The ease of formation on one hand and the decomposition on the other can be demonstrated by a simple experiment. If we fill a glass tube, drawn to a point on both ends, with the vapour of nickel carbonyl and place it, after the ends have been sealed, in a boiling water bath, after a time we observe the precipitation of a nickel mirror. If then the tube be taken out and allowed to lie a few days at room temperature, the mirror disappears again with reformation of nickel carbonyl vapour.”

From the above diagram we find that the melting point of the eutectic of  $\text{Cu-Cu}_2\text{S}$  is the highest; that is  $1067^{\circ}\text{C}$ . When we overheat the alloy containing oxide or sulphide, beyond  $1067^{\circ}\text{C}$ . the eutectics formed of the

impurities with copper or nickel are remelted, but the crystals made of nickel and copper, especially the nickel-rich crystals do not melt under about  $1200^{\circ}C$ . If the annealing temperature be  $1170^{\circ}C$ . the copper-rich crystals begin to melt, dissolving all the impurities within it and rejecting the nickel-rich grains.

The latter are assembled together at random and becomes larger, finally constituting a coarser structure as seen in Figs. 76, 77, 81 & 82. Some copper-rich crystals are enclosed in the nickel-rich part when cooled, and the margin of the white part being irregularly made we can suppose it to be an assemblage of many small crystals. The small triangles of black are seen everywhere; these being crystals of impurities united with copper or nickel.

The same or similar forms are visible in the black part of the picture, as shown in Figs. 81 & 82, which are from the same sample as Figs. 76 & 77, but the etching being not so deep as in the former case we can distinguish several figures among the black part.

To demonstrate the black part of the Fig. 81 & 82 copper and cuprous oxide alloy was intentionally made by means of an electric furnace and the eutectic was examined with microscope.

According to E. F. Law ("Alloys and their industrial applications" pp, 227-228).

"Copper possesses the somewhat unique property of forming a well-defined series of alloys with its own oxide. The alloys are a simple series, the eutectic, which is shown in photograph 50, containing 3.45% of cuprous oxide ( $Cu_2O$ ), equivalent to 0.39% of oxygen. This behaviour of copper towards its oxide is of considerable importance, and must be taken into account when considering the properties of the copper alloys.

Owing to the fact that the oxide separates in the spherical and not in the laminated form its influence on the mechanical properties of the metal is relatively small. Thus Hampe states that 0.45% of copper oxide (equivalent to 13% of eutectic) in pure copper does not affect its ductility, and

only when 2.25% of oxide, or 65% of eutectic (equivalent to 0.25% of oxygen) is exceeded, does the metal short.

The influence of impurities on the condition in which oxide exists in copper is of considerable interest. Thus, arsenic, which is present in nearly all commercial coppers and which forms a solid solution with copper, causes the copper oxide particles to aggregate or "ball up" until the eutectic structure disappears. On this account the eutectic structure is never detected in commercial coppers, even though they contain considerable quantities of oxygen."

Figs. 83 & 84 show the eutectic structure of Cu & Cu<sub>2</sub>O, i. e., a structure containing 3.45% of the cuprous oxide in copper or the equivalent of about 0.39% of oxygen. The average content of arsenic in the electrolytic copper being ascertained to be 0.00085%, the coalescence of copper oxide particles in the eutectic is slightly visible in the Fig. 84.

When it is etched with diluted ammonium cuprous chloride solution, as usual, pale-blue rounded crystals of Cu<sub>2</sub>O are visible in the ground mass of the eutectic of Cu<sub>2</sub>O & Cu.

But if it is etched with ferric chloride solution dark-blue rounded crystals can be seen instead of pale-blue in the ground mass of the eutectic, consisting of very fine crystals of triangular forms, though they are not clear, as shown in Fig. 83.

If it is etched with a very dilute ferric chloride solution pale-blue crystals appear in ground mass of the mixture of eutectic, as shown in Fig. 84.

In Fig. 82 we can see big figures of rhombus and triangular form of pale-blue. They may be considered as Cu<sub>2</sub>O crystals in the ground mass of eutectic surrounding the big crystals of the nickel and copper alloy.

4) According to R. Schenck ("The Physical Chemistry of the Metals," pp. 204).

"It is remarkable, however, that traces of foreign substances, for example, of oxygen, cause very great variations from the normal run. They work directly as poison for the nickel and destroy its combining ability."

5) In case of brittle blanks the heavy net-work forming the crystal boundaries can be supposed chiefly to be a continuous film of the eutectic of copper and cuprous oxide as stated in 3).

6) According to C. H. Desch (Metallography, pp. 183).

“Twinned crystals are sometimes observed in cooled metals which have not been subjected to strain, and are then known as “congenital twins”; but they are more often observed in metals and alloys which have been strained and subsequently annealed. Copper, and those alloys of copper which are isomorphous with copper, the so-called  $\alpha$  solutions, show this strain-twinning very well. Plate II, B, represents a specimen of brass, the  $\alpha$  solid solution of zinc and copper, after being strained and annealed. The arrangement of parallel laminae, with alternating orientation, closely resembles that which characterizes the feldspars in igneous rocks.

The  $\beta$  solutions do not undergo twinning when strained and annealed.”

Fig. 74 in this paper closely resembles that of Plate II, B, mentioned here, which is a twinned structure of brass (Cu 70, Zn 30%) annealed.

#### 15) Conclusions.

From the foregoing experiments the following conclusions may be drawn in regard to the brittleness of the cupro-nickel alloy.

- 1) Conclusions for Part I, as stated on page 14.
- 2) Conclusions for Part II.
  - a) A rapid cooling of a cast bar produces fine sharp nickel-rich crystals in the copper-rich ground mass, but slow cooling gives a coarser structure.
  - b) By annealing there is produced a diffusion between the different phases of the alloy; the degree of diffusion depends both upon the temperature as well as the duration of annealing.
  - c) The most efficient annealing or homogeneous solid solution of the alloy can be obtained at from  $800^{\circ}C.$  to  $900^{\circ}C.$  sustained for about 1 hour, but if we prolong the period of annealing the same effect can be obtained at a comparatively low temperature.
  - d) If the alloy contains some injurious substance as, for instance, oxygen, a thorough annealing or complete diffusion makes it brittle and a heavy net-work of oxide or other substances is produced.
  - e) In such a case, we have to be contented with subjecting the alloy to a half annealing or imperfect diffusion obtained by annealing at from  $600^{\circ}C.$  to  $700^{\circ}C.$ ; and  $650^{\circ}C.$  is the most proper temperature for practical purposes in the Mint.

3) Conclusions for Part III.

- a) The most injurious substances causing the brittleness of the coinage alloy is oxygen in the form of an oxide; its limit of doing harm may be considered as about from 0.026% to 0.030% total oxygen.
- b) The oxygen can be reduced by introducing a piece of magnesium-copper into the molten alloy contained in the crucible before pouring.

The amount of the magnesium-copper to be used depends upon the contents of oxygen.

- c) The injurious effect of sulphur is similar to that of oxygen; the limit of doing harm may be considered as about from 0.072% to 0.076% total sulphur; but the probability of getting actual content of more than the above limit is much less than in the case of oxygen, so that we need not be afraid about the injury from sulphur in practical operations.
- d) Carbon is not so injurious as oxygen or sulphur in our practical working.

4) Conclusions for Part IV.

- a) The most poisonous substance in the impurities of the cupro-nickel alloy is the eutectic of copper and cuprous oxide which extends as a continuous film between the crystalline polyhedra of the cupro-nickel alloy.
- b) If the alloy contains a proper amount of arsenic with respect to the oxide the injurious influences can be lessened or sometimes entirely removed due to the balling up of the eutectic layers.

I am greatly indebted to Mr. Y. Kōga, Kōgaku-hakushi, Superintendent of the Assay Department, for the supervision of assays which were undertaken by Mr. T. Aboshi, and Mr. S. Yamada, Accountant of the Operative Department, with the assistances of Mr. M. Masuoka, for taking records of the annealing and hardness tests, and lastly to Mr. T. Aboshi, metallurgist to the Operative Department, for the enormous amount of labour incident to taking photographs and making chemical analyses throughout the experiments.

THE END.

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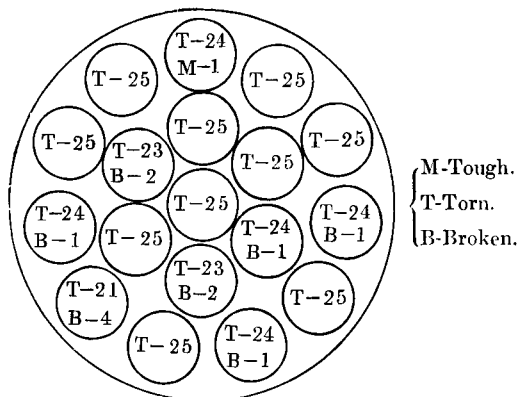
TABLE OF EXPLANATION OF THE ATTACHED FIGURES  
FROM MICROSCOPE.

Fig. No.	Magnified.	Polished face	Sample.	Degree of annealing.	Duration of heating.	Rmks.
12	65	Surface.	Bottom of ingot-bar cast.	No.	No.	
13	"	Section.	" "	"	"	
14	"	Surface.	Middle "	"	"	
15	"	Section.	" "	"	"	
16	"	Surface.	Top "	"	"	
17	"	Section.	" "	"	"	
18	"	Surface.	Ingot-bar.	Before.	"	
19	"	"	"	580°C.	60 min.	
20	"	"	"	Before.	"	
21	"	"	"	800°C.	"	
22	230	"	"	900°C.	"	
23	"	Section.	"	"	"	
24	55	Surface.	Fillet, 1/3 thick of bar.	Before.	"	
25	"	"	" "	500°C.	"	
26	"	Section.	" "	Before.	"	
27	"	"	" "	600°C.	"	
28	"	"	" "	Before.	"	
29	"	"	" "	700°C.	"	
30	"	"	" "	Before.	"	
31	"	"	" "	800°C.	"	
32	"	"	" "	Before.	"	
33	"	"	" "	900°C.	"	
34	65	Surface.	New 5-Sen blank.	Before.	"	
35	"	"	"	600°C.	70 min.	
36	"	"	"	Before.	"	
37	"	"	"	650°C.	"	
38	"	"	"	Before.	"	
39	"	"	"	700°C.	"	
40	"	"	"	Before.	"	
41	"	"	"	750°C.	"	
42	"	"	"	Before.	"	
43	"	"	"	800°C.	"	
44	"	"	"	Before.	"	
45	"	"	"	850°C.	"	
46	"	"	"	Before.	"	
47	"	"	"	900°C.	"	
48	430	"	"	Before.	"	



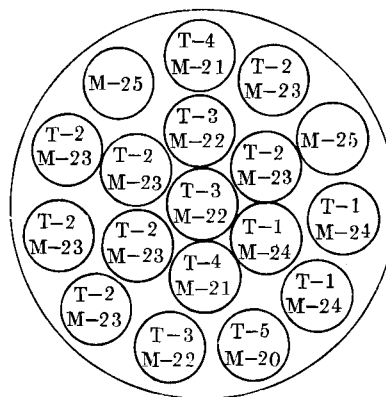
Fig. No.	Magnified.	Polished face.	Sample.	Degree of annealing.	Duration of heating.	Remarks.
49	430	Surface.	New 5-Sen blank.	600°C.	8 hours.	
50	"	"	"	Before.		
51	"	"	"	800°C.	5 hours.	
53	250	"	Crushed, Old 5-Sen blank.	—	—	
54	"	"	"	—	—	
55	430	"	"	—	—	
56	"	Section.	"	—	—	
57	65	Surface.	Squamous,	Before.		
58	300	"	"	650°C.	70 min.	Tough
59	250	"	"	770°C.	"	"
60	430	"	"	790°C.	"	Brittle.
61	250	"	"	840°C.	"	"
62	300	"	"	850°C.	"	"
63	280	"	Blank containing 0.0139% of O.	600°C.	60 min.	Tough.
64	"	"	"	700°C.	"	"
65	"	"	" 0.0555%	"	"	Crushed.
66	"	"	"	800°C.	"	"
67	230	"	"	859°C.	"	"
68	430	Section.	" 0.035%	700°C.	"	Tough.
69	"	"	"	"	10 hours.	Brittle.
70	100	"	Perfectly deoxidized ingot-bar.	—	—	
71	430	"	" " "	—	—	
72	100	"	Imperfectly deoxidized "	—	—	
73	430	"	" " "	—	—	
74	100	Surface.	Perfectly deoxidized blank.	900°C.	100 min.	Tough.
75	430	"	Imperfectly deoxidized blank.	850°C.	90 "	Brittle.
76	100	Section.	Originally brittle blank.	To half melt.		Tough.
77	430	"	"	"		"
81	100	"	"	"		
82	430	"	"	"		
83	"	"	Alloy of Cu & Cu <sub>2</sub> O, etched with FeCl <sub>3</sub> solution.			
84	"	"	" " very diluted FeCl <sub>3</sub> .			

*N. B.* All the samples were etched with a saturated solution of ferric-chloride except those of Figs, 81, 82 & 84, which were etched with a very diluted ferric chloride solution.



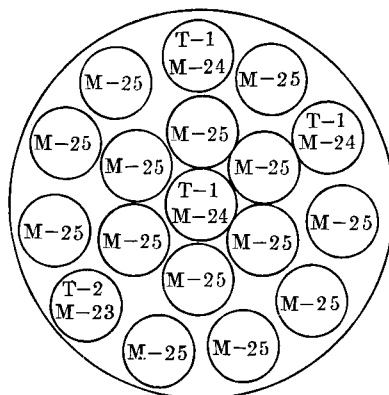
Heated for 30 min.

Fig. 1.



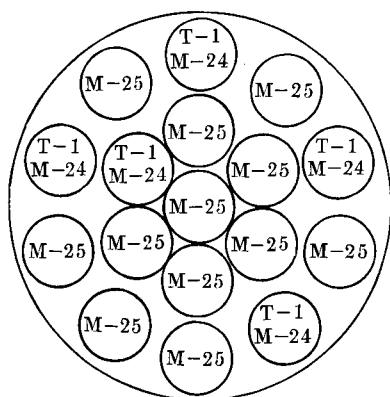
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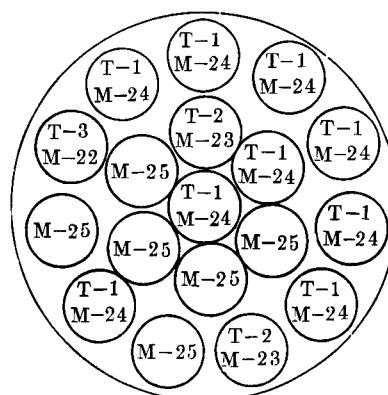
Heated for 60 min.

Fig. 3.



Heated for 75 min.

Fig. 4.



Heated for 90 min.

Fig. 5.

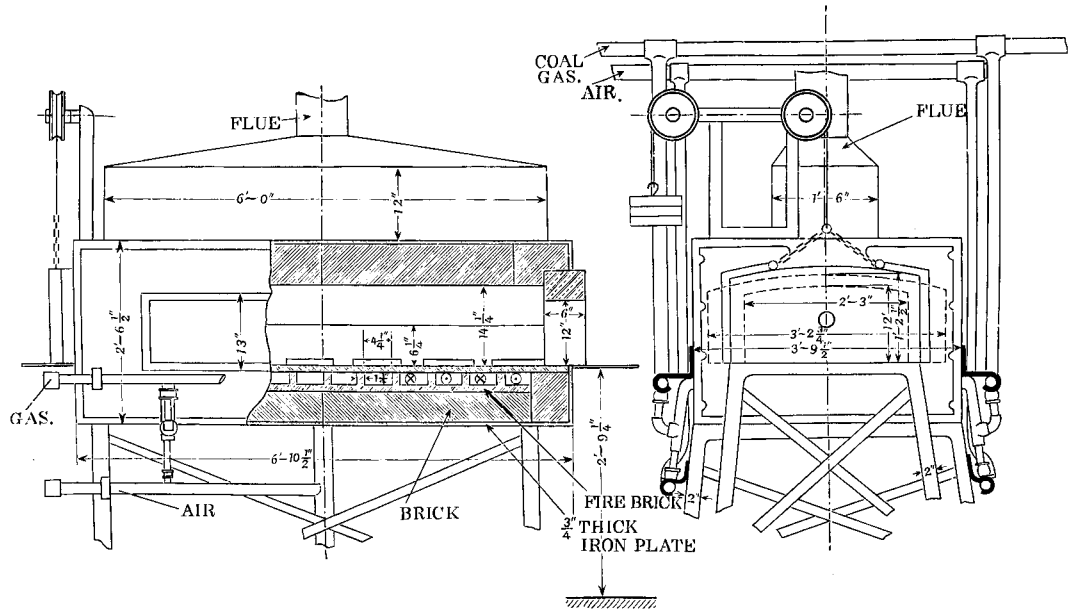


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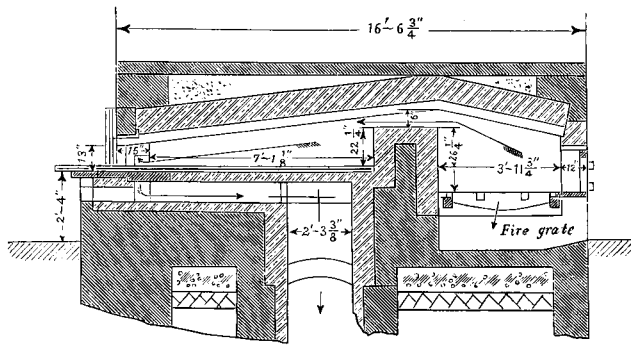


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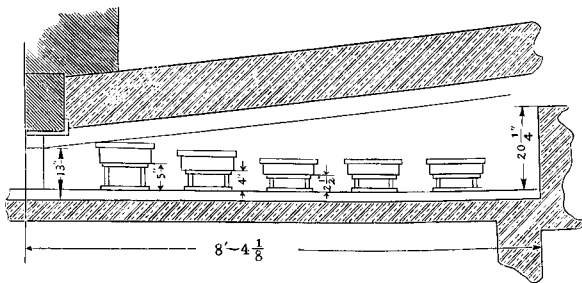


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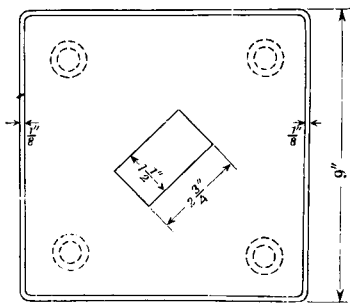
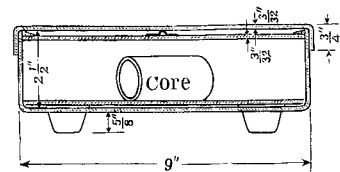


Fig. 9.

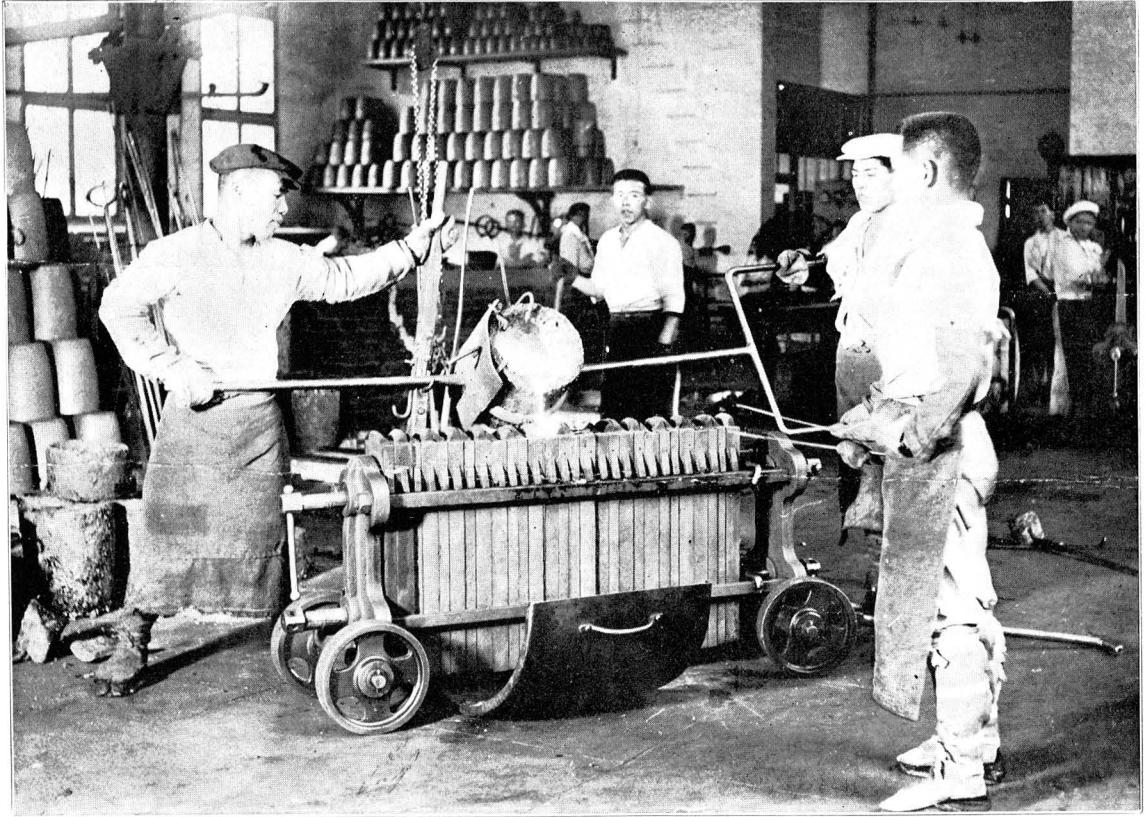


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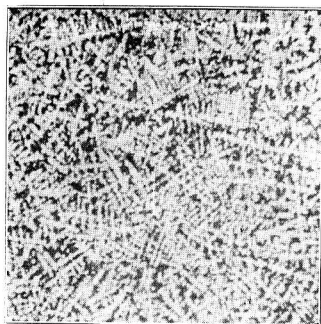


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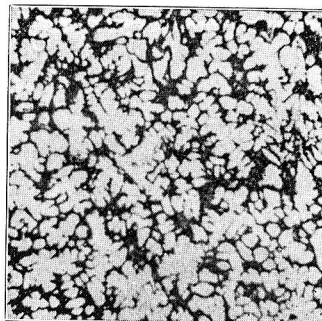


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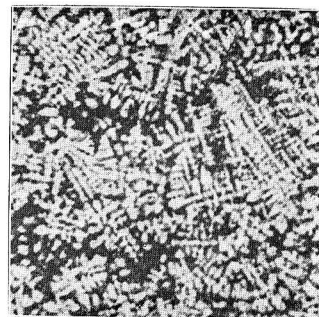


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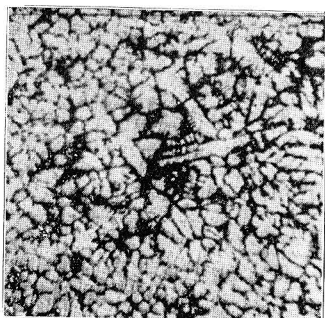


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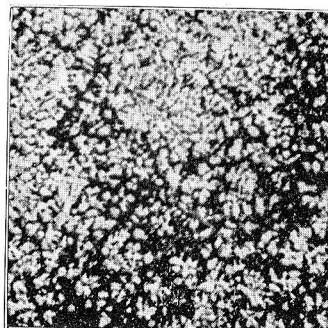


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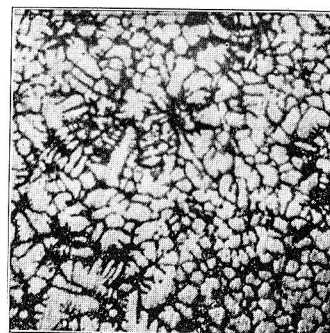


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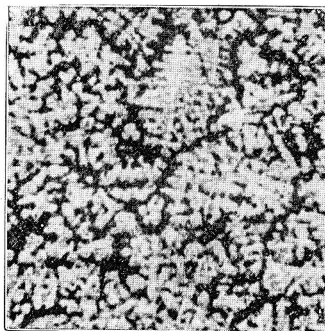


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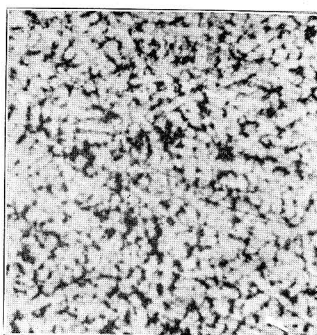


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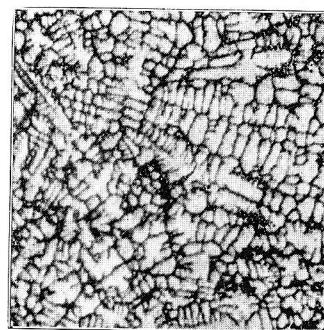


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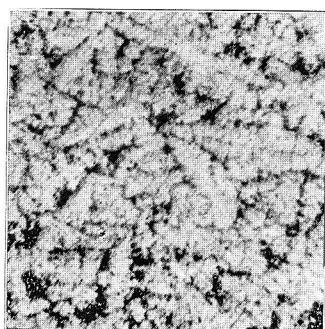


Fig. 21.



Fig. 22.

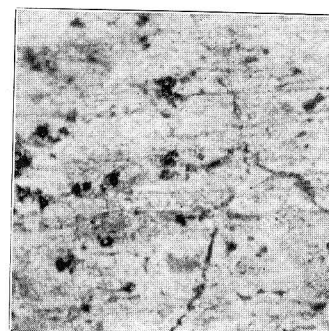


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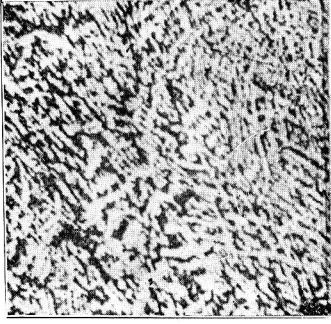


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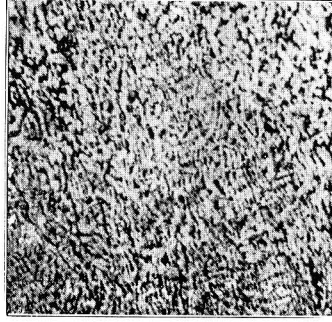


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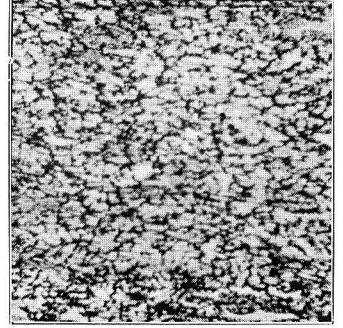


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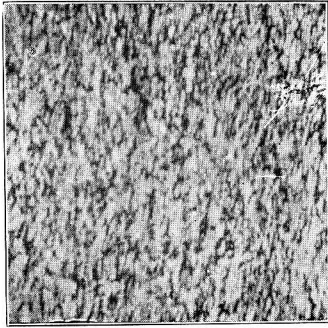


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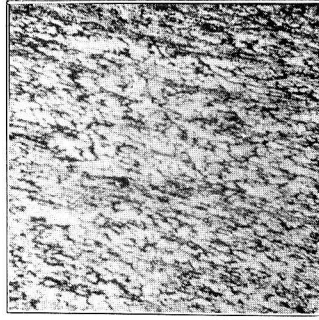


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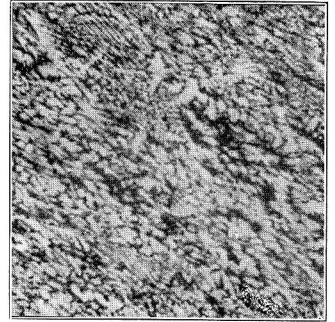


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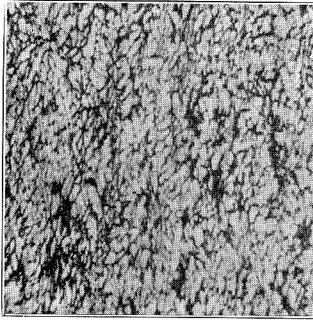


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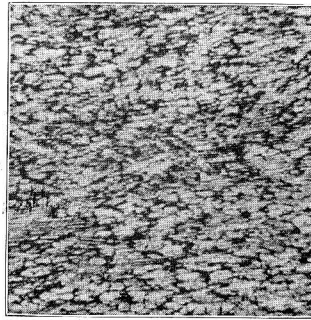


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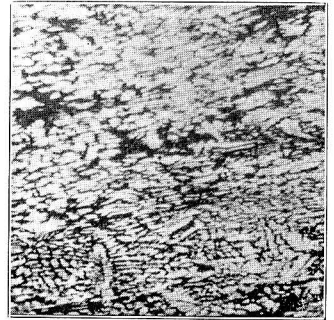


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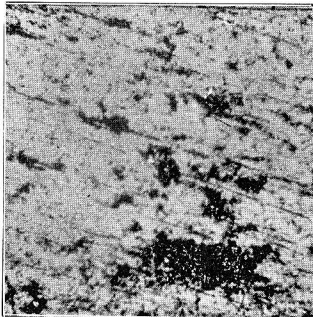


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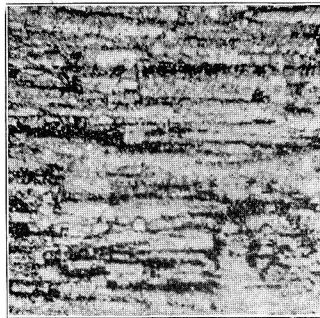


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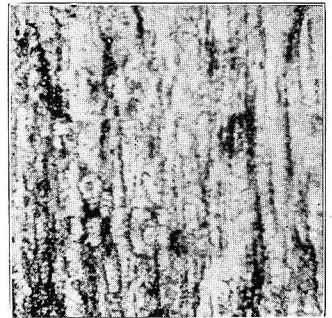


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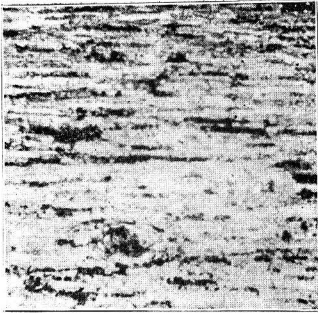


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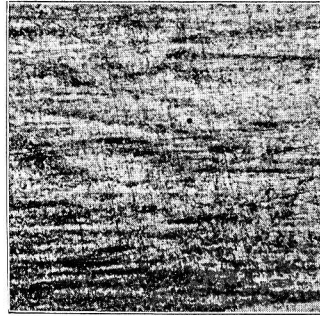


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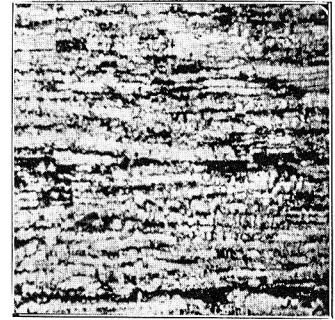


Fig. 38.



Fig. 39.

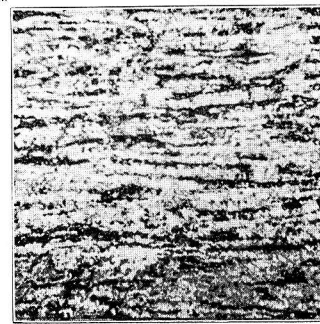


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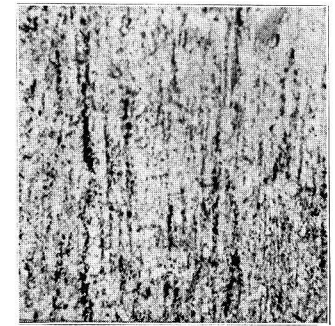


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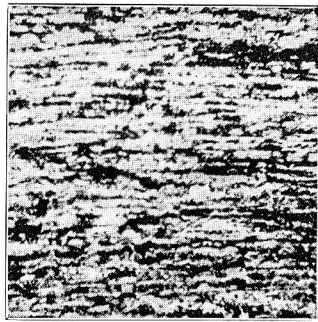


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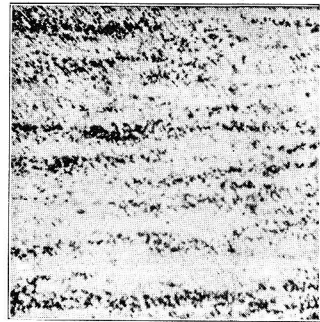


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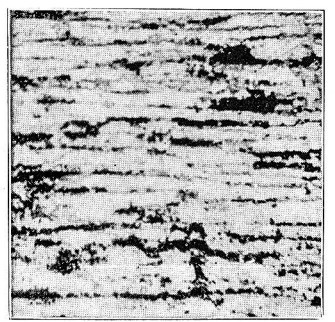


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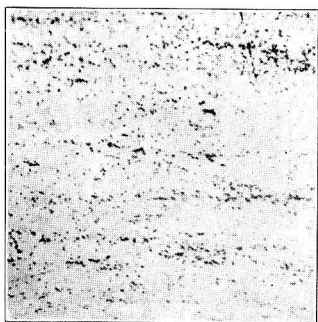


Fig. 45.



Fig. 46.

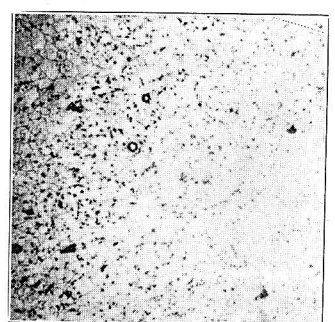


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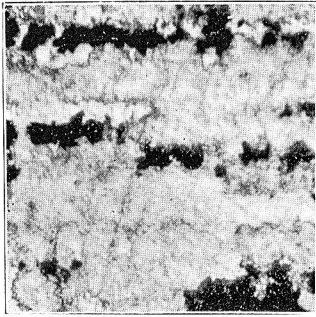


Fig. 48.



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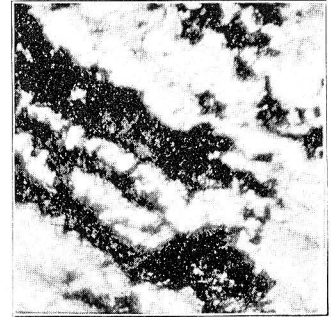


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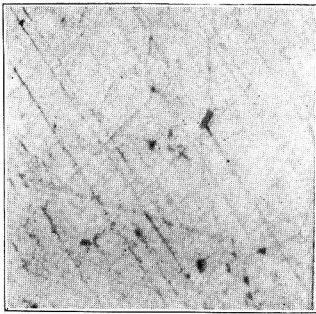


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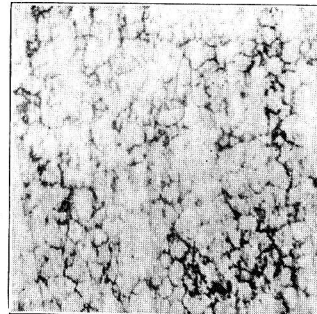


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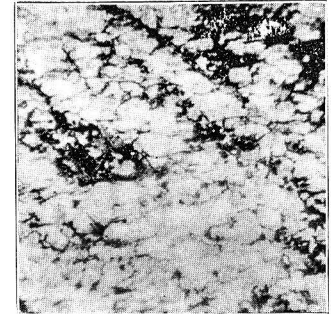


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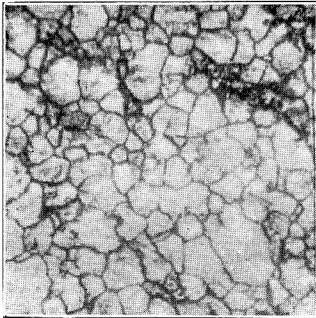


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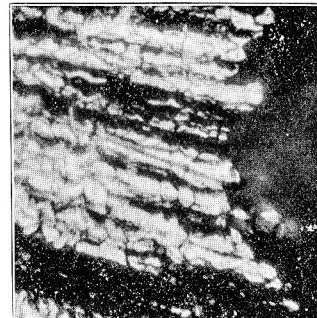


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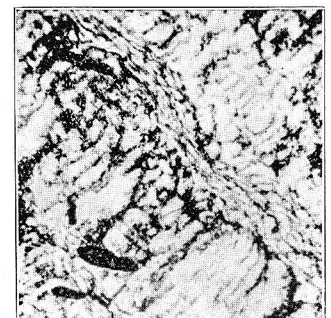


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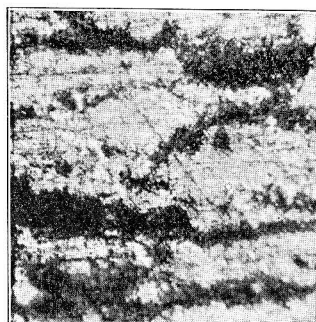


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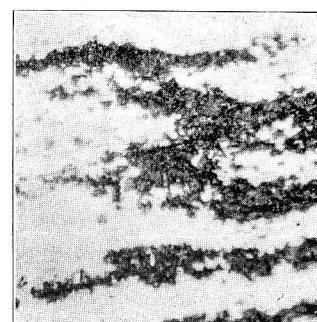


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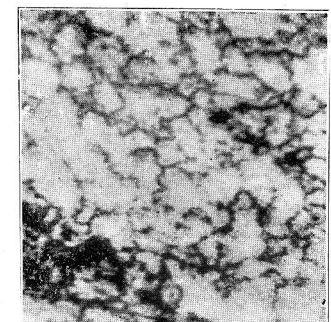


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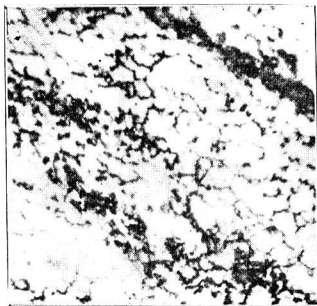


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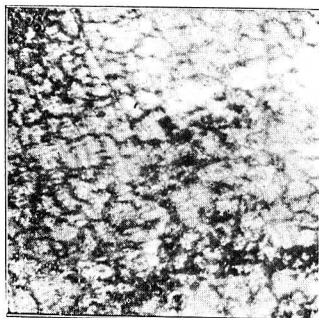


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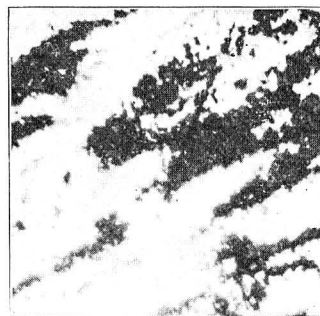


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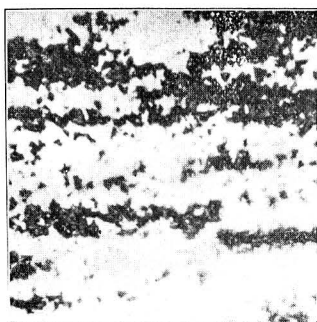


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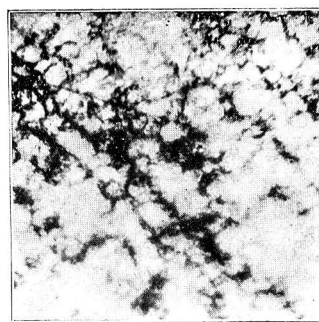


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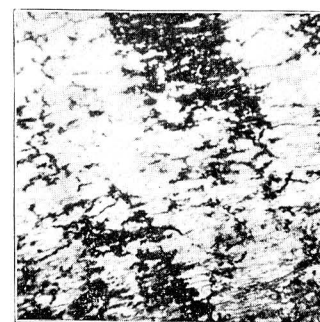


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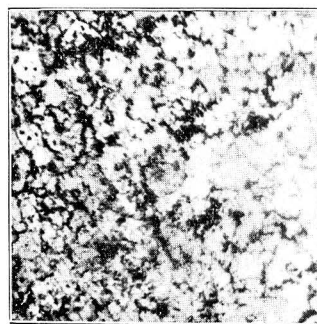


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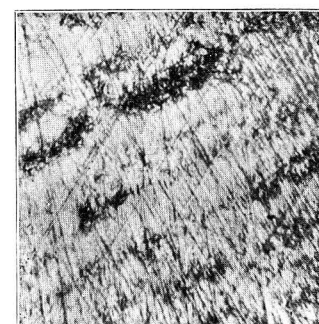


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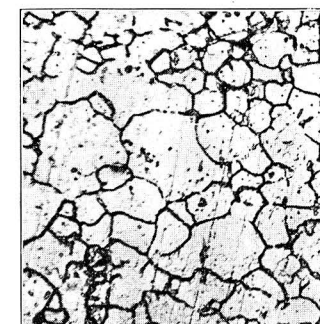


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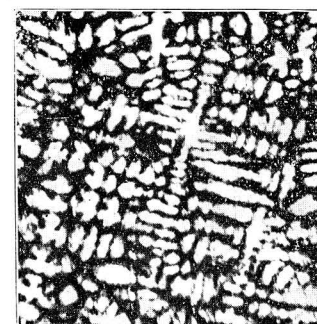


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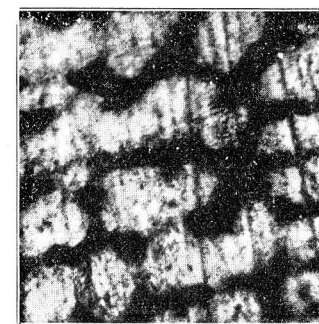


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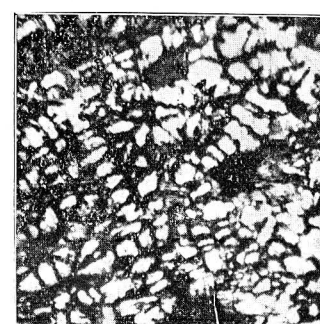


Fig. 72.



Fig. 73.



Fig. 74.

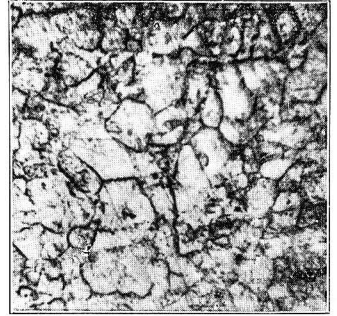


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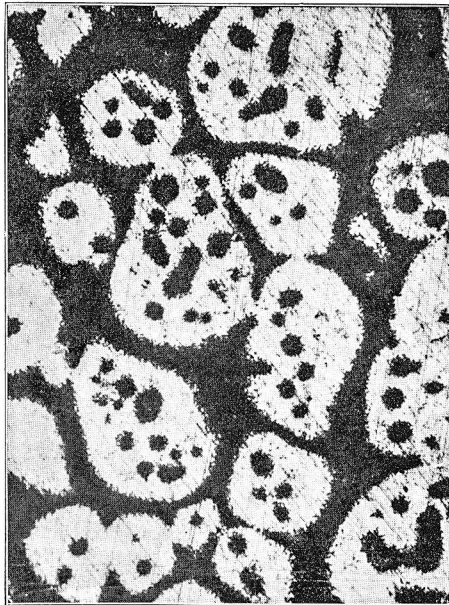


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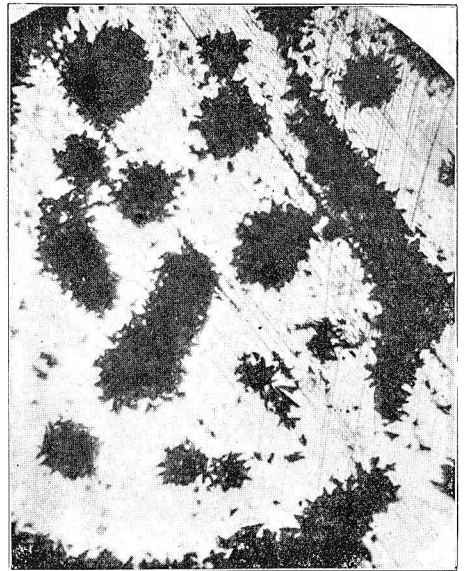


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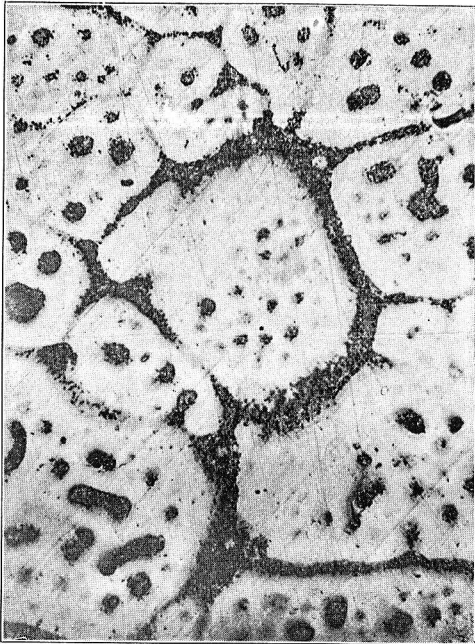


Fig. 81.

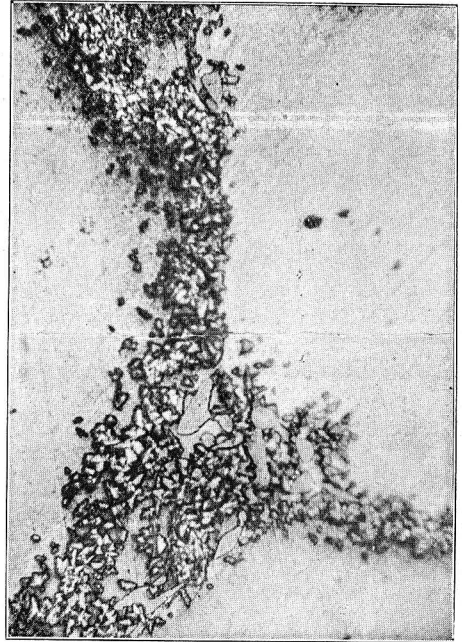


Fig. 82.

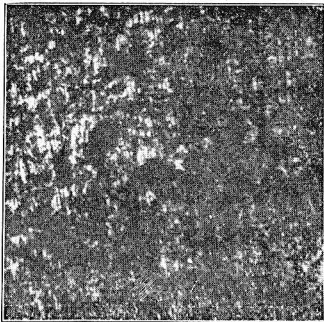


Fig. 83.

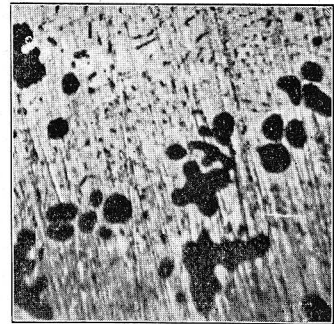


Fig. 84.