OTTAWA February 2, 1945.

REPORT

of the

ORE DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 1782.

Preliminary Report on Investigation into the Influence of Carbon on the Rates of Creep of Austenitic Iron-Nickel-Chromium Alloys of the 35% Nickel 15% Chromium Type at Temperatures above 1100° C. (2012° F.).

(Copy No. 6.)

Abstract

Seven heats of 35% Nickel 15% Chromium austenitic iron-nickel-chromium alloy, having carbon contents varying from 0.10 per cent to 0.60 per cent, have been prepared in a 50-pound high-frequency induction furnace. Room-temperature mechanical properties and resistance to creep at temperatures between 1100° and 1200° C. have been determined. Microstructures as cast and after creep tests also have been studied.

It is shown that up to about 0.35 per cent carbon the room-temperature ductility is not seriously impaired, but that above 0.5 per cent carbon this type of alloy can be expected to become fairly brittle. It is also shown that the rate of creep at temperatures between 1100° and 1200° C. may be expected to decrease as the carbon content is increased. The results are somewhat tentative as technical difficulties in these early tests made accurate temperature measurement difficult.

Bureau of Mines
Division of Metallic
Minerals
--
Physical Metallurgy
Research Laboratories

CANADA
DEPARTMENT
OF
MINES AND RESOURCES
Mines and Geology Branch
O T T A W A February 2, 1945.

R E P O R T

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Introduction.

The following investigation was conducted for the Dominion Magnesium Limited, of Haley, Ontario, the object being to determine the relative merits of various austenitic alloys of the iron-nickel-chromium type for service in the retort castings used in the production of magnesium by the ferrosilicon reduction process.

The immediate aim of the work reported herein is to show how variations in carbon content of alloys of the 35% nickel, 15% chromium type influence the room-temperature mechanical properties and the creep rate at temperatures over 1100° C. (2012° F.).

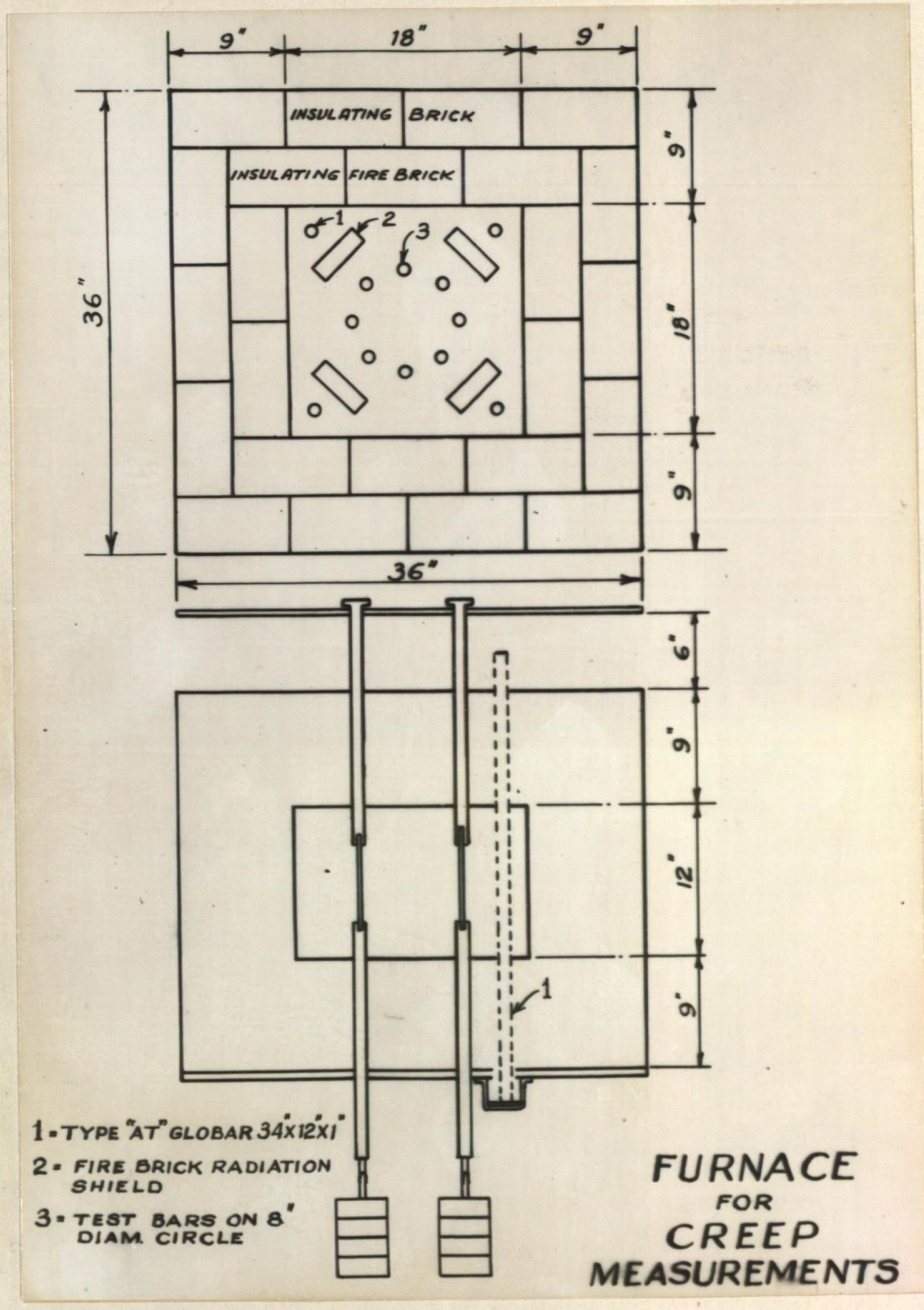
Description of Creep Testing Equipment:

The type of furnace used for the creep measurements is shown in Figure 1. This furnace was developed by the National Research Council, Ottawa, which carried out initial work in this field. This furnace is heated by four Type "AT" globars, 34 in. x 12 in. x 1 in. These bars are connected in series across 220 volts.

(Figure 1 comprises Page 2.)
(Text continues on Page 3.)

(Description of Creep Testing Equipment, cont'd) -

Figure 1.



-

(Description of Creep Testing Equipment, cont'd) -

It is a characteristic of globar heating elements that their resistance increases with use. It is also characteristic that in no two globars of the same type and size are the rate and amount of increase identical. In view of this, it is evident that there are certain inherent disadvantages to this type of heating. It is possible for the resistance of one of the globars to become greater than that of the others. This will cause the temperature in the neighbourhood of this globar to be higher than in the rest of the furnace. The resistance of the globars used was measured both before and after the two creep tests conducted. The values obtained are listed in Table I. The same globars were not used in both tests.

TABLE I. - Globar Resistance (in Ohms).

Globar Position		Before Test No. 1	After Test No. 1	Before Test No. 2	After Test No. 2
1	-	0.78	1.2	1.6	1.9
2	-	0.78	1.1	1.6	1.85
3	-	0.78	4.6	1.55	1.7
4	-	0.78	1.4	1.6	2.0

An anticipating thermocouple* was installed for the first test. A diagram of this thermocouple is shown in Figure 2.

Figure 2.

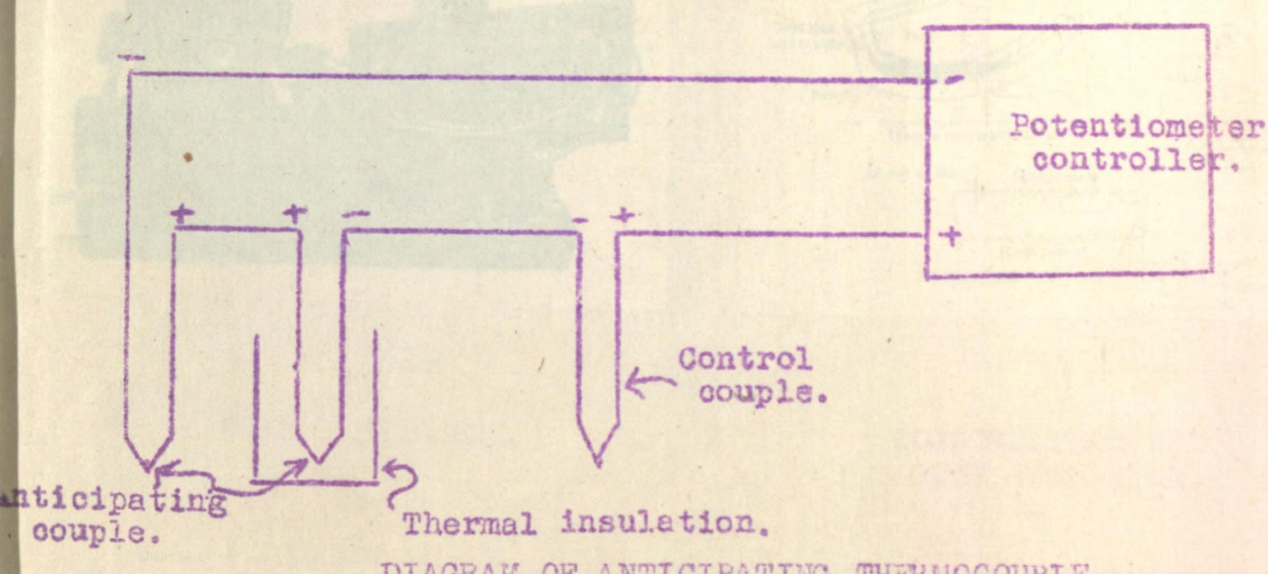


DIAGRAM OF ANTICIPATING THERMOCOUPLE.

* "Anticipating Thermocouple for Close Temperature Control" - by R. L. Longine, Product Engineering, Jan. 1944, p. 49.

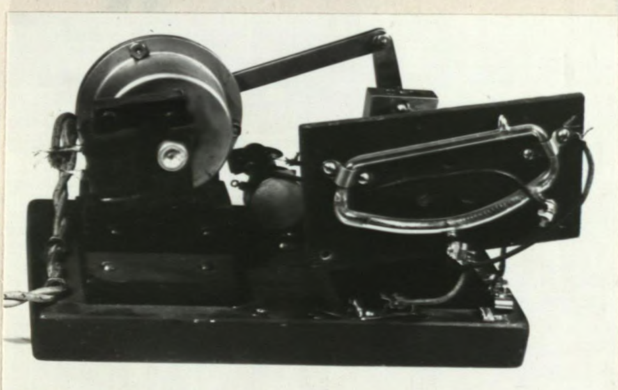
(Description of Creep Testing Equipment, cont'd) -

The control couple in the installation used was a Leeds & Northrup Ray-o-Tube (with micromax control). The anticipating couple consisted of two chromel-alumel thermocouples. One of these thermocouples was also intended to be used for temperature measurements.

This type of control arrangement gave control within $\pm 2\frac{1}{2}°$ C. of the mean temperature. It was used for the first creep test. However, at the temperature of operation the calibration of the chromel-alumel thermocouples shifted badly, and before the test was completed these thermocouples became useless. The exact temperature of the test was therefore not known.

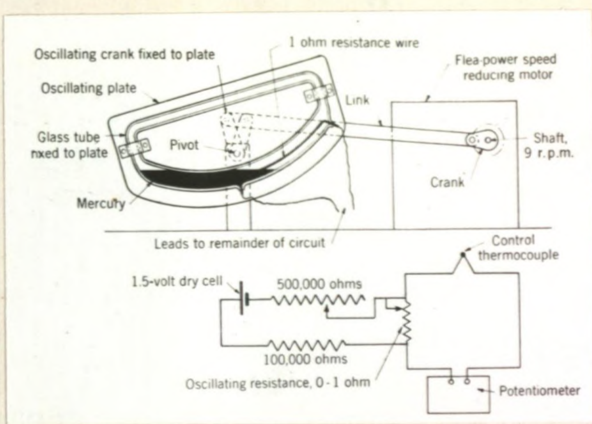
For the second test a device known as the "Gouy Modulator"* was installed. A photograph of the apparatus is shown in Figure 3, and a diagram showing the construction and electrical circuit is shown in Figure 4.

Figure 3.



GOUY MODULATOR.

Figure 4.



CONSTRUCTION OF GOUY MODULATOR.

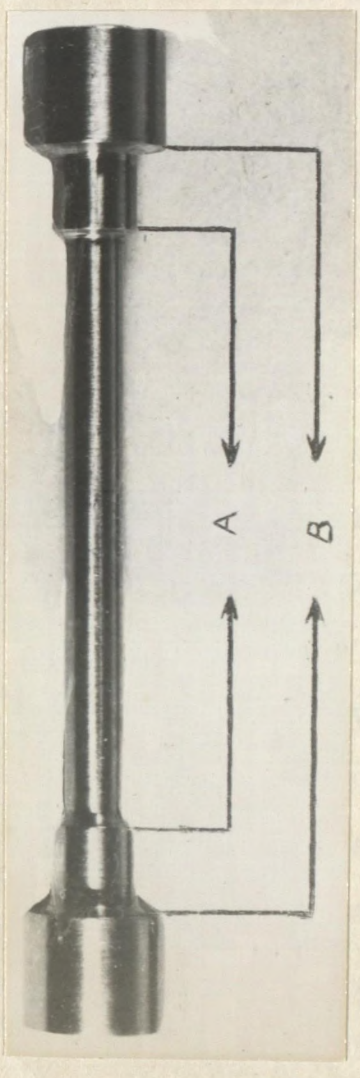
* "Temperature, Its Measurement and Control in Science and Industry" - Symposium, November, 1939, American Institute of Physics. Published by Reinhold Publishing Corp., pp. 613-614.

(Description of Creep Testing Equipment, cont'd) -

With this equipment the temperature was controlled within limits of $\pm 1\frac{1}{2}^{o}$ C. During this test a platinum, platinum-rhodium thermocouple was used to measure the temperature. However, this thermocouple became contaminated due to faulty protection and installation, and it was not possible to determine the exact temperature of this test.

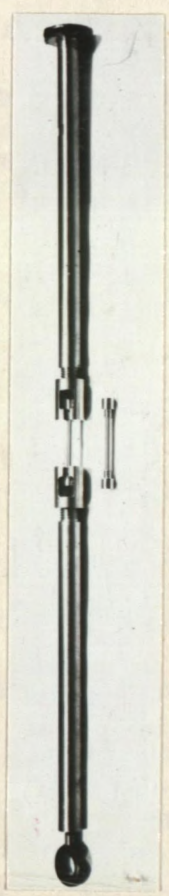
The test bars used to measure creep are shown in Figure 5. These bars have a nominal length of $2\frac{1}{2}$ inches (dimension A) and a diameter of 0.252 inch in the gauge length. Dimensions A and B are indicated in Figure 5. A shoulder type of grip was used. Figures 6, 7 and 8 show this grip in detail and assembled on the holder bars.

Figure 5.



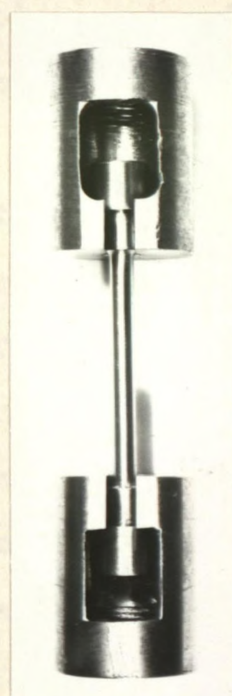
PHOTOGRAPH, ACTUAL SIZE, OF CREEP TEST BAR.

Figure 6.



PHOTOGRAPH, ABOUT 1/8 ACTUAL SIZE, SHOWING TEST BAR AND HOLDERS ASSEMBLED AS IN TESTING.

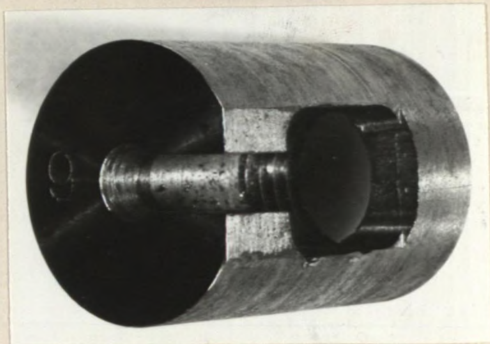
Figure 7.



PHOTOGRAPH, ABOUT $\frac{1}{2}$ ACTUAL SIZE, SHOWING HOW TEST BAR FITS IN HOLDERS.

(Description of Creep Testing Equipment, cont'd) -

Figure 8.



PHOTOGRAPH SHOWING DETAILS
OF TEST BAR HOLDER.

(Approximately $\frac{3}{4}$ size).

This type of holder was used in an attempt to facilitate the removal of the test bar after testing. However, it was found to be impossible to remove the test bar even from this holder. The grips must still be removed from the furnace and this necessitates taking the roof off the furnace after every test.

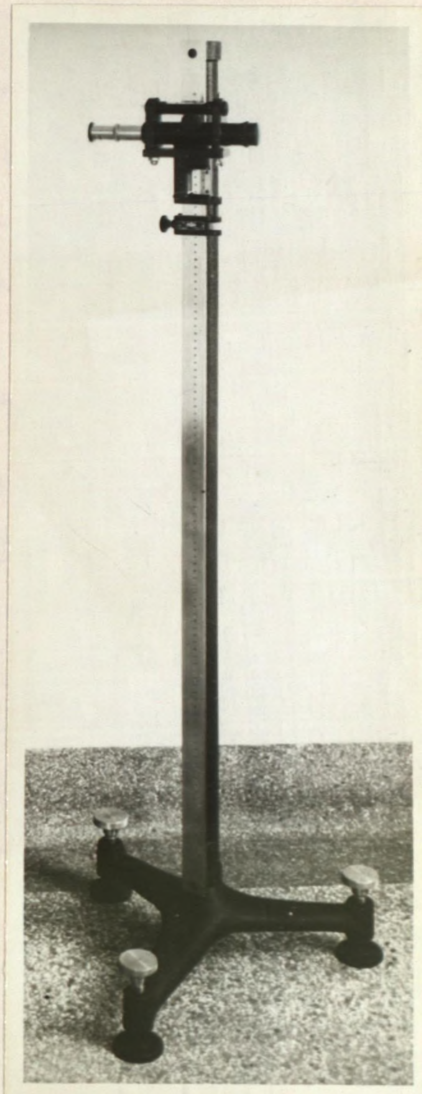
In the original tests conducted at the National Research Council, the test bar ends were threaded and fitted into holes drilled and tapped in the ends of the holder bars. It was impossible to remove the bars from these holders after a test, thereby necessitating the use of new holder bars for each test. In view of the difficulties encountered with the new type of grip used for the tests conducted here, it is considered advisable to return to the original method. Holder bars will be cast in our own foundry as required.

The creep is determined by measuring the distance between reference marks on the upper and lower holder bars above and below the furnace. These measurements are made with a cathetometer, which is shown in Figure 9.

(Continued on next page)

(Description of Creep Testing Equipment, cont'd) -

Figure 9.



CATHETOMETER.

(Approximately 1/10 actual size).

Casting of Test Bars:

Seven heats of alloy were prepared in a 50-pound-capacity high-frequency induction furnace. These alloys were melted without the addition of flux. Calcium silicon was used as a deoxidizing agent. Sixty grams of this was added to each heat. No aluminium was used.

The charges were made up of scrap metal of the

(Casting of Test Bars, cont'd) -

approximate analysis required; high-carbon and low-carbon ferrochromium, to adjust the carbon content; electrolytic nickel and nickel "F" shot, also used in the proper proportions to give the desired carbon and silicon; boiler plate scrap; and, where necessary, ferrosilicon and ferromanganese.

The analyses of the heats produced and their temperatures are given in Table II.

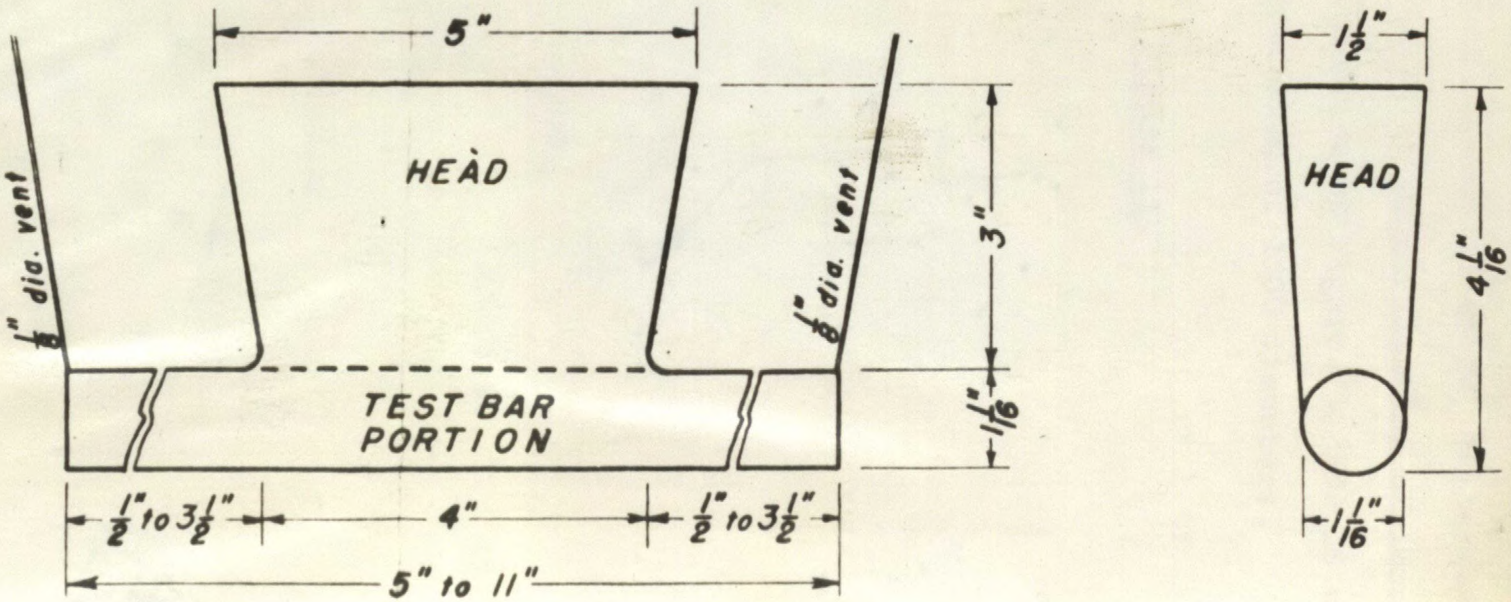
TABLE II. - Analysis and Casting Temperature of Alloys Produced.

Heat No.	Carbon, per cent	Silicon, per cent	Manganese, per cent	Chromium, per cent	Nickel, per cent	Temperature, in degrees Centigrade
10 -	0.10	2.18	1.08	17.89	37.58	1549
11 -	0.17	1.14	1.26	19.44	37.17	1599
12 -	0.30	1.16	1.08	18.40	37.54	1466
13 -	0.37	1.40	1.27	19.95	38.84	1610
14 -	0.54	1.51	1.27	21.42	38.55	1560
15 -	0.60	1.25	1.50	19.44	38.52	1532
19 -	0.20	1.51	0.67	18.49	39.23	1543

The design of the test bar used is shown in Figure 10. All test bar castings were radiographed before machining test bars from them, in order to be assured of their soundness.

(Figure 10 comprises Page 9.)
(Text continues on Page 10.)

Figure 10.



Pour through head. Cover molten head with powdered charcoal, fine coke dust, or sand immediately after pouring in order to keep head fluid as long as possible. Castings made after this design produce radiographically sound test bars provided the mold (especially the head) is completely filled.

ALLOY CASTING INSTITUTE

SUGGESTED STANDARD

TENSILE TEST BAR

GROSS WEIGHT UP TO $8\frac{1}{2}$ POUNDS

SCALE $\frac{1}{2}$" = 1" | AUGUST 6, 1941

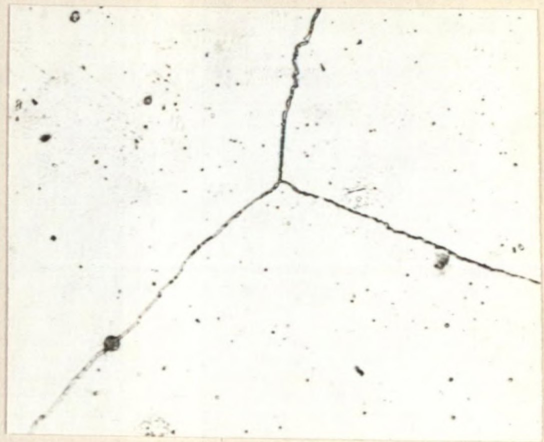
"As Cast" Microstructure:

The "as cast" microstructure of these alloys is shown in Figures 11 to 24, inclusive. These photomicrographs were taken at magnifications of 100 and 500 diameters, with the exception of Figures 14 and 16 which were taken at a magnification of 1500 diameters.

Figure 11.

X100.

Figure 12.



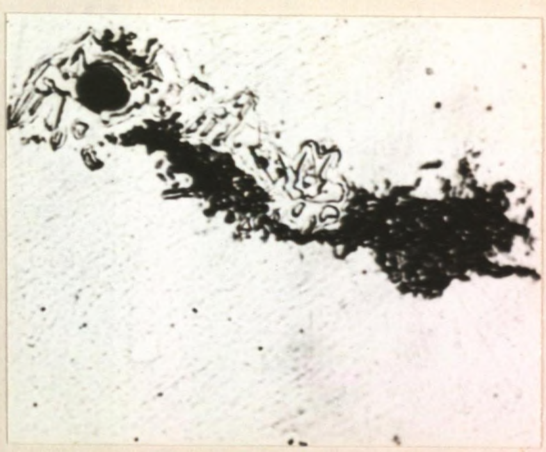
X500.

ALLOY NO. 10, AS CAST.

Figure 13.

X100.

Figure 14.

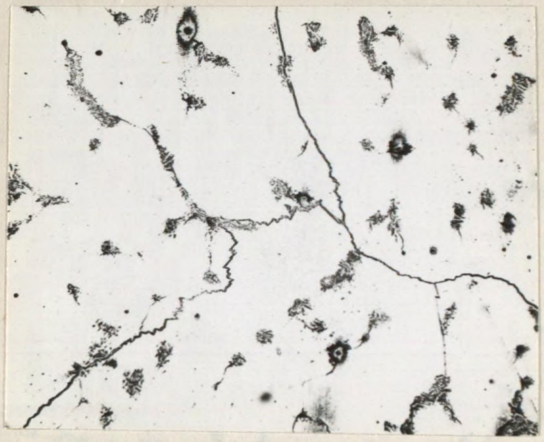


X1500.

ALLOY NO. 11, AS CAST.

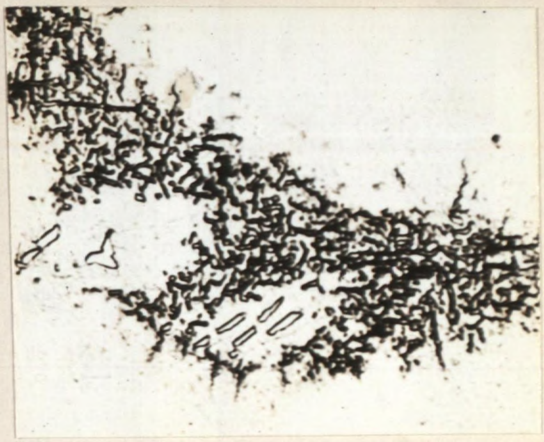
("As Cast" Microstructure, cont'd) -

Figure 15.



X100.

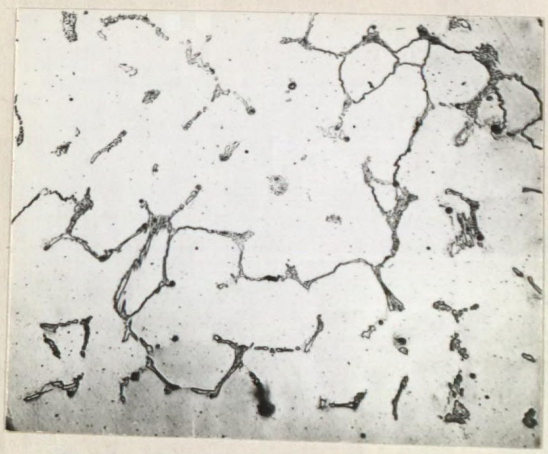
Figure 16.



X1500.

ALLOY NO. 19, AS CAST.

Figure 17.



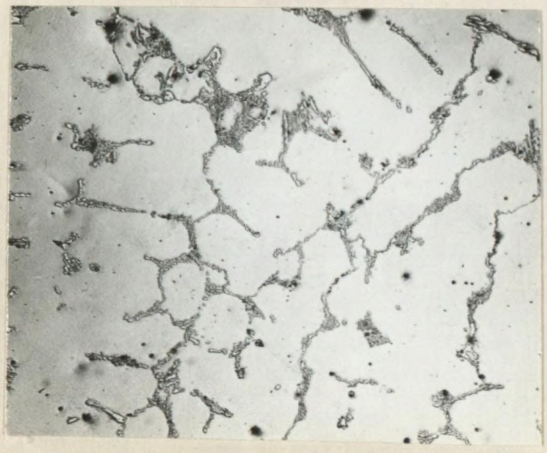
X100.

Figure 18.

X500.

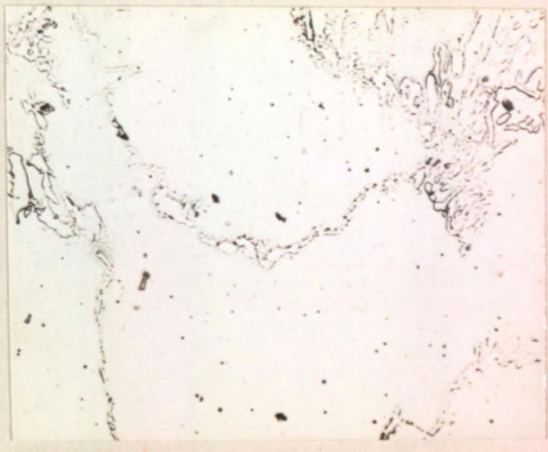
ALLOY NO. 12, AS CAST.

Figure 19.



X100.

Figure 20.

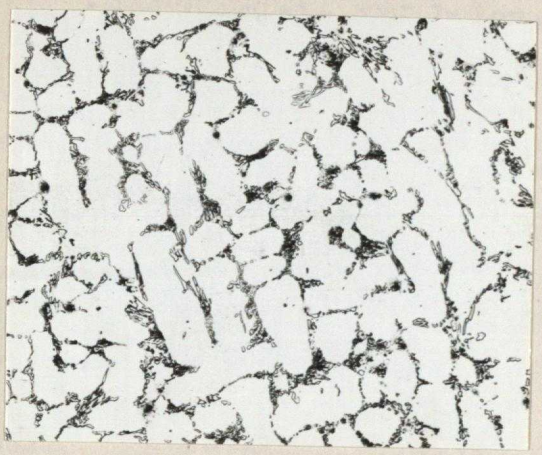


X500.

ALLOY NO. 13, AS CAST.

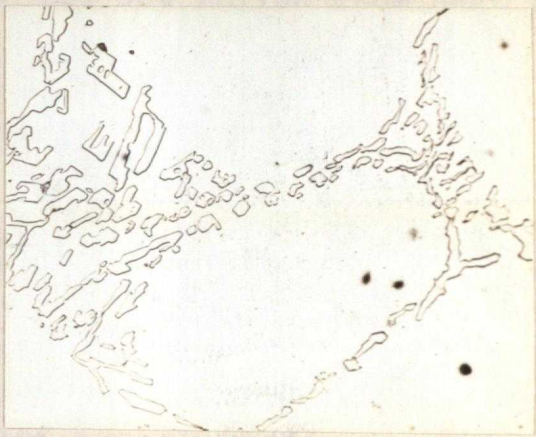
("As Cast" Microstructure, cont'd) -

Figure 21.



X100.

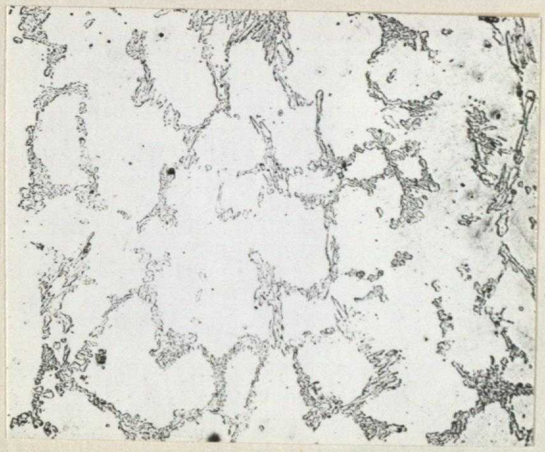
Figure 22.



X500.

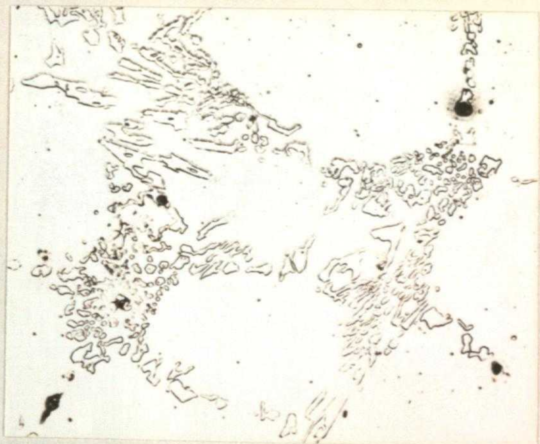
ALLOY NO. 14, AS CAST.

Figure 23.



X100.

Figure 24.



X500.

ALLOY NO. 15, AS CAST.

Room Temperature Mechanical Properties:

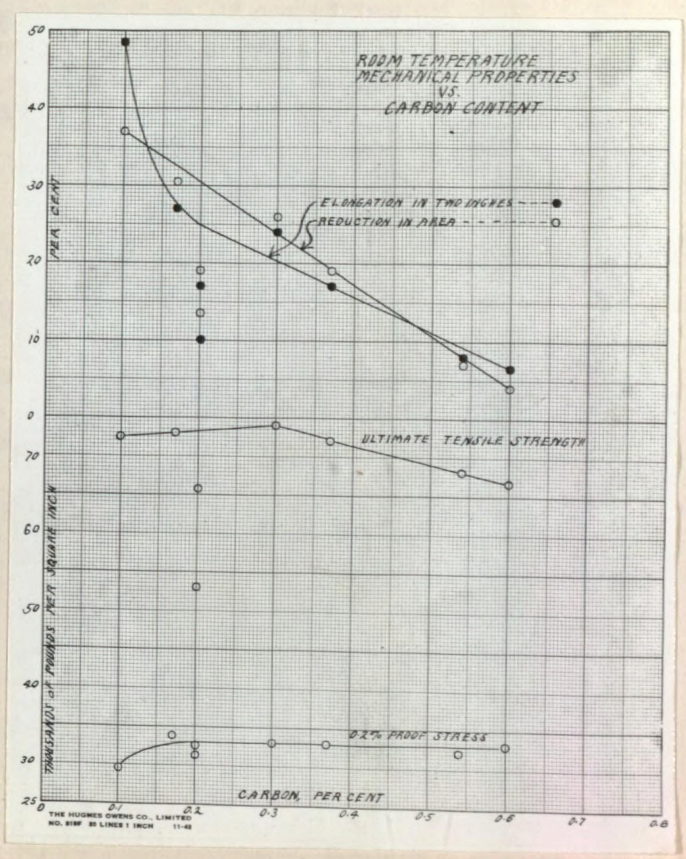
Standard tensile test bars, 0.505 inch in diameter and having a 2-inch gauge length, were prepared from each heat. These bars were pulled at room temperature. The results obtained are given in Table III, below. A second bar was pulled from Heat No. 19 since the results of the first bar seemed to be out of line.

TABLE III. - Room-Temperature Mechanical Properties.

Heat No.	Carbon, per cent	Ultimate tensile strength, p.s.i.	0.2 per cent proof stress, p.s.i.	Elongation in two inches, per cent	Reduction in area, per cent
10	- 0.10	72,500	29,500	48.5	37.0
11	- 0.17	73,000	33,800	27.0	30.5
19	- 0.20	53,000	31,200	10.0	13.5
		65,700	32,500	17.0	19.0
12	- 0.30	74,000	33,000	24.0	26.0
13	- 0.37	72,000	33,000	17.0	19.0
14	- 0.54	68,100	32,100	8.0	7.0
15	- 0.60	66,700	33,000	6.5	4.0

These data are shown graphically in Figure 25. It is apparent that, with the exception of Heat No. 19, there is evidence of a relationship between room-temperature ductility properties and carbon content.

Figure 25.



CREEP TEST NO. 1.

One test bar from each of the heats shown in Table II was tested under a stress of 500 pounds per square inch for a period of 500 hours. As previously pointed out, an accurate measurement of the temperature throughout this test was not obtained, but it is known that the temperature was over 1100° C. (2012° F.).

The type of test bar shown in Figure 5 was used. Measurements taken on these bars before and after test are shown in Table IV.

TABLE IV. - Test Bar Dimensions Before and After Test.

Test Bar No.	Diameter	DIMENSION "A"* Before	After	Difference	DIMENSION "B"* Before	After	Difference
	- I n			I n c h e s -			
10 -	0.252	2.531	2.828	0.297	3.298	3.571	0.273
11 -	0.252	2.523	2.580	0.057	3.286	3.338	0.052
12 -	0.253	2.566	2.692	0.126	3.330	3.457	0.127
13 -	0.251	2.459	2.558	0.099	3.246	3.342	0.096
14 -	0.251	2.487	2.545	0.058	3.240	3.308	0.068
15 -	0.252	2.478	2.510	0.032	3.245	3.282	0.037
19 -	0.252	2.494	2.716	0.222	3.261	3.493	0.273

* See Figure 5 for dimensions.

Since the increases in "A" and "B" are about the same, it is assumed that all elongation took place in the reduced portion of the bar and that the differences in these two increments are merely due to error in measuring. These measurements were taken with inside callipers. These two increments have, therefore, been averaged and the result is shown as a per cent elongation of the original dimension "A" in Table V.

(Continued on next page)

(Creep Test No. 1, cont'd) -

TABLE V. - Total Elongation, Per Cent Elongation and Carbon Content.

Test Bar No.		Carbon Content, per cent	Dimension "A" before test, inches	Average total elongation, inches	Total elongation, per cent
10	-	0.10	2.531	0.285	11.25
11	-	0.17	2.523	0.055	2.18
19	-	0.20	2.494	0.227	9.13
12	-	0.30	2.566	0.127	4.90
13	-	0.37	2.495	0.098	3.98
14	-	0.54	2.487	0.063	2.53
15	-	0.60	2.478	0.035	1.41

The measurements of creep taken during the test are shown graphically in Figures 26 and 27. They are plotted as percentage of elongation against time.

The rate of creep, in per cent elongation per hour, was determined from the slope of the straight portion of the curves in Figures 26 and 27. These are tabulated against carbon content in Table VI:

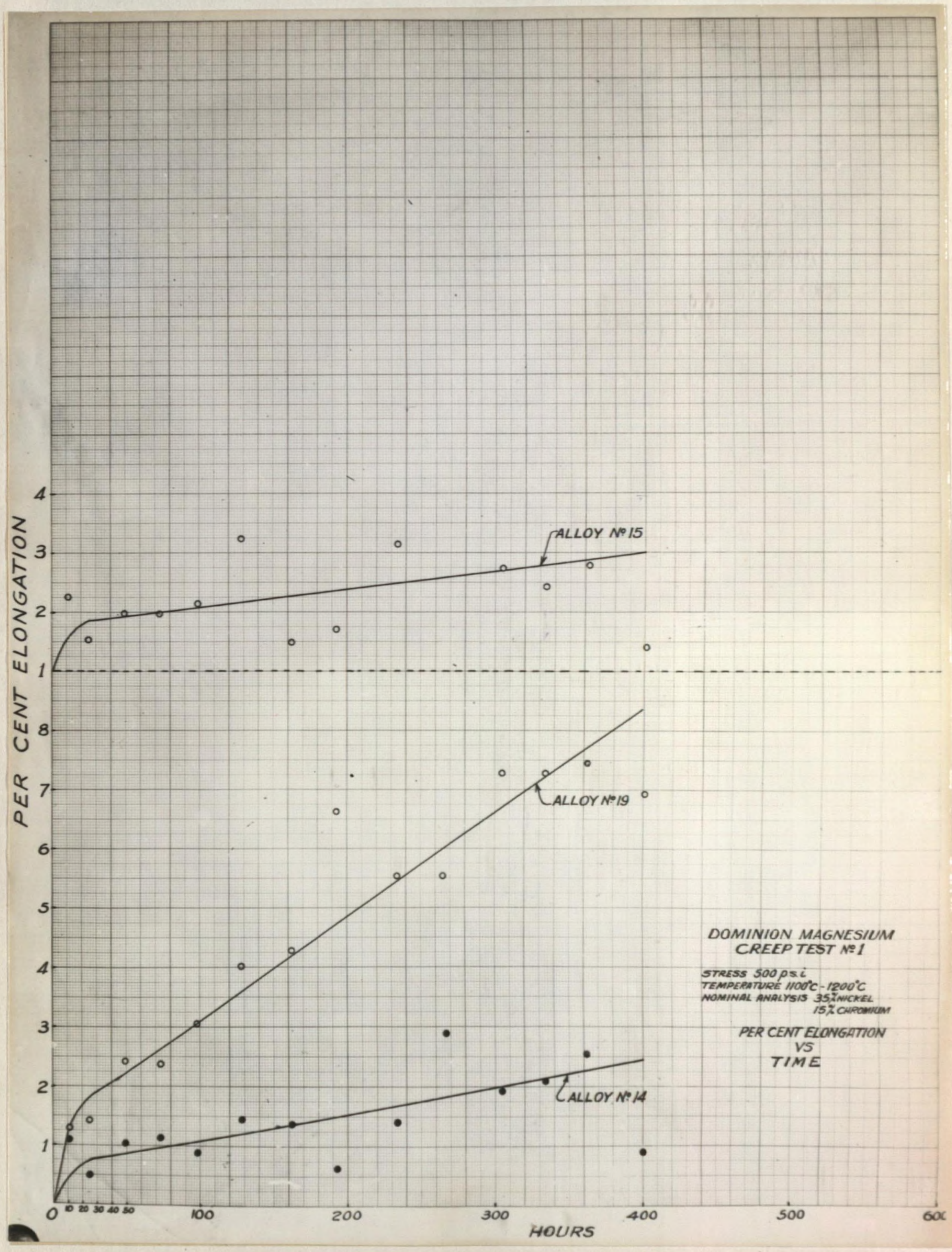
TABLE VI. - Rate of Creep vs. Carbon Content.

Test Bar No.		Carbon Content, per cent	Rate of Creep, per cent per hour
10	-	0.10	0.0255
11	-	0.17	0.0038
19	-	0.20	0.0168
12	-	0.30	0.0123
13	-	0.37	0.0080
14	-	0.54	0.0046
15	-	0.60	0.0030

(Figures 26 and 27 follow,)
(on Pages 16 and 17.)
(Text continues on Page 18.)

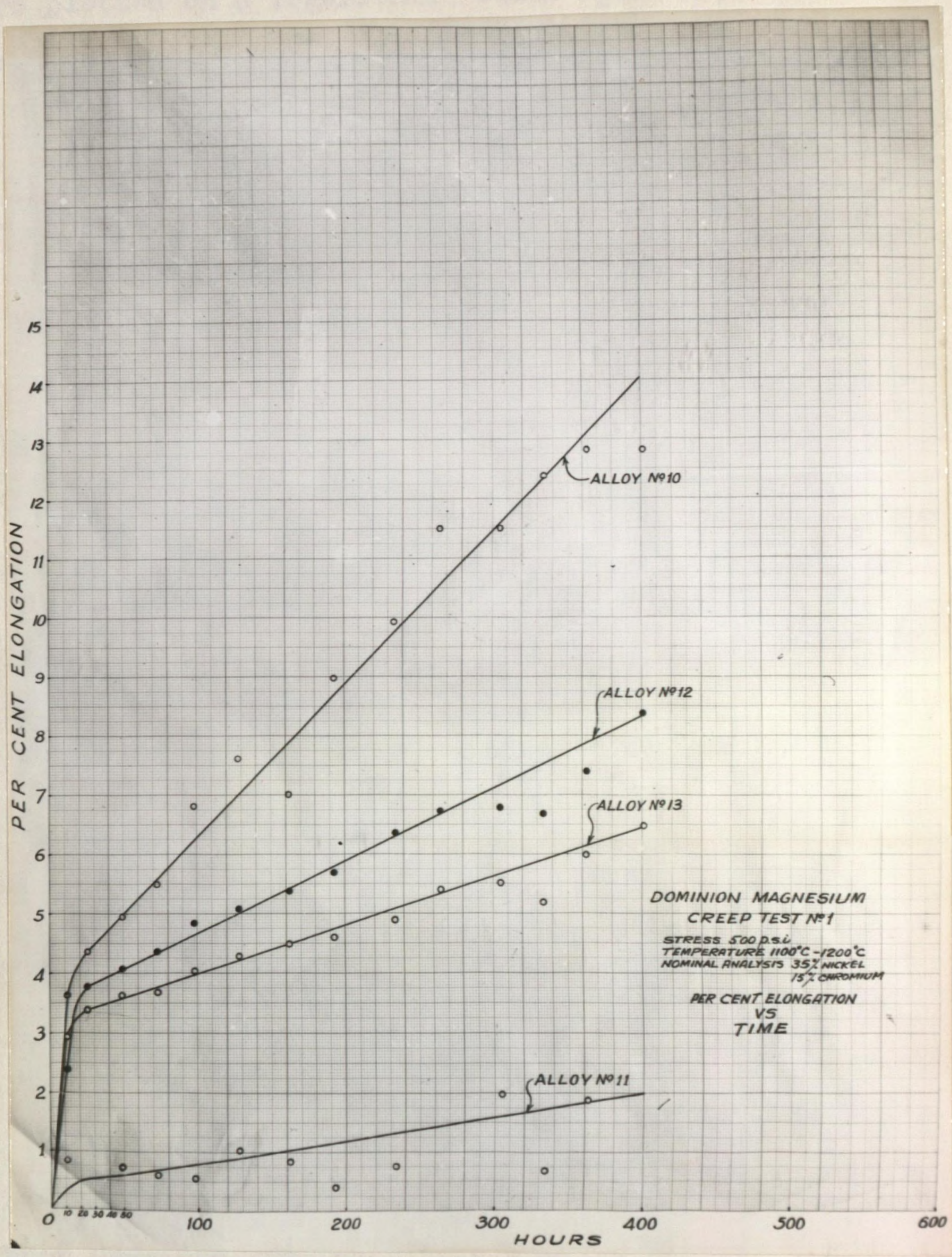
(Creep Test No. 1, cont'd) -

Figure 26.



(Creep Test No. 1, cont'd) -

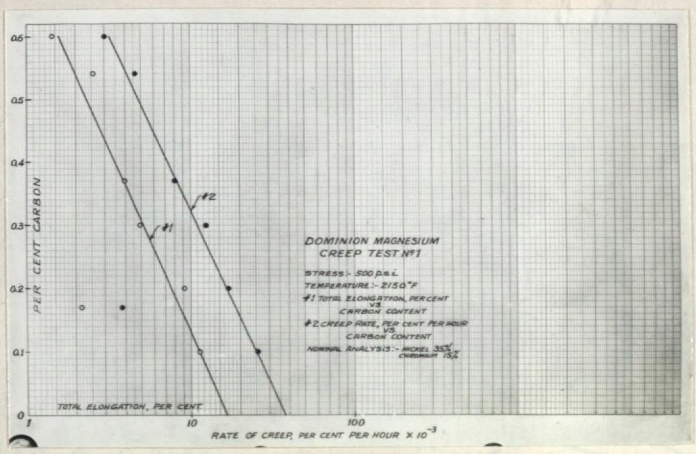
Figure 27.



(Creep Test No. 1, cont'd) -

The data given in Tables V and VI are shown graphically in Figure 28 in which rates of creep and total elongation are plotted on a logarithmic scale against per cent carbon which is plotted on an arithmetic scale.

Figure 28.



At the end of 500 hours the test was terminated. The power was shut off and the furnace allowed to cool down. When cool the test bars were removed and measured. Specimens were then cut out of the gauge length for metallographic examination. Samples were also taken from the gauge length for chemical analysis, to check on the total carbon loss. The results of this analysis, given in Table VII, show no very great loss of carbon.

(Continued on next page)

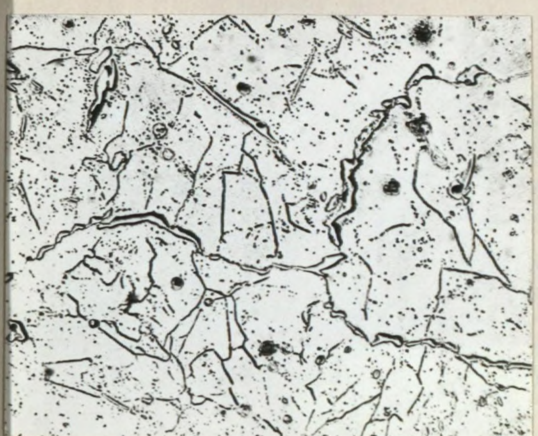
(Creep Test No. 1, cont'd) -

TABLE VII. - Carbon Content Before and After Test.

Test Bar No.		Carbon, per cent before test	Carbon, per cent after test
10	-	0.10	0.07
11	-	0.17	0.19
19	-	0.20	0.16
12	-	0.30	0.29
13	-	0.37	0.33
14	-	0.54	0.50
15	-	0.60	0.59

The microstructures obtained on the specimens cut from the bars after the test are shown in Figures 29 to 42, at magnifications of 100 and 500 diameters.

Figure 29.



X100.

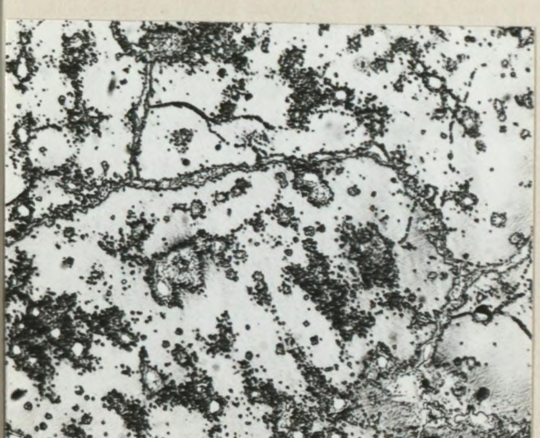
Figure 30.

X500.

TEST BAR NO. 10, STRUCTURE AFTER CREEP TEST.

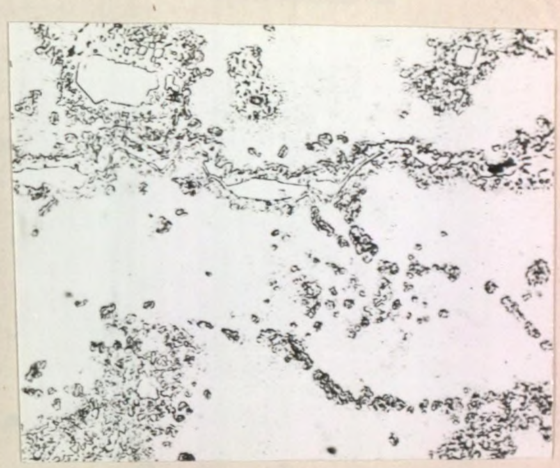
-

Figure 31.



X100.

Figure 32.



X500.

TEST BAR NO. 11, STRUCTURE AFTER CREEP TEST.

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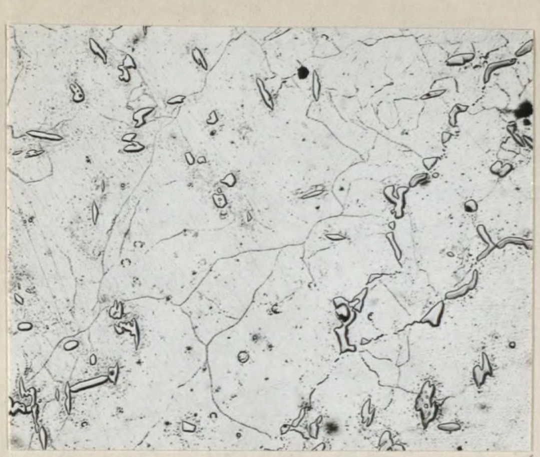
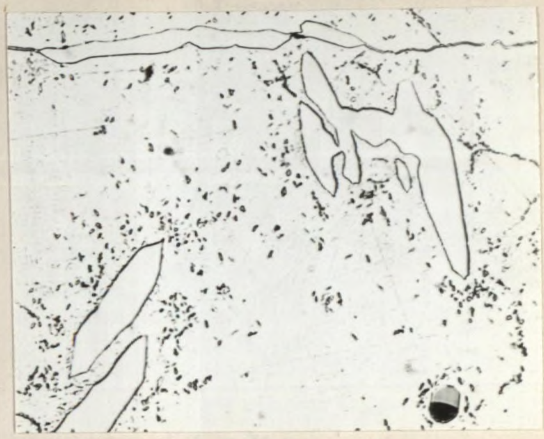
(Creep Test No. 1, cont'd) -

Figure 33.



X100.

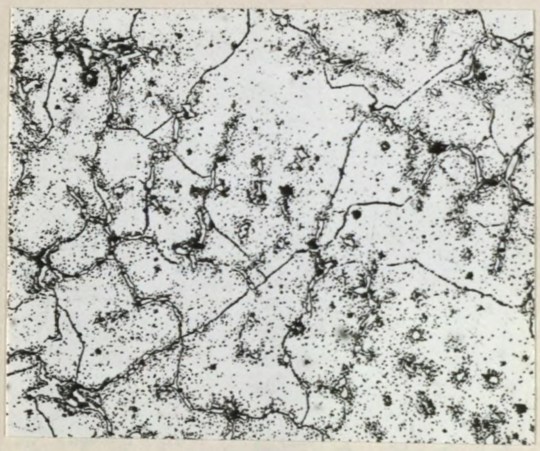
Figure 34.



X500.

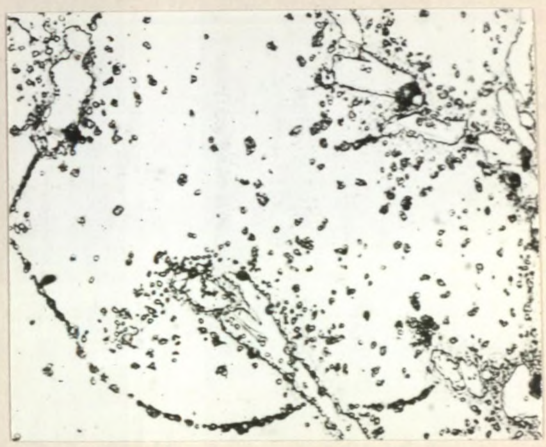
TEST BAR NO. 19, STRUCTURE
AFTER CREEP TEST.

Figure 35.



X100.

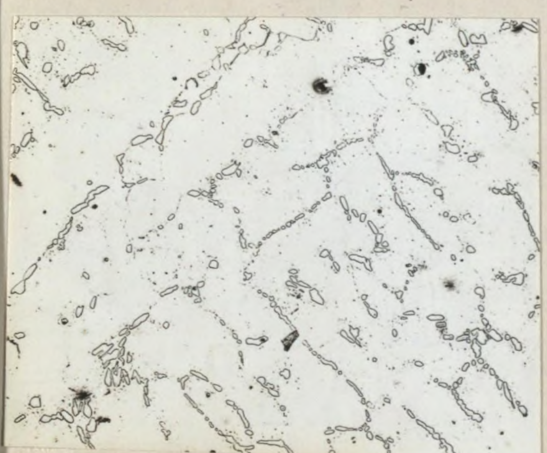
Figure 36.



X500.

TEST BAR NO. 12, STRUCTURE
AFTER CREEP TEST.

Figure 37.



X100.

Figure 38.

X500.

TEST BAR NO. 13, STRUCTURE
AFTER CREEP TEST.

(Creep Test No. 1, cont'd) -

Figure 39.

X100.

Figure 40.

X500.

TEST BAR NO. 14, STRUCTURE
AFTER CREEP TEST.

-

Figure 41.

X100.

Figure 42.

X500.

TEST BAR NO. 15, STRUCTURE
AFTER CREEP TEST.

-

These structures all show two types of carbide constituent. There are the large, massive carbides and very small carbides such as might be formed by a precipitation treatment. It is possible that these fine carbide particles did precipitate while the test bars were cooling in the furnace. To determine whether or not this was the case, additional specimens were cut from the test bars used in

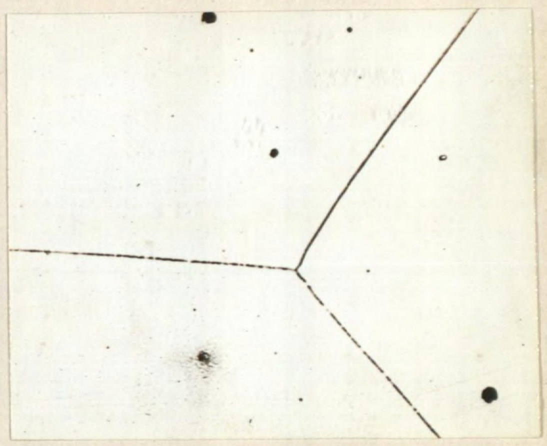
(Creep Test No. 1, cont'd) -

this test. These were heated to 1180° C. (2156° F.) for a period of 24 hours and then water-quenched. The resultant structures are shown in Figures 43 to 52, which are photo-micrographs taken at magnifications of 100 diameters and 500 diameters.

Figure 43.

X100.

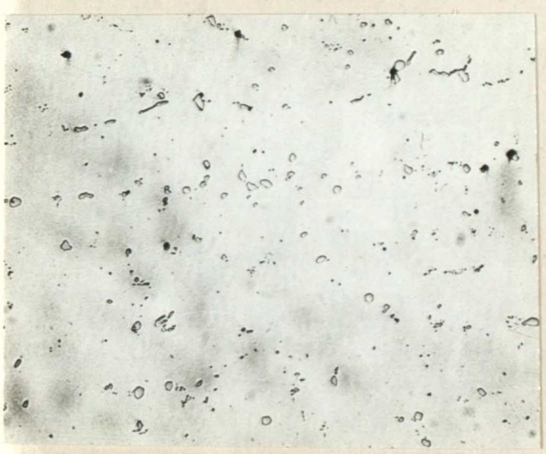
Figure 44.



X500.

TEST BAR NO. 10, WATER-QUENCHED FROM 1180° C. AFTER CREEP TEST.

Figure 45.



X100.

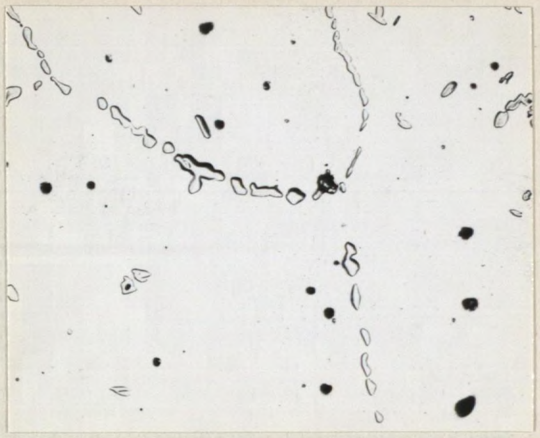
Figure 46.

X500.

TEST BAR NO. 11, WATER-QUENCHED FROM 1180° C. AFTER CREEP TEST.

(Creep Test No. 1, cont'd) -

Figure 47.



X100.

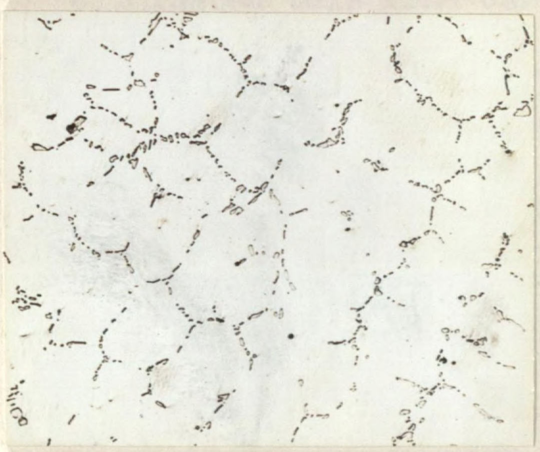
Figure 48.

X500.

TEST BAR NO. 19, WATER-QUENCHED
FROM 1180° C. AFTER CREEP TEST.

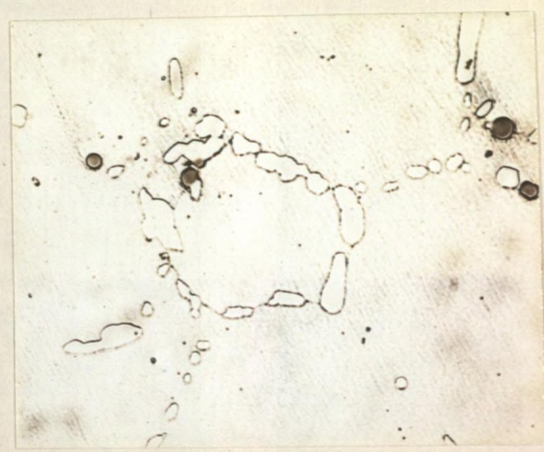
-

Figure 49.



X100.

Figure 50.



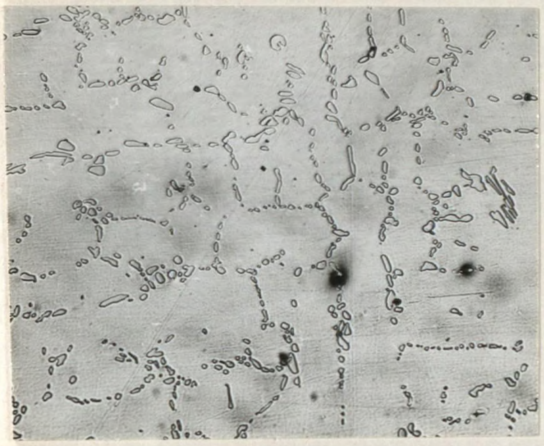
X500.

TEST BAR NO. 12, WATER-QUENCHED
FROM 1180° C. AFTER CREEP TEST.

-

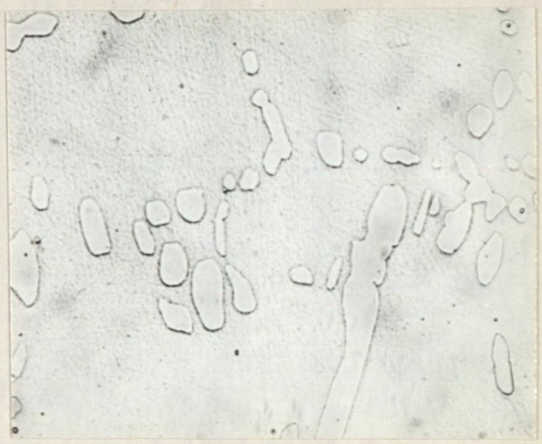
(Creep Test No. 1, cont'd) -

Figure 51.



X100.

Figure 52.



X500.

TEST BAR NO. 14, WATER-QUENCHED
FROM 1180° C. AFTER CREEP TEST.

It will be noted that this heat treatment has eliminated this fine carbide constituent. It is therefore reasonable to assume that these fine carbides were precipitated while the test bars were cooling in the furnace.

CREEP TEST NO. 2.

Creep Test No. 1 was duplicated. The same type of bar was used. The Gouy modulator was used to give finer temperature control, and a platinum, platinum-rhodium thermocouple was welded onto one of the bars for temperature measurements. Owing to contamination of this couple, the temperature varied considerably during the test and it was impossible to determine the exact temperature of test. At 239 hours it was obvious that the temperature had become higher than desired, and it was therefore reduced by some 80° C.

The percentage elongation as determined from the cathetometer readings is plotted against time in Figures 53 and 54. It will be noted that there are two sharp changes of slope common to all curves. These occur at 89 hours and 239 hours. At the latter time the temperature was purposely changed. It is assumed that another sharp temperature change occurred at 89 hours. Accordingly, this test is divided into three periods and rates of creep have been calculated from Figures 53 and 54 for each of these periods. These rates are recorded in Table VIII.

TABLE VIII. - Rates of Creep vs. Carbon Content, Creep Test No. 2.

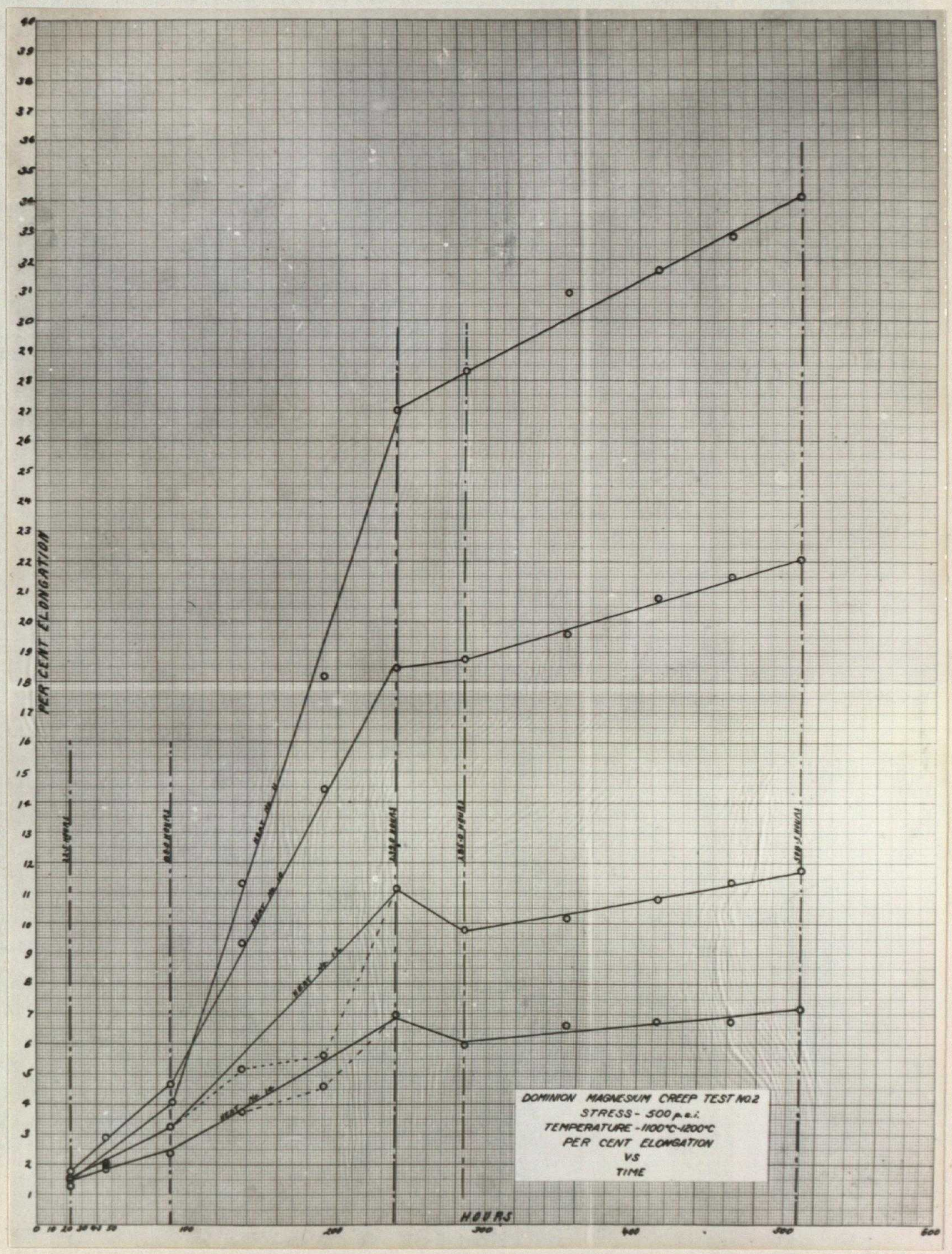
Heat No.		Carbon, per cent	PERIODS OF CONSTANT CREEP RATE		
			22.5 hours to 89.0 hours	89.0 hours to 239.0 hours	285.0 hours to 508.3 hours
10	-	0.10	0.0441	0.0919	0.0148
11	-	0.17	0.0386	0.1530	0.0259
19	-	0.20	0.0458	0.1104	0.0195
12	-	0.30	0.0256	0.0526	0.0089
13	-	0.37	0.0106	0.0366	0.0096
14	-	0.54	0.0158	0.0290	0.0049
15	-	0.60	0.0182	0.0326	0.0055

These values are plotted on a semi-logarithmic chart, in Figure 55.

(Figures 53, 54 and 55 follow,)
(on Pages 26, 27 and 28.)
(Text continues on Page 28.)

(Creep Test No. 2, cont'd) -

Figure 53.



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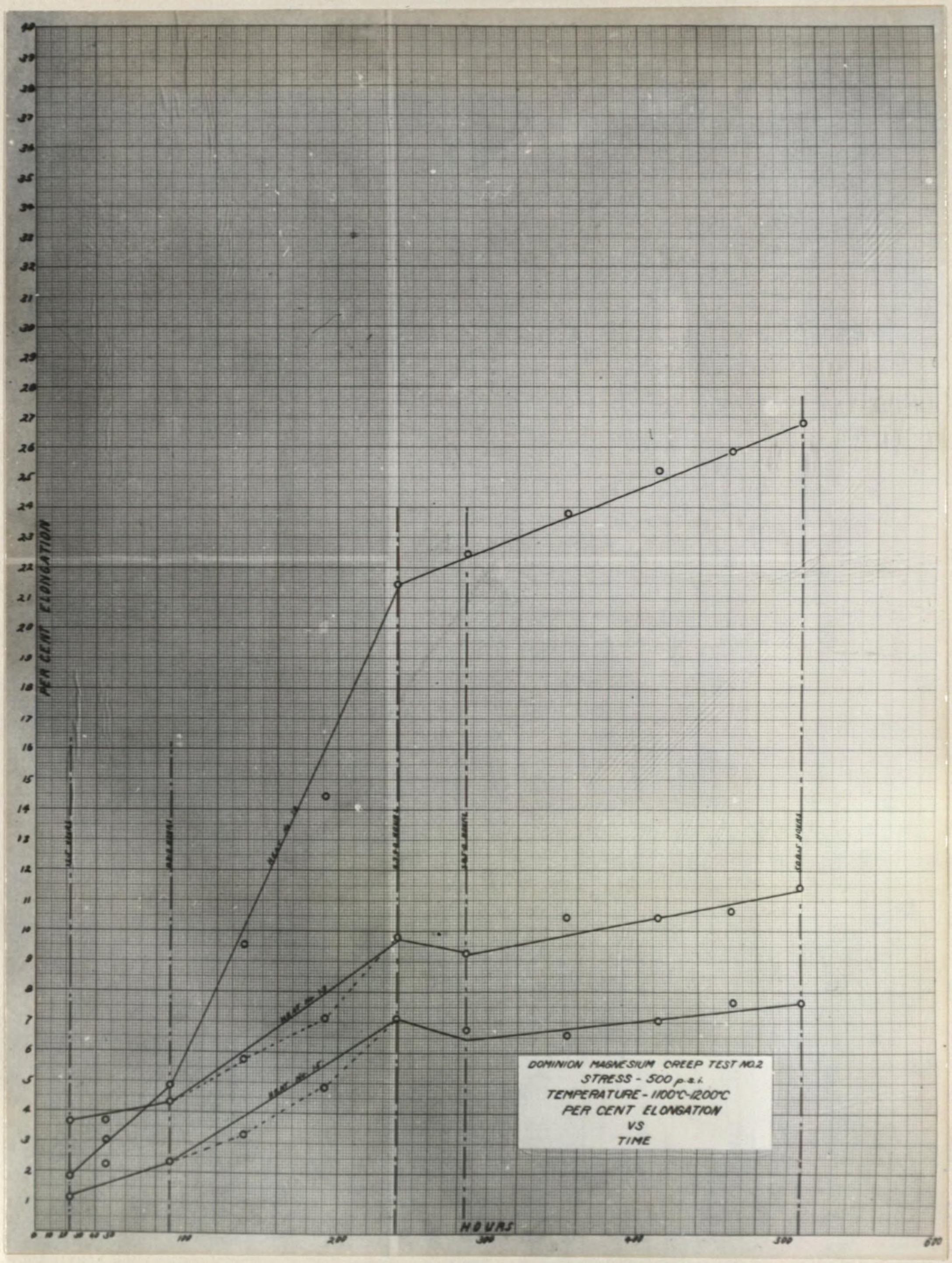
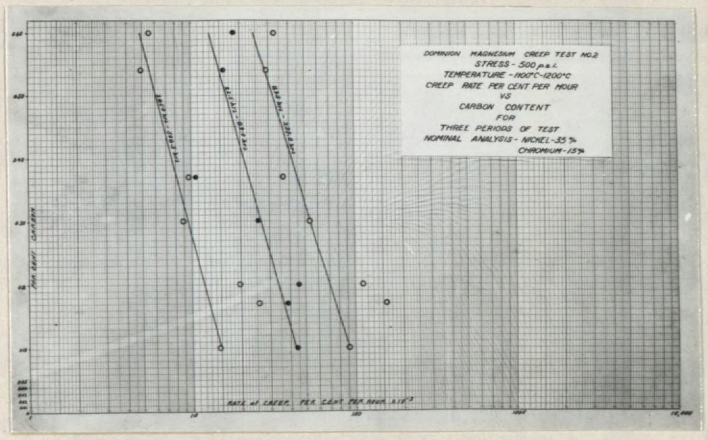
(Creep Test No. 2, cont'd)

Figure 54.



(Creep Test No. 2, cont'd) -

Figure 55.



Rate of Creep vs. Carbon Content.

DISCUSSION OF RESULTS:

(a) Remarks on Furnace Design -

It has been pointed out that inequalities of temperature are to be expected, owing to the characteristic behaviour of globar elements. These inequalities can be minimized to a certain extent by matching globars according to resistance, before every test. However, to obtain a high refinement of temperature uniformity, each globar should be supplied from an individual variable tap auto transformer so that an even distribution of power input can be maintained. The necessity of doing this can better be judged after a number of tests have been conducted using thermocouples on each test bar.

(b) Room-Temperature Mechanical Properties -

The room-temperature mechanical properties of Heat No. 19 seem to be out of line with the rest of the heats. The chemical analysis has been checked and found to be correct. It is possible that the difference in microstructure, as shown

(Discussion of Results, cont'd) -

in Figures 13, 14, 15 and 16, may be the reason for this.

Apparently, variations in carbon content have little effect on the ultimate tensile strength and 0.2 per cent proof stress of this type of alloy but do have a marked effect on the elongation and the reduction in area. From Figure 25 it would appear that a carbon content above 0.4 per cent might result in a casting not quite as tough as is desirable.

(c) Creep Tests -

The results of Creep Test No. 1 are very satisfactory, with the exception of Test Bar No. 11. The only possible explanation of the analogous behaviour of this test bar is that its temperature was lower than that of the other bars. This is partially substantiated by the microstructure as revealed by Figures 31 and 32.

Creep Test No. 2 was, on the whole, very unsatisfactory, owing to technical difficulties arising in temperature control and measurement. An attempt has been made to place some logical interpretation on the results. It is evident that they do, in a very rough qualitative manner, confirm the trend shown by Creep Test No. 1, but the lack of uniformity in them leaves much to be desired.

However, in spite of these irregularities, it is felt that the results from both tests do show that carbon content does influence the rate of creep of the 35% Nickel 15% Chromium type of austenitic alloy at temperatures above 1100° C. Higher carbon contents apparently produce lower rates of creep.

GENERAL CONCLUSIONS AND RECOMMENDATIONS:

It is indicated, from the work to date, that retorts cast from a 35% Nickel 15% Chromium type of alloy having a relatively high carbon content should be expected to deform less rapidly than retorts cast in this alloy having a low carbon content. This is more or less in line with the trend shown in Figure 3 of Report of Investigation No. 1415, dated May 26, 1943, which was a survey of retort records up to May 12, 1943.

From the point of view of welding and repairing these retorts, it is not desirable to have the metal too brittle. Brittle metal could result in cracks from thermal shock in welding or mechanical shock of chipping before welding.

For this reason, therefore, it is recommended that a carbon content of 0.30 per cent to 0.40 per cent, with a maximum of 0.45 per cent, should be expected to result in retort castings that would have improved performance.

FUTURE PLANS:

The design of the test bar has been altered. The critical dimensions of the gauge length and diameter have been maintained but threaded grips will be used in future.

Improvements will be made in temperature measuring technique.

It is intended to conduct a third test, using the same series of bars previously used. Following this, a program has been prepared to determine the influence of chromium and nickel variations over the range of 10 per cent to 30 per cent chromium and 10 per cent to 40 per cent nickel. This will reveal which of the two main types of alloys now being used for these retorts is the better. The number of alloys required to cover this range has been reduced to 28.

A short program has also been initiated to study

(Future Plans, cont'd) -

quantitatively the effect of carbon content between the ranges of 0.1 per cent to 0.6 per cent on the susceptibility of the 35% Nickel 15% Chromium type of alloy to cracking from thermal shocks due to welding.

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