OTTAWA August 9, 1945.

REPORT

of the

ORE DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 1916.

(Further to Report of
(Investigation No. 1782,
(dated February 2, 1945.)

Investigation into the Influence of Nickel and Chromium on the Rates of Creep of Austenitic Iron-Nickel-Chromium Alloys at Temperatures above 1100° C. (2012° F.).

(Copy No. 10.)

Bureau of Mines
Division of Metallic
Minerals.

Physical Metallurgy
Research Laboratories

CANADA

DEPARTMENT
OF
MINES AND RESOURCES

Mines and Geology Branch

OTTAWA August 9, 1945.

REPORT

of the

ORE DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 1916.

(Further to Report of
(Investigation No. 1782,)
(dated February 2, 1945.)

Investigation into the Influence of Nickel and Chromium on the Rates of Creep of Austenitic Iron-Nickel-Chromium Alloys at Temperatures above 1100° C. (2012° F.).

Introduction:

This project was initiated at the request of Dominion Magnesium Limited, of Haley, Ontario, to determine the relative merits of various austenitic alloys of the iron-nickel-chromium type for service in retort castings used in the production of magnesium by the ferro-silicon reduction process.

The immediate aim of the work reported herein is to show how variations in nickel and chromium content influence room-temperature mechanical properties and creep rates at temperatures over 1100° C.

Description of Testing Equipment:

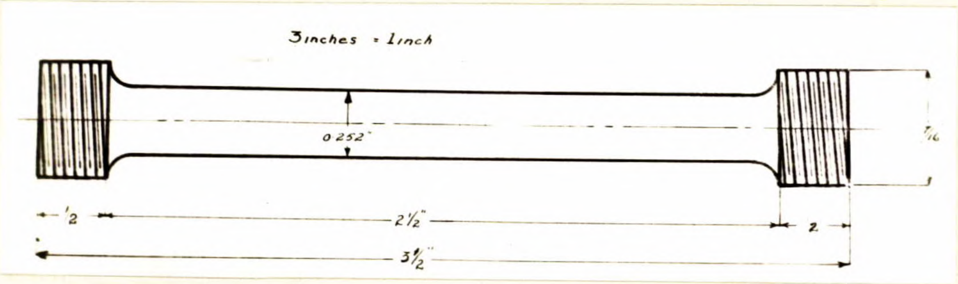
The equipment in use was fully described in Report of Investigation No. 1782. Two furnaces are now in use.

The limitation of the equipment is that it is not possible to maintain all test bars at the same temperature. A temperature difference of as much as 12° C. (21.6° F.) between hot and cold bars has been measured.

Temperatures for these tests were measured with a Leeds & Northrup optical, potentiometer type, pyrometer.

The test bar design has been changed to threaded ends (see Figure 1). A new set of holder bars is cast for each test.

Figure 1.



TEST BAR USED FOR CREEP TESTS.

Chemical Analyses of Heats Tested:

Sixteen heats of alloys were cast into test bars. The standard tensile test bar of the Alloy Casting Institute was used (see Figure 2). The analyses of these alloys are given in Table I. It was the intention to maintain carbon constant at 0.30 per cent.

(Continued on next page)

(Chemical Analyses of Heats Tested, cont'd) -

TABLE I. - Chemical Analyses.

Alloy No.	Carbon		Silicon		Manganese		Chromium		Nickel	
	Nominal	Actual	Nominal	Actual	Nominal	Actual	Nominal	Actual	Nominal	Actual
	- Per Cent -									
21	0.30	0.33	1.05	1.37	1.05	1.08	10	11.25	10	10.23
22	.30	.30	1.05	1.31	1.05	1.08	10	10.47	18	18.01
23	.30	.26	1.05	1.46	1.05	1.03	10	9.60	25	23.86
24	.30	.25	1.05	1.47	1.05	0.97	10	10.47	30	29.96
25	.30	.24	1.05	1.51	1.05	1.02	10	10.64	35	34.82
26	.30	.31	1.05	1.52	1.05	0.95	10	10.46	40	39.69
27	.30	.29	1.05	1.35	1.05	1.12	15	16.78	10	10.96
28	.30	.29	1.05	1.27	1.05	0.99	15	15.22	18	17.77
29	.30	.22	1.05	1.44	1.05	1.02	15	14.96	25	24.84
213	.30	.14	1.05	1.24	1.05	1.06	20	19.98	10	10.70
214	.30	.24	1.05	1.22	1.05	1.03	20	19.46	18	17.77
215	.30	.17	1.05	1.26	1.05	1.11	20	19.72	25	25.32
219	.30	.30	1.05	1.60	1.05	1.03	25	24.48	18	17.05
220	.30	.28	1.05	1.51	1.05	1.03	25	24.30	25	21.90
224	.30	.29	1.05	1.40	1.05	0.99	30	27.94	18	17.53
225	.30	.32	1.05	1.48	1.05	1.03	30	25.43	25	22.40

-

(Figure 2 comprises Page 4.)
(Text continues on Page 5.)

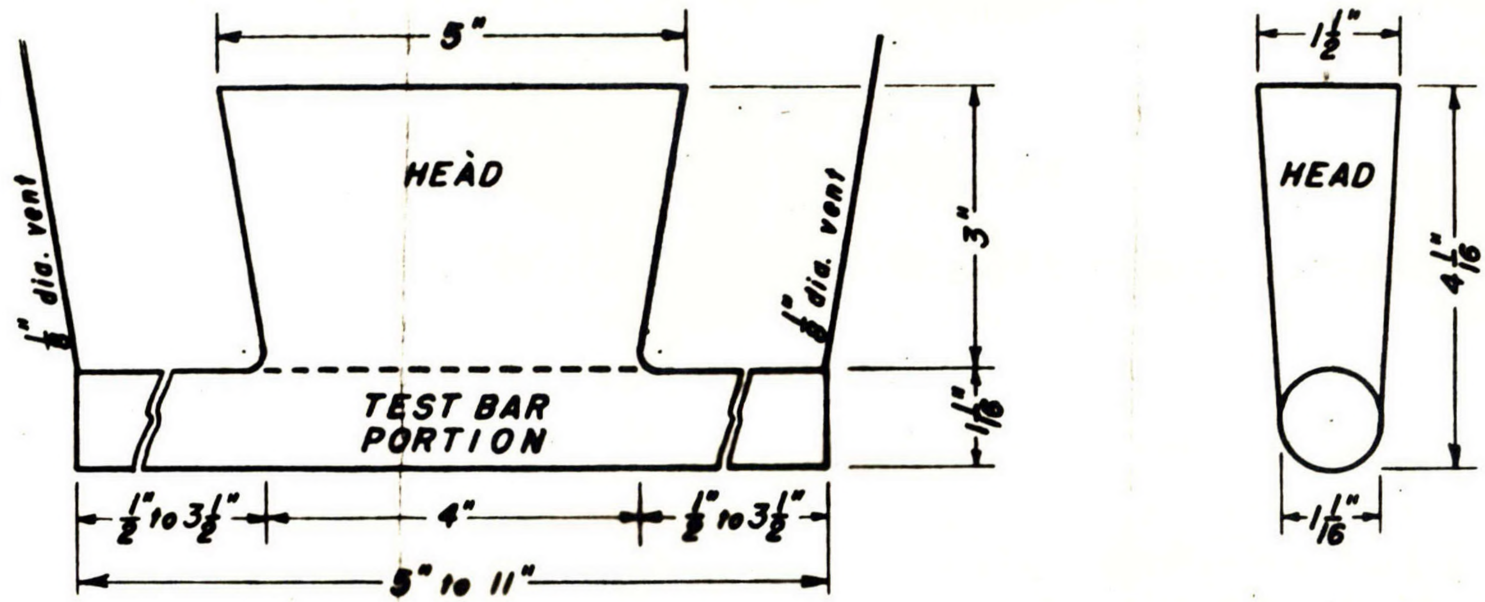


Figure 2.

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 1/2 POUNDS

SCALE 1/2" = 1"	AUGUST 6, 1941

Room-Temperature Tensile Tests:

Tensile tests were carried out on standard 0.505-inch-diameter test bars prepared from each heat. The results are shown in Table II.

TABLE II. - Room-Temperature Tensile Tests.

Alloy No.	Ultimate Tensile Strength, p.s.i.	0.2% Proof Stress, p.s.i.	Elongation: in 2 inches, per cent	Reduction in Area, per cent
21	71,700	40,500	23.0	27.3
22	76,800	33,000	46.0	47.0
23	68,000	33,500	24.0	32.0
24	65,000	33,700	20.0	23.5
25	69,000	36,700	20.0	20.5
26	63,500	42,300	8.0	8.5
27	70,500	39,300	25.0	29.0
28	68,700	35,200	20.0	30.5
29	74,900	35,300	24.0	25.0
213	74,500	36,500	35.0	31.5
214	75,200	36,500	26.0	28.5
215	76,500	36,000	25.0	24.5
219	66,800	34,100	14.1	16.1
220	66,000	34,500	15.5	15.0
224	63,200	37,500	9.5	11.0
225	70,400	38,700	14.5	11.5

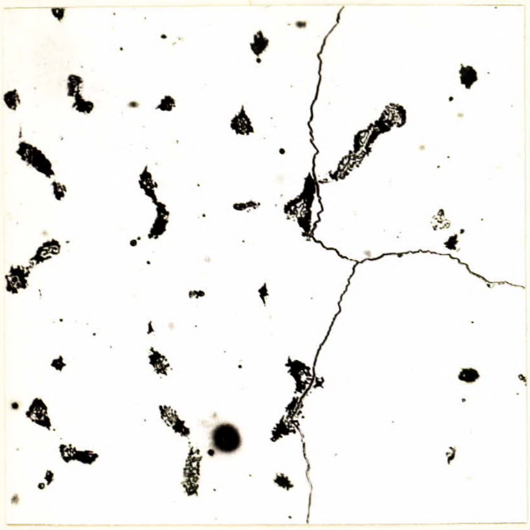
"As Cast" Microstructure:

Typical "as cast" microstructures are shown in Figures 3, 4 and 5.

(Figures 3 to 5 follow)
(on Page 6. Text is)
(resumed on Page 7.)

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

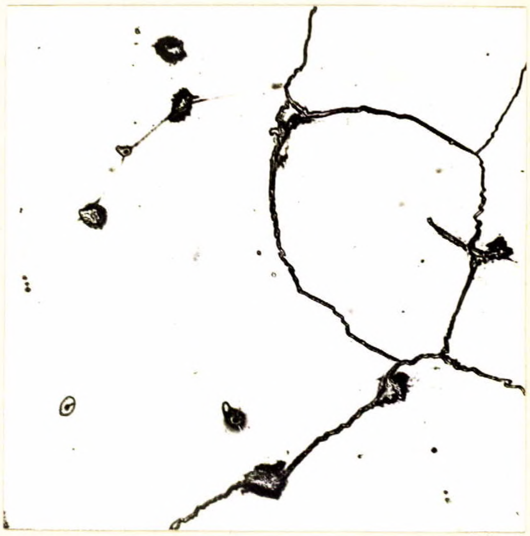
Figure 3.



X200, etched in
Vilella's reagent.

ALLOY NO. 24.

Figure 4.



X200, etched in
Vilella's reagent.

ALLOY NO. 29.

Figure 5.

X200, etched in
Vilella's reagent.

ALLOY NO. 225.

CREEP TESTS:

Creep tests have been conducted on these alloys at a temperature of approximately 1170° C. However, since actual temperature differences of as much as 12° C. between different test bars have been measured in the same furnace, it is impossible to state an exact test temperature. The applied stress was 500 pounds per square inch.

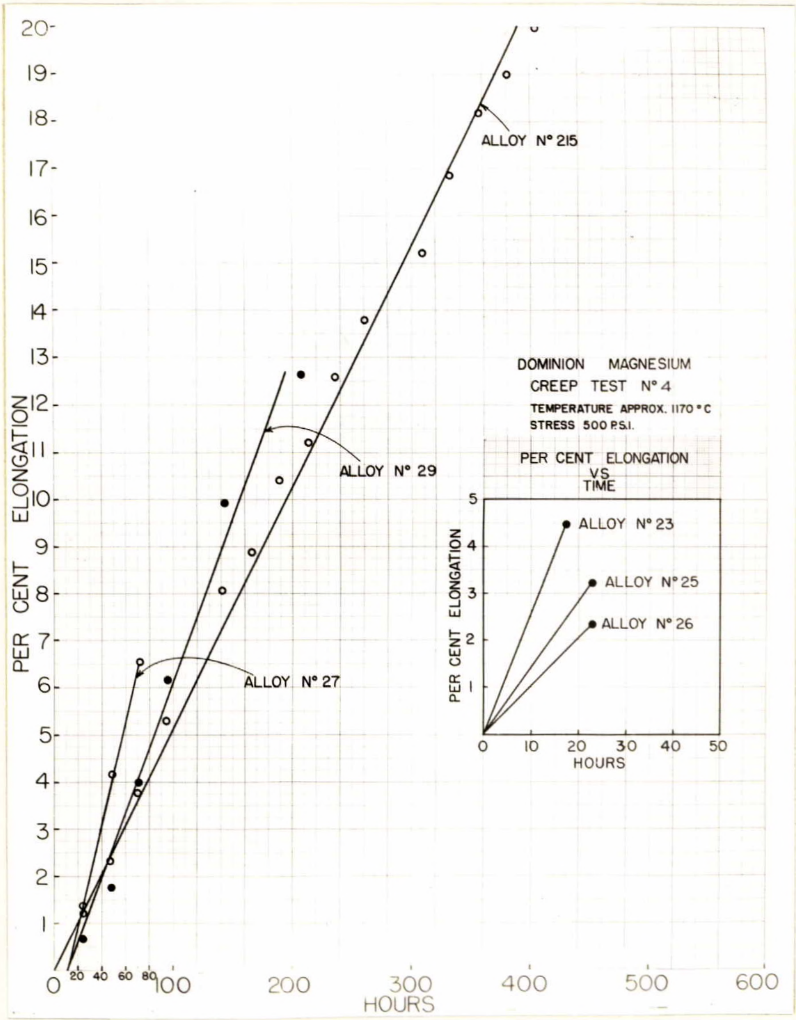
The results of Creep Tests Nos. 4, 5, 6 and 7 are given in Table III and Figures 6, 7, 8, 9 and 10.

TABLE III. - Creep Test Data.

Alloy No.	Life of Bar, hours	Total Elongation at End of Test, per cent
Creep Test No. 4.		
21	0	--
23	17.5	4.48
25	23	3.12
26	23	2.34
27	72	6.54
28	10	--
29	167.5	12.54
215	Unbroken after 500 hours.	20.83
Creep Test No. 5.		
22	19	--
24	43	4.43
213	92	9.68
214	139	16.54
219	Furnace	17.49
220	failed	11.79
224	after	17.06
225	400 hours.	12.81
Creep Test No. 6.		
21	1/4	--
22	19	--
23	19	2.86
24	67	7.14
25	--	--
26	67	9.97
27	19	3.59
28	43	5.42
Creep Test No. 7.		
29	263	26.64
213	215	19.48
214	143	15.99
215	167	24.70
219	No fracture in 500 hours.	30.08
220	" " " "	31.61
224	" " " "	32.01
225	" " " "	15.16

(Creep Tests, cont'd) -

Figure 6.



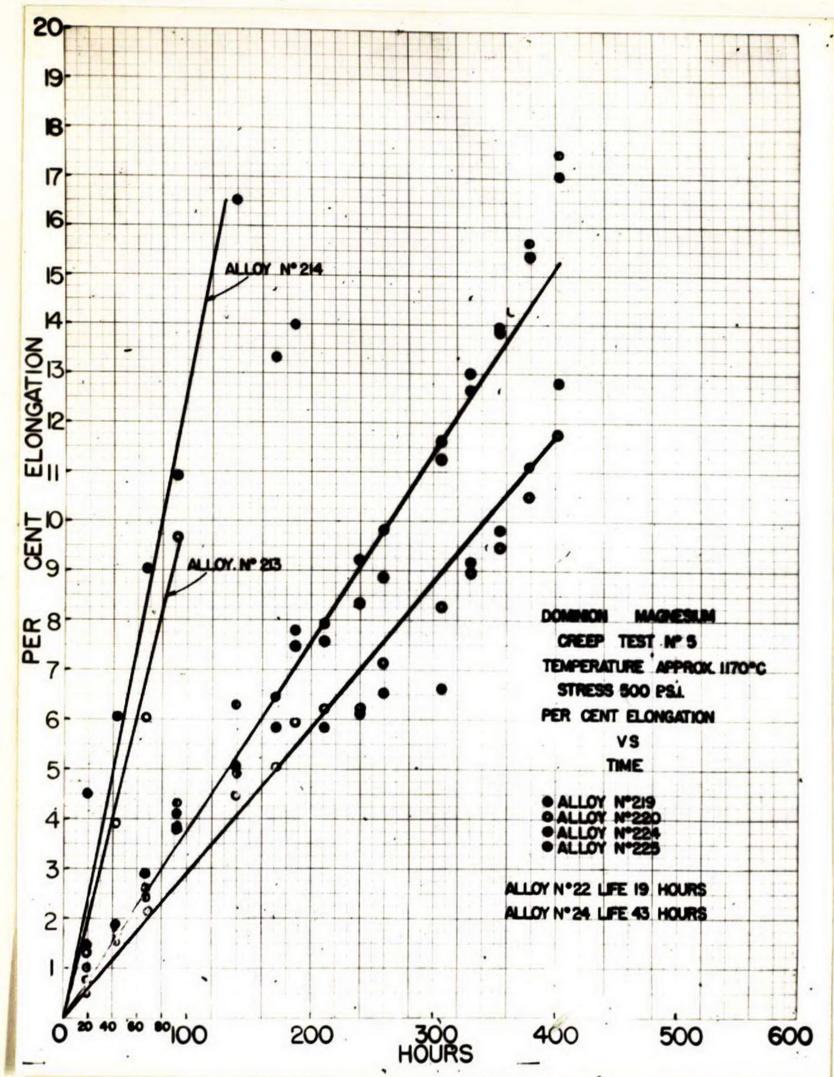


Figure 7.

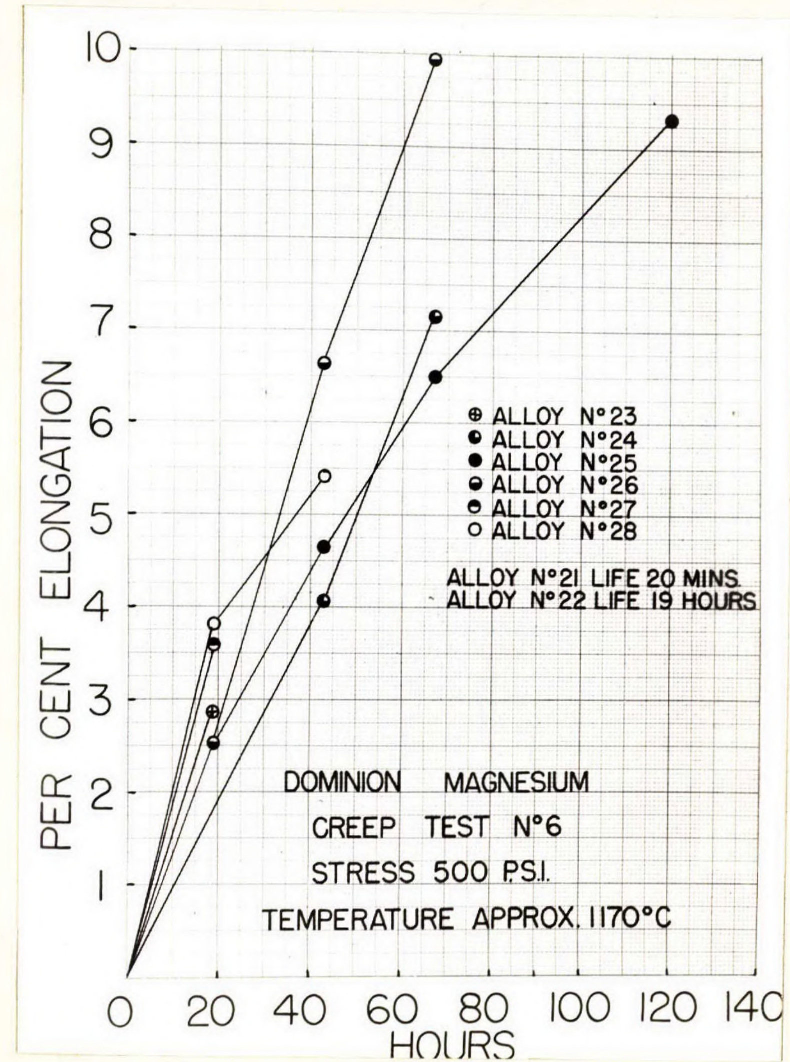


Figure 8.

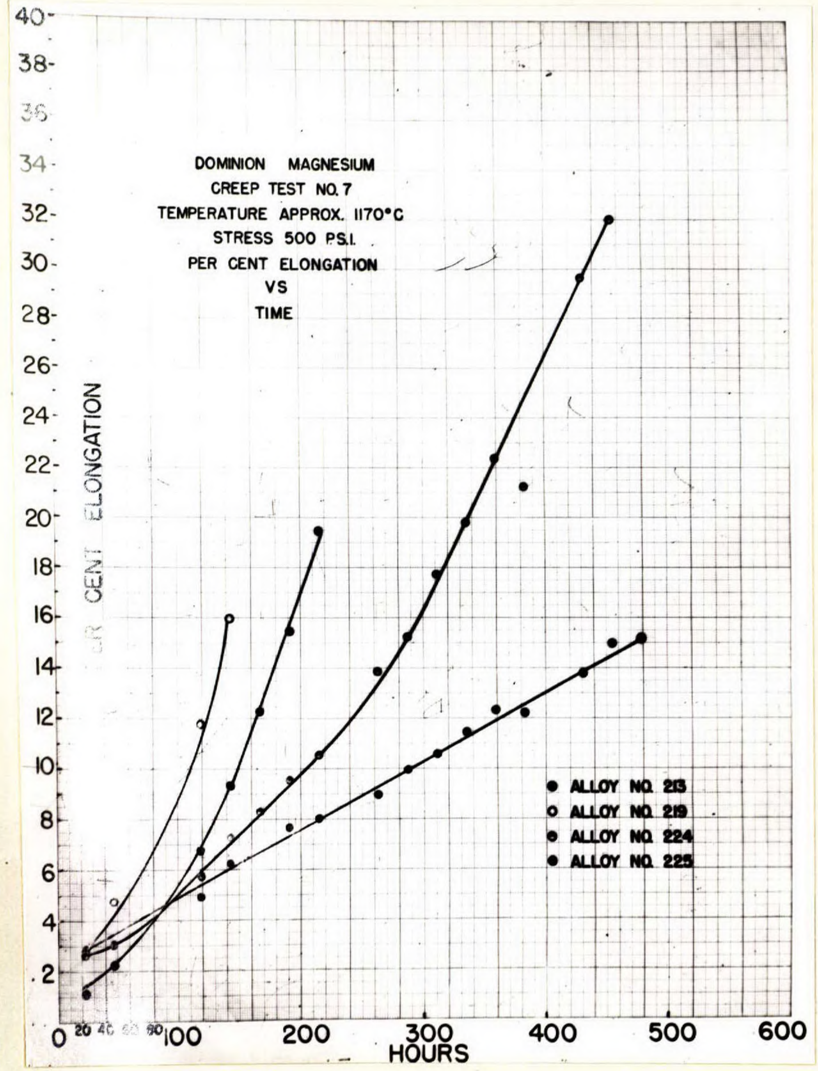


Figure 9.

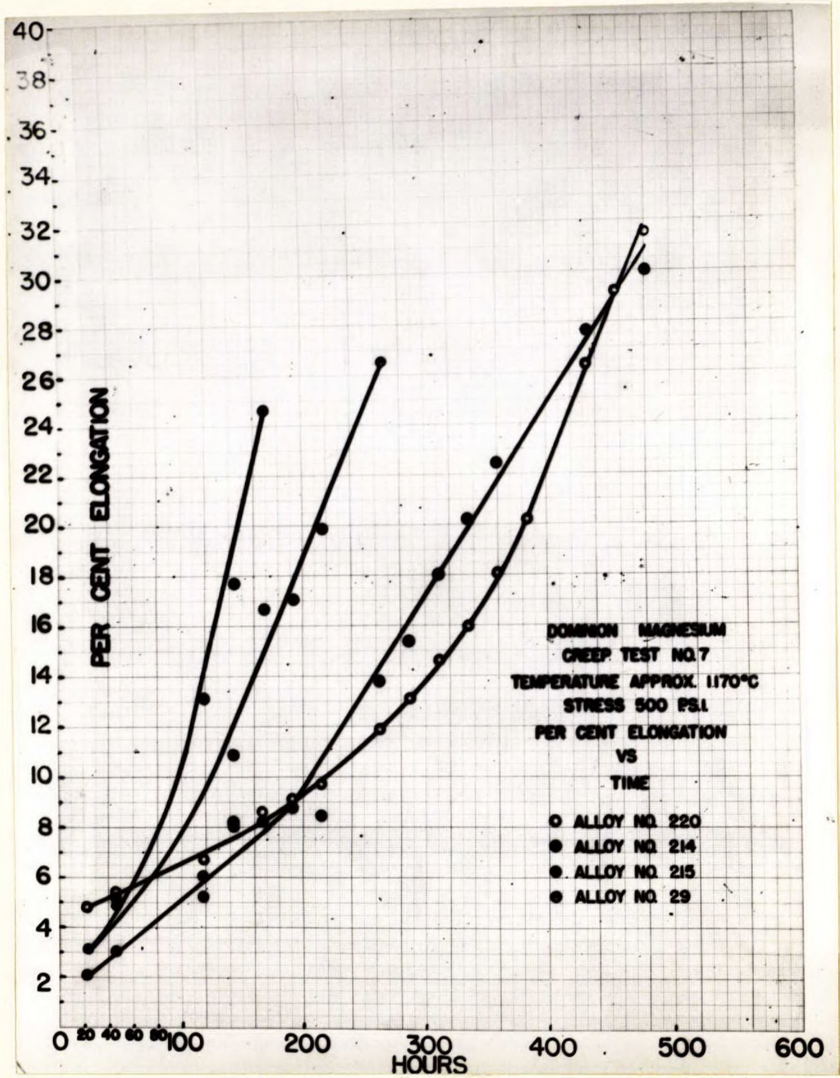


Figure 10.

(Note: Curve for Alloy No. 219 should be for Alloy No. 214 and curve for Alloy No. 214 should be for Alloy No. 219.)

(Creep Tests, cont'd) -

In an attempt to illustrate the effect of increasing the nickel content while holding the chromium content constant, Tables IV to VII are presented below:

TABLE IV. - Creep Results of 10 Per Cent Chromium Alloys with Varying Nickel Content.

Alloy No.	Chromium, per cent	Nickel, per cent	TIME TO FRACTURE, HOURS: Test No. 4	Test No. 5	Test No. 6	ELONGATION, PER CENT: Test No. 4	Test No. 5	Test No. 6
21	11.25	10.23	--	--	$\frac{1}{4}$	--	--	--
22	10.47	18.01	--	19	19	--	--	--
23	9.60	23.86	17.5	--	19	4.48	--	2.86
24	10.47	29.96	--	43	67	--	4.43	7.14
25	10.64	34.82	23	--	--	3.21	--	--
26	10.46	39.69	23	--	67	2.34	--	9.97

TABLE V. - Creep Results of 15 Per Cent Chromium Alloys with Varying Nickel Content.

Alloy No.	Chromium, per cent	Nickel, per cent	TIME TO FRACTURE, HOURS: Test No. 4	Test No. 6	Test No. 7	ELONGATION, PER CENT: Test No. 4	Test No. 6	Test No. 7
27	16.78	10.96	72	19	--	6.54	3.59	--
28	15.22	17.77	10	43	--	--	5.42	--
29	14.96	24.84	167.5	--	263	12.54	--	26.64

TABLE VI. - Creep Results of 20 Per Cent Chromium Alloys with Varying Nickel Content.

Alloy No.	Chromium, per cent	Nickel, per cent	TIME TO FRACTURE, HOURS: Test No. 4	Test No. 5	Test No. 7	ELONGATION, PER CENT: Test No. 4	Test No. 5	Test No. 7
213	19.98	10.70	--	92	215	--	9.68	19.48
214	19.46	17.77	--	139.0	143	--	16.64	15.99
215	19.72	25.32	No fracture.	--	167	20.83	--	24.70

TABLE VII. - Creep Results of 25 Per Cent to 28 Per Cent Chromium Alloys with Varying Nickel Content.

Alloy No.	Chromium, per cent	Nickel, per cent	ELONGATION AT END OF: 400 HOURS, PER CENT: Test No. 5	Test No. 7	RATE OF CREEP, PER CENT PER HOUR: Test No. 5	Test No. 7
219	24.48	17.05	17.5	24.8	0.044	0.062
220	24.30	21.90	11.75	22.0	0.029	0.055
224	27.94	17.53	11.2	26.5	0.043	0.066
225	25.43	22.40	12.8	15.5	0.032	0.039

(Creep Tests, cont'd) -

Tables VIII to X are intended to illustrate the effect of increasing chromium while holding nickel constant.

TABLE VIII - Creep Results of 10 Per Cent Nickel Alloy with Varying Chromium Content.

Alloy No.	Nickel, per cent	Chromium, per cent	TIME TO FRACTURE, HOURS: Test No. 4	Test No. 5	Test No. 6	Test No. 7	ELONGATION, PER CENT: Test No. 4	Test No. 5	Test No. 6	Test No. 7
21	10.23	11.25	¼	--	¼	--	--	--	--	--
27	10.96	16.78	72	--	19	--	6.54	--	3.59	--
213	10.70	19.98	--	92	--	215	--	9.68	--	19.48

TABLE IX. - Creep Results of 18 Per Cent Nickel Alloy with Varying Chromium Content.

Alloy No.	Nickel, per cent	Chromium, per cent	TIME TO FRACTURE, HOURS: Test No. 5	Test No. 6	Test No. 4	Test No. 7	ELONGATION, PER CENT: Test No. 5	Test No. 6	Test No. 4	Test No. 7
22	18.01	10.47	19	19	--	--	--	--	--	--
28	17.77	15.22		43	10	--	--	5.42		
214	17.77	19.46	139.0	--	--	143.0	16.54	--	--	15.99
219	17.05	24.48	No fracture.				17.5	--	--	24.8
224	17.53	27.94	No fracture.				17.2	--	--	26.5

TABLE X. - Creep Results of 25 Per Cent Nickel Alloys with Varying Chromium Content.

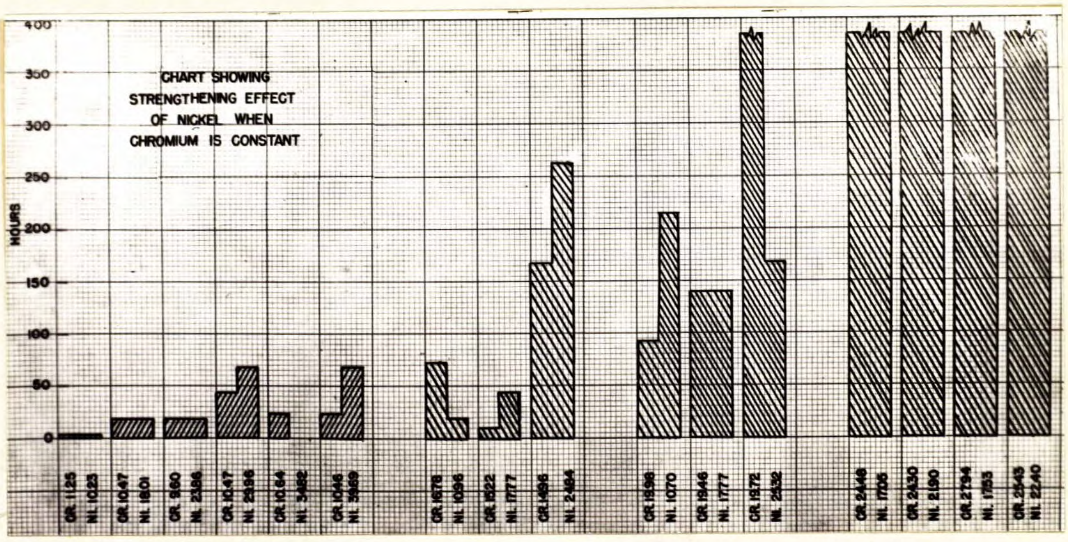
Alloy No.	Nickel, per cent	Chromium, per cent	TIME TO FRACTURE, HOURS: Test No. 4	Test No. 5	Test No. 6	Test No. 7	ELONGATION, PER CENT: Test No. 4	Test No. 5	Test No. 6	Test No. 7
23	23.86	9.60	17.5	--	19	--	4.48	--	2.86	--
29	24.84	14.96	167.5	--	--	263	12.54	--	--	26.64
215	25.32	19.72	No fracture.	--	--	167	20.83	--	--	24.70
220	21.92	24.30	No fracture.				--	11.75	--	22.0
225	22.40	25.43	No fracture.				--	12.8	--	15.5

The data given in Tables IV and VII are shown graphically in Figure 11. The data given in Tables VIII to X are shown graphically in Figure 12.

(Figures 11 and 12)
(follow, on Page 13.)

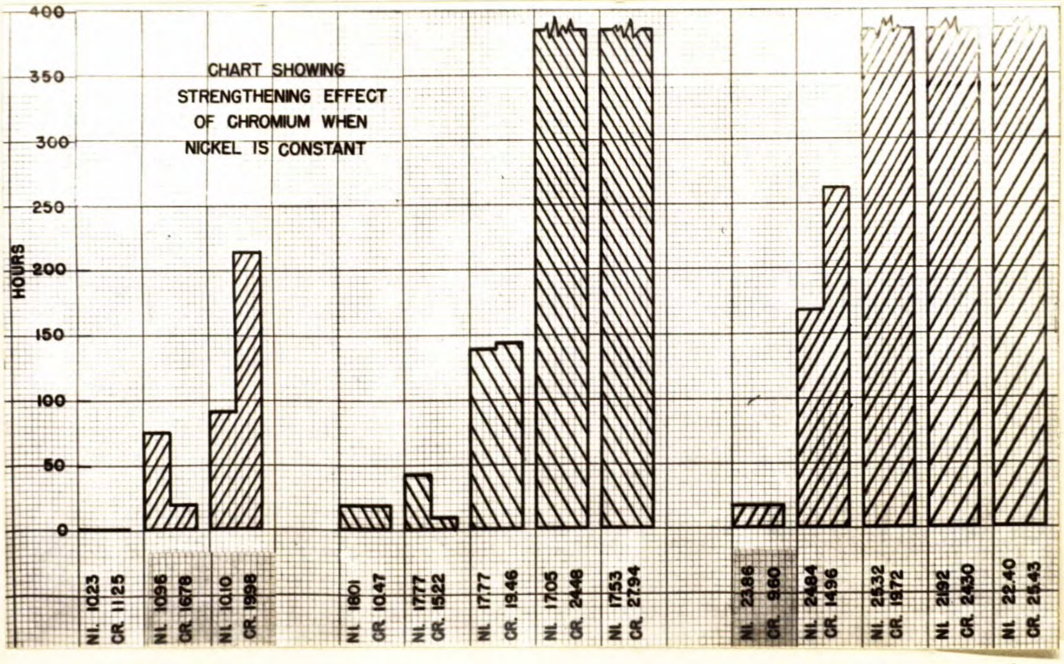
(Creep Tests, cont'd) -

Figure 11.



GRAPHICAL PRESENTATION OF DATA IN
TABLES IV, V, VI and VII.

Figure 12.



GRAPHICAL PRESENTATION OF DATA IN
TABLES VIII, IX and X.

DISCUSSION OF RESULTS:

It was noted that the test bars that contained only 10 per cent chromium oxidized very readily at the test temperature of 1170° C. (2138° F.). However, retorts of 15 per cent chromium, 35 per cent nickel have shown excellent oxidation resistance when exposed to a normal air atmosphere at this temperature over periods as long as one year. It is therefore evident that to give adequate protection against oxidation in a normal air atmosphere at temperatures over 1100° C.(2012° F.), at least 15 per cent chromium is necessary.

An examination of the data as arranged in Tables IV to VII, and as depicted graphically in Figure 6, shows that nickel has a strengthening effect. With 15 per cent chromium present, as the nickel increases from 10 per cent to 25 per cent the life of the test bar increases. However, even 25 per cent nickel in conjunction with 15 per cent of chromium is not enough to satisfactorily strengthen the alloy. Since 35 per cent nickel in conjunction with 15 per cent chromium does give an alloy that behaves satisfactorily in the retorts in the Pidgeon magnesium process, it would be a reasonable assumption that in the presence of 15 per cent chromium at least 35 per cent nickel is necessary to impart the required strength to the alloy.

The data as arranged in Tables VIII to X and shown graphically in Figure 7 show that as the chromium is increased less nickel is required to give the alloy satisfactory strength. However, the strengthening effect of increasing nickel is still in evidence when the chromium content is in the range of 24 per cent to 28 per cent, as is evident in Table VII. Alloys Nos. 219 and 224 show a higher creep rate than do Alloys Nos. 220 and 225. The extra chromium in Alloy No. 224 does not seem to have any beneficial effect here. However, the higher nickel

(Discussion of Results, cont'd) -

in Alloys Nos. 220 and 225 seems to stiffen the metal considerably.

-

CONCLUSIONS:

1. Chromium must be present in excess of 15 per cent in nickel-chromium austenitic steels to give satisfactory resistance to oxidation at temperatures over 1100° C. (2012° F.) in a normal air atmosphere.

2. Increasing chromium above 15 per cent increases the resistance to creep.

3. Increasing nickel increases resistance to creep. With 15 per cent chromium, 25 per cent nickel is not enough but 35 per cent nickel gives good strength at high temperatures.

FUTURE WORK:

To round out the phase of the work that has to do with the variations in the nickel-chromium ratio of the nickel-chromium austenitic alloys, and in an attempt to determine whether the 28 per cent chromium, 20 per cent nickel alloy or the 35 per cent nickel, 15 per cent chromium alloy is better for applications at temperatures above 1100° C. (2012° F.), a test will be run using Alloys Nos. 12, 13, 19, 220 and 224. Nos. 12, 13 and 19 are of the 35 per cent nickel, 15 per cent chromium type and Alloys Nos. 220 and 224 are of the 28 per cent chromium, 20 per cent nickel type.

It is not considered expedient at present to investigate further the effect of variations in the nickel-chromium ratio on the properties of these alloys at temperatures over 500° F.

The next stage will be to investigate the effect of silicon on a 35 per cent nickel, 15 per cent chromium type

(Future Work, cont'd) -

of alloy. Silicon is reported to retard the decarburizing rate of these alloys. Accordingly, a series of heats ranging from 0.25 per cent silicon to 2.0 per cent silicon have been prepared. These will be tested at 1170° C. (2138° F.) to determine both the relative rates of decarburization and the relative strength.

oooooooooooo
oooooooo
oo

HVK:LB.