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THE MICROSTRUCTURE
OF
SOME COMMERCIAL NICKEL STEELS

R.N. Stubbs, Jr.

April, 1920

Investigations leading to the degree of
Bachelor of Science in Metallurgy

Approved:

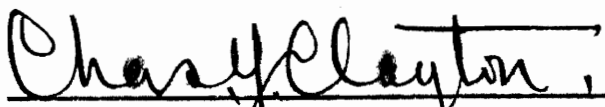

Assoc. Professor of Metallurgy

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THE MICROSTRUCTURE
OF
SOME COMMERCIAL NICKEL STEELS

In this thesis it was the author's intention to take up the microstructure of such nickel steel as is generally employed for structural work. Two steels, A and B, were used throughout the investigations, having the following respective compositions:

Steel A

C	0.38%
Mn.	0.55
P	0.026
S	0.022
Si	0.09
Ni	2.76
Cr	0.16

Steel B

C	0.26%
Mn.	0.40
P	0.02
S	0.02
Si.	0.08
Ni.	3.5
Cr.	0.15

The material designated as A was from a nickel-steel forging while that designated as B was a rolled, nickel-steel rod.

Heat Treatments

Specimen Number	1st Quench from	In	2d Quench from	In
1 (Original)				
2	700 degrees C	Water		
3	750 " "	" "		
4	800 " "	" "		
5	850 " "	" "		
6	900 " "	" "		
7	950 " "	" "		
8	900 " "	" "	600 degrees C	Water
1-16	950 " "	Brine		
2-9	950 " "	Air		
3-10	950 " "	Oil		
4-11	950 " "	Furnace		
5-12	900 " "	Air		
6-13	900 " "	Brine		
7-14	850 " "	Air		
8-15	850 " "	Brine		
9	900 " "	Water	200 " "	" "
10	900 " "	" "	300 " "	" "
11	900 " "	" "	400 " "	" "
12	900 " "	" "	500 " "	" "
13	900 " "	" "	600 " "	" "
14	900 " "	" "	650 " "	" "
15	900 " "	" "	700 " "	" "
16	900 " "	" "	750 " "	" "
9-1	1100 " "	Air		
10-2	1100 " "	Water		
11-3	1100 " "	Furnace		
12-4	1100-600	"	600 room temp.	"
17	1100 " "	Brine		
18	1100-850	Furnace	850 " "	" "
19	1100-800	"	800 " "	" "
20	1100-750	"	750 " "	" "
21	1100-700	"	700 " "	" "
22	1100-650	"	650 " "	" "
23	1100-500	"	500 " "	" "
24	1100 " "	" "		
40	1325 " "	Water		
41	1325 " "	Air		
42	1350 " "	Water		
43	1350 " "	Air		
44 / /	1380 " "	Water		
45	1380 " "	Air		

Specimen Number	Max. Temp.	Time	Cooled in
25 . . .	950 degrees C	1/2 hr.	Air
26 . . .	1100 "	1 hr.	"
27 . . .	1100 "	1 1/2 hr.	"
28 . . .	1100 "	2 hr.	"
29 . . .	1100 "	2 1/2 "	"
30 . . .	1100 "	3 hr.	"
31 . . .	1150 "	"	"
32 . . .	1150 "	"	Water
33 . . .	1200 "	"	Air
34 . . .	1200 "	"	Water
35 . . .	1250 "	"	Air
36 . . .	1250 "	"	Water
37 . . .	1250 "	"	Oil
38 . . .	1265 "	"	Brine
39 . . .	1265 "	"	Furnace

For heating purpose, three furnaces were used: two electric resistance furnaces, the one depending upon nichrome wire elements for its resistance, the other upon carbon plates; one muffle type, gasoline fired furnace. Low temperature measurements (up to 700 degrees C) were made with a base metal ("Pyod") couple; high temperatures were measured with a platinum-platinum rhodium couple and Wilson-Maulen indicator.

All bars were treated in the muffle furnace, and due to insufficient size of available mufflers some bars received uneven heating and consequently gave poor results, some even breaking in the grips of the testing machine.

Etching Reagents

The following are the principal solutions used for microscopic work:

1% HCl solution in ethyl alcohol --
Hoyt recommends this solution for iron --
carbon alloys in hardened or annealed state.

Iodine--

Suggested by Osmond who used tincture of iodine, putting a drop or two on the polished surface and leaving until decolorized.

Nitric acid--

5% solutions in alcohol are usually used. Lautsberry says that the success of this method depends on thoroughly washing the specimen in alcohol, and that no water should be allowed to touch the specimen. The author experienced no difficulty, however, from washing the specimen in water after etching. The best method of application is by the use of cotton dipped into the solution and then rubbed on the specimen. This method did away with the severe darkening effect nitric acid so often has on the polished surface.

Picric acid--

As generally used, it is a saturated solution in alcohol.
Archbutt uses: 80 vols. picric acid in alcohol
20 vols. 2% nitric acid in alcohol.

Rosenhains and Houghton's reagent--

Ferric chloride	30 gms.
Hydrochloric acid (conc)	100 c.c.
Cupric chloride	10 gms.
Stannous chloride5 "
Water.	1000 c.c.

Sodium picrate--

Sodium hydroxide	24 gms.
Picric acid	2.0 "
Water.	74 c.c.

Appendix

Triangulated structure.-- In specimen number 9-1 (air cooled from 1100 degrees C) will be seen a structure common to commercial nickel steels which have been air cooled from the neighborhood of 1100 degrees. Regarding this triangular structure, it might be well to quote from "Tests of Metals, 1917", as follows: "The structure developed was in all probability developed by overheating previous to the quenching in oil (air for small specimens)"***"The cracks (in pulling tests) occurred in the triangular structure". *** "Analysis showed that areas of predominating triangular structure were lower in carbon and chromium", ***"The triangular arrangement of ferrite and the constituent containing carbon was developed by heating to a high temperature and cooling in air. Holding the piece at maximum temperature assisted materially in developing the triangulated structure. In no case, regardless of time of soaking, was the structure developed when the temperature did not exceed 800 degrees C.

Texture constituents of nickel steels.-- Most authorities agree that there are four characteristic structures in nickel steels, which structure being present depending upon the nickel content and the heat treatment. These constituents are: (1) iron carbide in the form of pearlite or cementite, (2) nickel ferrite, (3) nickel martensite, (4) nickel polyhedra. The pearlite of nickel steels contains, according to Waterhouse, 0.7% carbon and is formed in slowly cooled material at varying temperatures, depending upon the nickel and carbon content. A close approximation of the constituents in a slowly cooled steel would be as follows:

	<hr style="width: 10%; margin: 0 auto;"/>	
0.12%C	0.25%C	0.8%C
0-10% Ni -pearlite and ferrite	0-9% Ni-pearlite	0-5% Ni-ferrite
10-12% Ni Martensite and ferrite	7-10% Ni-Martensite	5-7% Ni-Martensite
12-27% Ni-Martensite	Above 15% Ni-polyhedric	7-10% Ni-Martensite
Above 27% Ni-polyhedric		10-15% Ni-Martensite and polyhedric
		Above 15%-polyhedric.

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PHOTOMICROGRAPHS

No. 1:

Original steel A, having been slightly forged; shows large grain size and characteristic pearlite-ferrite structure of nickel steels. The cementite and ferrite layers are less laminated in nickel pearlite than in the pearlite of plain-carbon steels.

No. 2:

After heating to 700 degrees C and quenching in water. The micrograph shows no evidence of carbon diffusion or grain refinement. This due to the short length of time held at the temperature. Pearlite-ferrite structure.

No. 3:

After heating to 750 degrees C and quenching in water. Shows some diffusion of carbon and a little refinement of grain, Pearlite-ferrite.

No. 4:

Quenched in water from 800 degrees C: Fine Martensitic structure.



No. 1

X 730



No. 2

X730

Nickel Steel A



No. 3

X730



No. 4

X730

No. 5:

Heated to 850 degrees C and quenched in water,
fine grained Martensite

No. 6:

Quenched in water from 900 degrees C. Martensite
is becoming more coarse.

No. 7:

Quenched in water from 950 degrees C. Coarse Mar-
tensite.

No. 8:

The complete grain refinement in this specimen
was obtained by quenching in water from 900 degrees
C, reheating to 600 degrees and again quenching.

Specimens No. 9 to No. 13 inclusive showed fine
grain structures similar to No. 8. Specimen 14,15,16,
had a coarse Martensitic structure.



No. 5

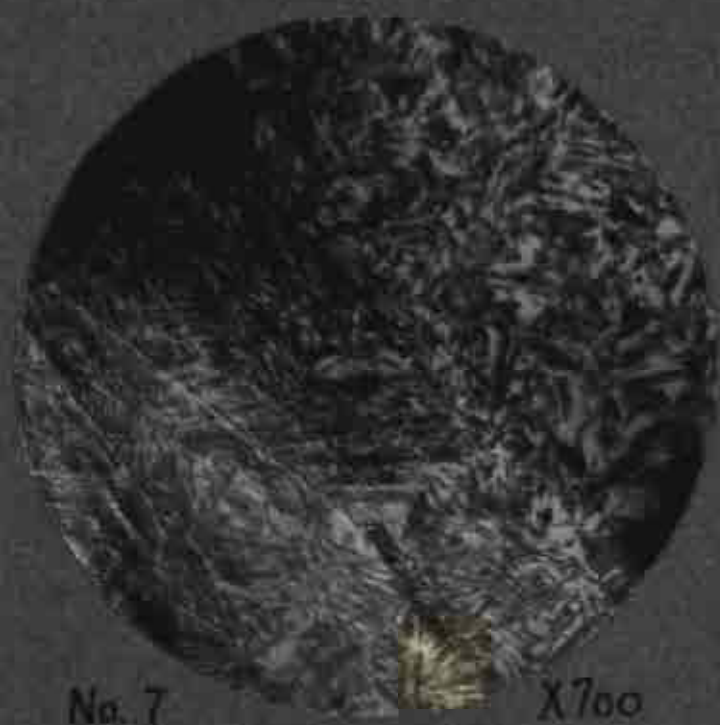
X730



No. 6

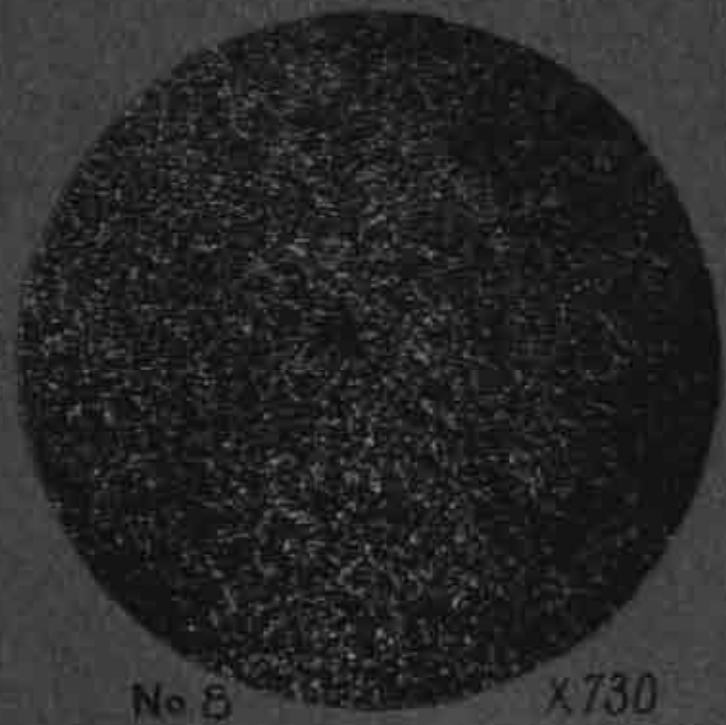
X700

Nickel Steel A



No. 7

X700



No. 8

X730

No. 1-16:

Coarse Martensite obtained by heating to 950 degrees C, and quenching in ice brine.

No. 2-9:

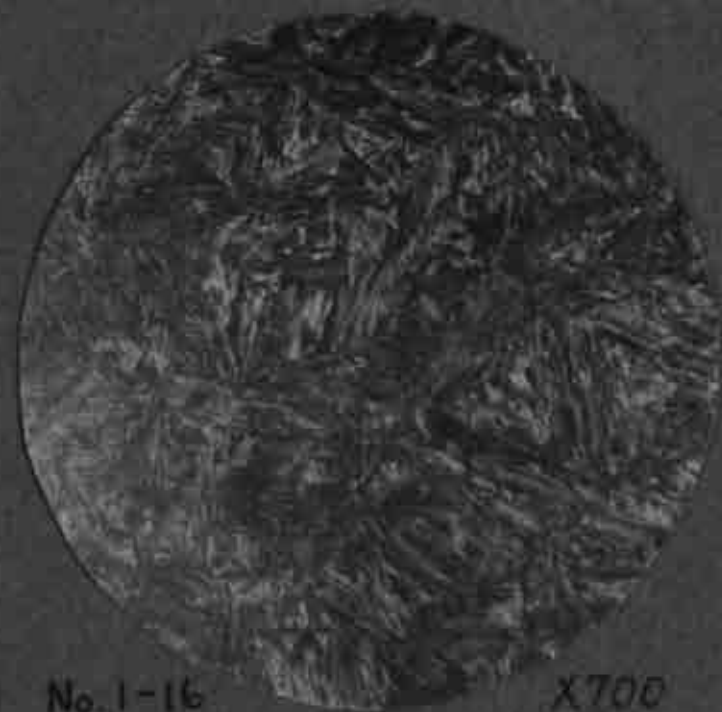
Cooled in air from 950 degrees C. Large pearlite grains with ferrite segregation along boundary lines.

No. 3-10:

Quenched in lubricating oil from 950 degrees C. Fine grain Martensite.

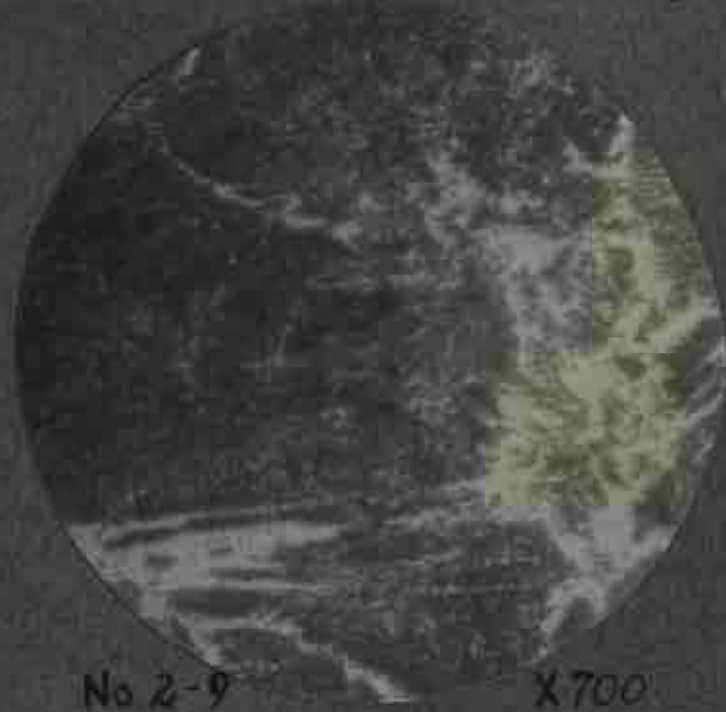
No. 4-11:

Furnace cooled from 950 degrees C. Large ferrite (white) segregation with pearlite (black).



No. 1-16

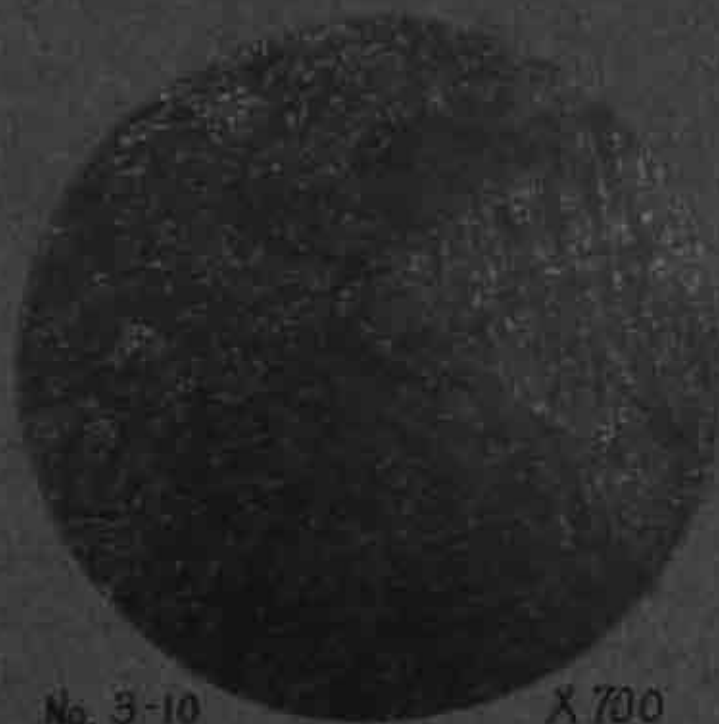
X700



No. 2-9

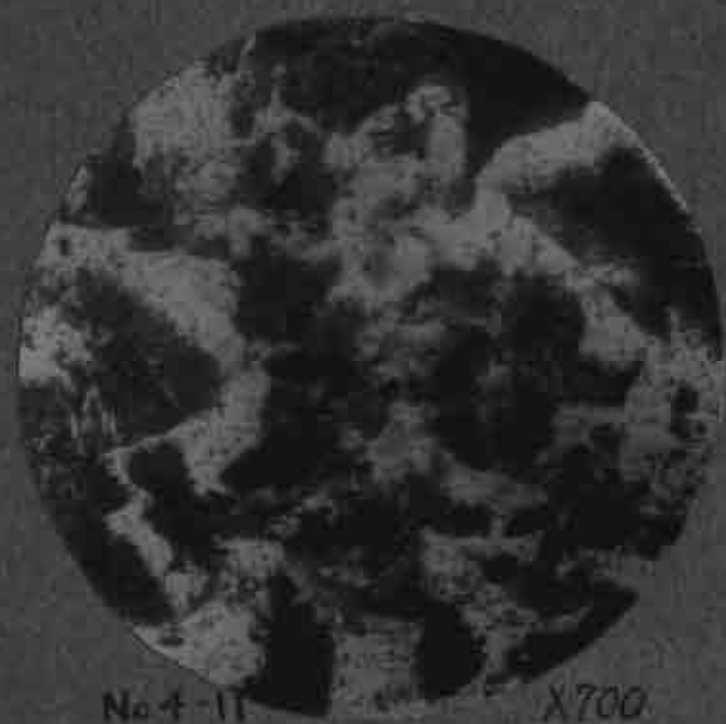
X700

Nickel Steel A



No. 3-10

X700



No. 4-11

X700

No. 5-12:

Air cooled from 900 degrees C, Ferrite precipitation around pearlite grains.

No. 6-13:

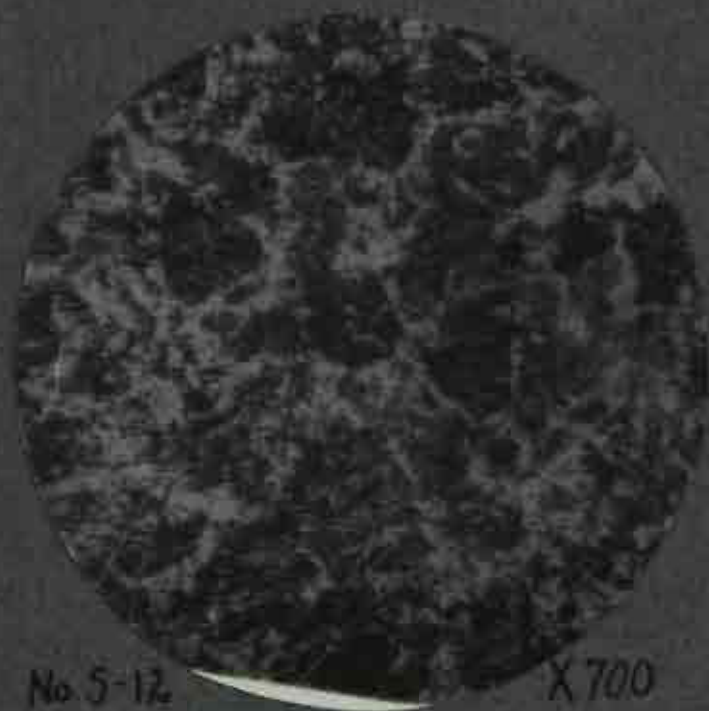
Quenched in ice brine from 900 degrees C, Coarse Martensite.

No. 7-14:

Air cooled from 850 degrees C. Ferrite segregation around pearlite grains.

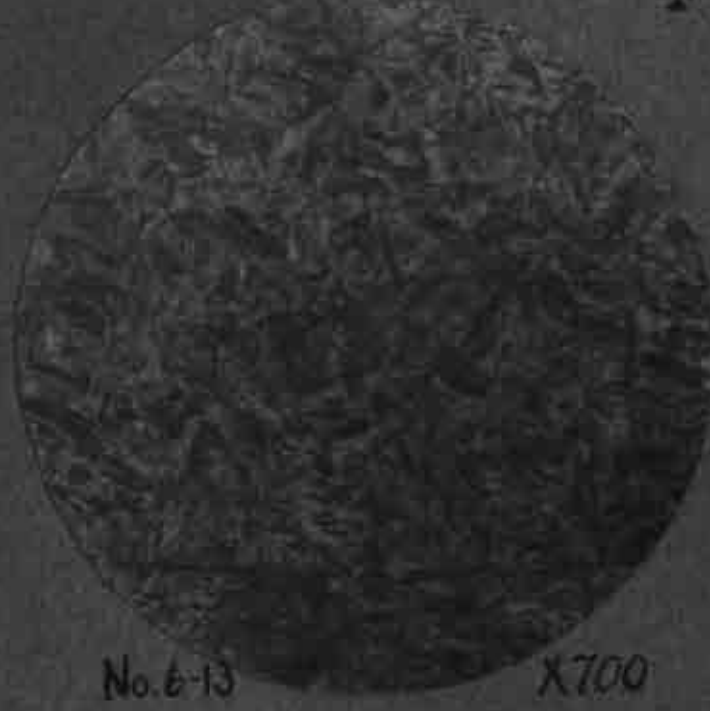
No. 8-15:

Quenched in ice brine from 850 degrees C. Martensitic structure.



No. 5-12

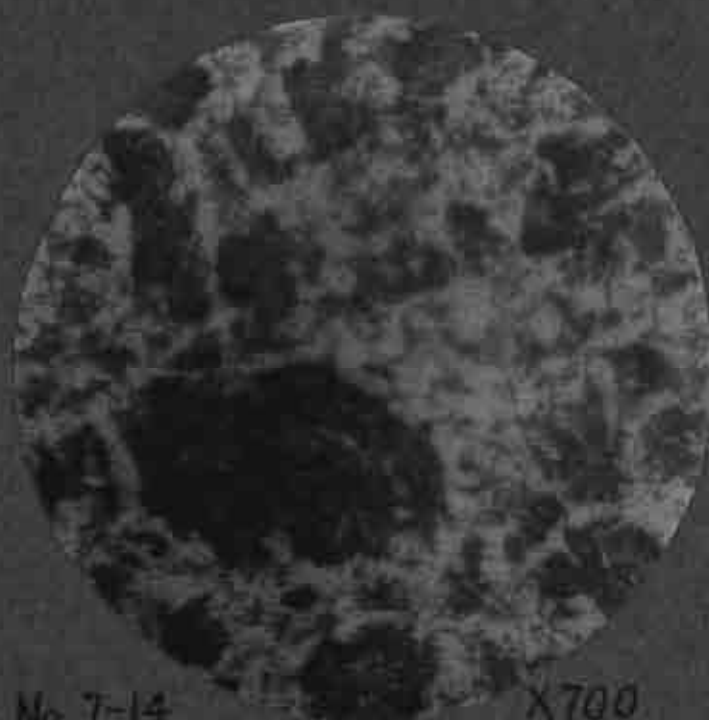
X700



No. 6-13

X700

Nickel Steel A



No. 7-14

X700



No. 8-15

X700

No. 9-1:

Air cooled from 1100 degrees C. Ferrite and pearlite. See appendix for discussion of triangulated structure in air cooled specimens.

No. 10-2 (a):

Quenched in water from 1100 C. Martensite and ferrite.

No. 10-2 (b):

Higher magnification of Martensite area of specimen quenched in water from 1100 degrees C.

No. 11-3:

Cooled slowly in furnace from 1100 degrees C. Ferrite and pearlite.



No. 9-1

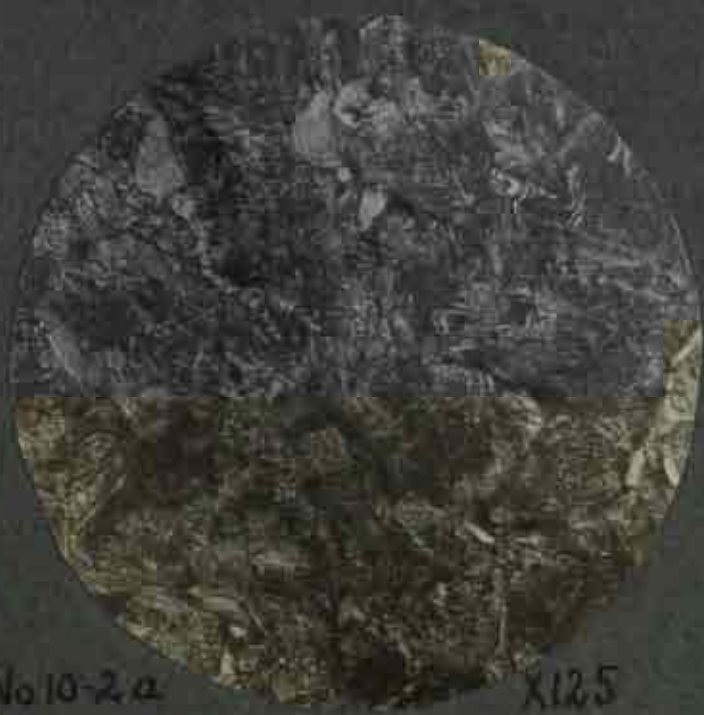
X125



No. 10-2b

X700

Nickel Steel A



No. 10-2a

X125



No. 11-3

X125

No. 12-4:

Furnace cooled from 1100 to 600 degrees.
Quenched in water from 600. Ferrite and Martensite.

Nos. 17,18,19,20,21,22,23:

Martensite similar to that of No. 12-4

No. 24:

Exactly like No. 11-3: Duplicate treatment

Nos. 25-31 inclusive, and Nos. 33 and 35 showed structures
similar to No. 9-1.

Nos. 32,34, and 37 showed Martensite similar to that in
No. 1-16.

No. 38:

Coarse Martensite having twinned appearance,
Quenched in brine from 1265 degrees C.

No. 39:

Furnace cooled from 1265 degrees C. Ferrite and
pearlite.

No. 46:

Cast nickel steel, ferrite and pearlite.



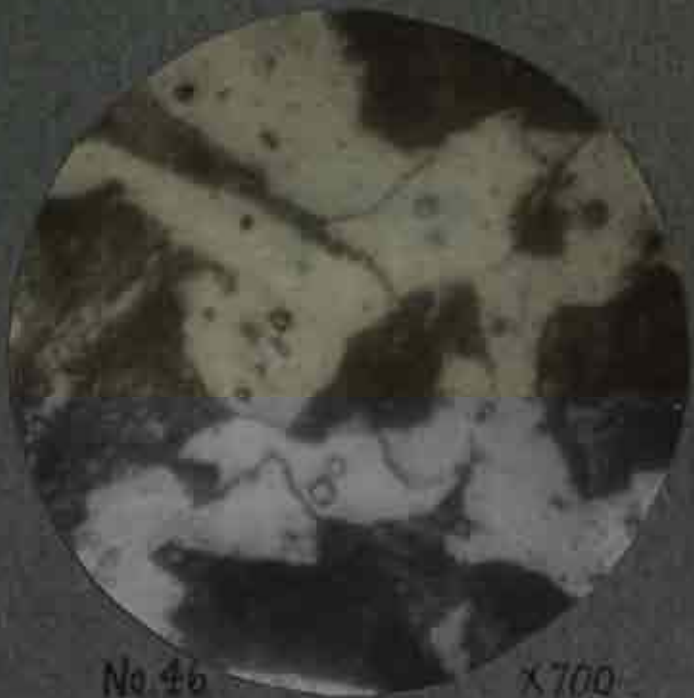
No. 17-4 Ni Steel A X125



No. 38 Ni Steel B X700



No. 39 Ni Steel B X100



No. 46 Ni Steel B X700

No. 47 and No. 48:

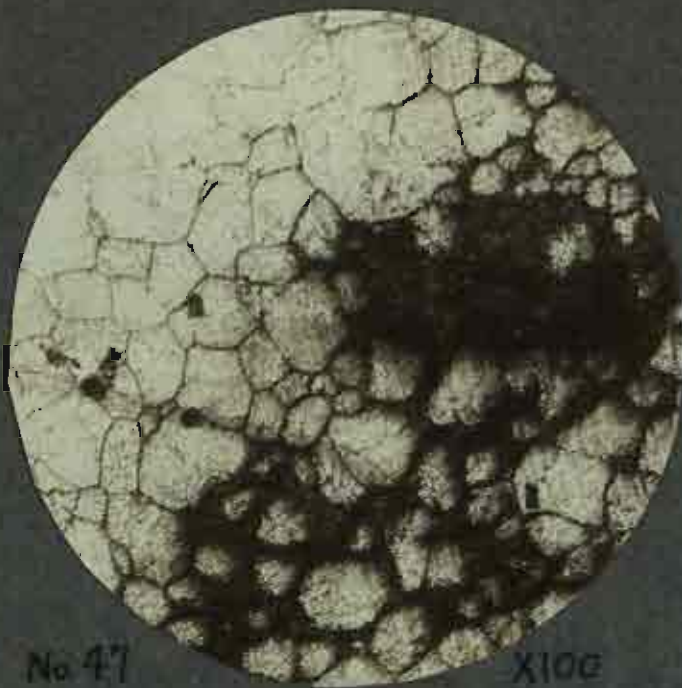
Decarburized areas around edges of specimens cooled in air from high temperatures. The dark grain boundaries are oxide films.

No. 49:

Micrograph of original nickel steel B, showing pearlite and ferrite. The material had been rolled and annealed.

No. 50:

Low magnification of nickel steel A, showing ferrite and pearlite. Material had been forged and slightly annealed.



No. 47

X100

Ni. Steel B



No. 48

X100

Ni. Steel B



No. 49

X100



No. 50

X100