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## The microstructure of some commercial nickel steels

**Robert Newton Stubbs** 

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#### THE MICROSTRUCTURE

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SOME COMMERCIAL NICKEL STEELS

R.N. Stubbs, Jr. April, 1920

Investigations leading to the degree of Bachelor of Science in Metallurgy

Approved:

SN Fasoc, Professor of Metallurgy

TABLE OF CONTENTS

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- (1) Steels Treated
- (2) Heat Treatments
- (3) Etching Reagents
- (4) Physical Tests
- (5) Appendix
- (6) Bibliography
- (7) Photomicrographs

#### THE MICROSTRUCTURE

#### OF

#### SOME COMMERCIAL NICKEL STEELS

Steel A

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:

с,,	, ,		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

Mn.	•	٠	•	•	٠	٠	•	٠	٠	0.40
Ρ.	٠	٠	•	٠	٠	٠	•	٠	•	0.02
s.	٠	•	•	٠	•	•	٠	•	•	0.02
Si.	٠	٠	٠	•	•	•	٠	•	•	0.08
Ni.	٠	٠	٠	٠	٠	٠	•	٠	•	3.5
Cr.	•	٠	•	•	٠	٠	٠	•	٠	0.15

· .

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

Heat Treatments

Specimen l Number	lst Quench fro	om	In	2d Qu	ench :	from	In
l (Original)							
1 (Original) 2	700 degrees	C	Water				
3	<b>7</b> 50  "	Ħ	TT				
4 • • • • •	800 <sup>II</sup>	π	TT				
4 • • • • • 5 • • • • •	850 "	TT	31				
6	<b>9</b> 00 <sup>m</sup>	TT	TT				
7	950 "	TT.	TT				
8	900 "	11	TT	600	degre	es C	Water
1-16	950 "	77	Brine		0		
2-9	950 "	17	Air				
3-10	950 "	17	Oil				
4-11	950 "	Ħ	Furnace				
5-12	900 <sup>m</sup>	TT	Air				
6-13	900 "	<b>11</b> .	Brine				
7-14	850 "	Ħ	Air				
8-15	850 "	11	Brine				
9	900 "	11	Water	200	11	ंग	Ħ
10	900 "	11	- 11	300	11	Ħ	11.
11	900 "	17	11	400	11	T	Ħ
12	900 "	Ħ	17	500	TT	TŤ,	11
$1\tilde{3}$	900 "	TT	TT	600	TT	<b>TI</b>	<b>75</b> .
14	900 1	11	71	650	T	TT	TT
15	900 "	11	11	700	11	11	11
	900 "	17	11	750	TT	Π	11
	1100 "	11	Air	100			
	1100 "	11	Water				
	1100 "	11	Furnace				
	1100-600		t ur nacco	600	room	temp.	TT
	1100 "	11	Brine	000	100m	oomp.	
	1100-850		Furnace	850	11	Ħ	TT
	1100-800		I UTHACE	800	TT	π	π
	1100-750	11	11	<b>7</b> 50	TT	11	۲
	1100-700		11	700	17	Ħ	π
	1100-650		TT	650	11	Ħ,	FT.
	1100-500		TT	500	TT	ŦŤ.	<b>FT</b> .
	1100-500	11	ŧT	500			
	1325 "	11	Water	-			
		11	Air				
	10~0	11					
	1000	11	Water				
	<b>A</b> 000	TT	Air				
	1000	11	Water				
45	1380 "	1	Air				
				<u>m</u>			

4

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 hr. "   30 1100 " 2 hr. "   30 1100 " 3 hr. "   31 1150 " " Water   32 1150 " " Water   33 1200 " Air   34 1200 " Water   35 1250 " Water   36 1250 " Brine   37 1250 " " Brine   38 1265 " " Furnace	Spe Num	cime ber	n	Max	Temp.		Time	Cooled in	
	26 27 28 29 30 31 32 33 34 35 36 37 38			1100 1100 1100 1100 1150 1200 1200 1250 125	ा ग ग ग ग ग ग ग ग ग ग ग ग ग ग ग ग ग ग ग	11 11 11 11 11 11 11 11 11	1 hr. 1 1/2 hr. 2 hr. 2 1/2 "	n n n Water Air Water Air Water Oil Brine	

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 platinumplatinum 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 vels. picris 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 chloride . . . . 5 " Water. . . . . . . . . . 1000 c.c.

Sodium picrate--

Sodium hydroxide . . . 24 gms. Picric acid . . . . . . . 2,0 " Water. . . . . . . . . . . . . . 74 c.c.

## Macroscopic Etching

The best results were obtained by the following solution:

## Physical Tests

Speci- man	Treatment	Ten.Str.	Red Area%	Elong,%	Brinel: No.	1 El. L <b>îm</b> .
l	Original- Rolled and	76,433	51	23,4	196	22 <b>,290</b>
2	annealed Q-water-700	87,040	47	Broke in grips	652	
3	Q-furnace-900	82,802	52	21 <b>.9</b>	228	
4	Q-air-900	89,170	35(?)	Broke in grips	207	12,740
5	Q-furnace-950	81,111	5 <b>1</b>	21.9	228	52,450
6	Q-water-900	159,235	None	None	113	50 <b>,</b> 955
7	Q-air-830	89,170	51	Broke in grips	228	55 <b>,730</b>
8	Q-water-800		break with	lead of l	96 <b>,7</b> 00 j	poun <b>ds</b>
9	Q-air-720	81,580	sq. in. 58	Broke in grips	220	48,800
10	Q-air-950	90,980	5 <b>7</b>	18,7	321	
11	Q-furnace-800	81,100	58	23.4	230	52,450

#### 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", "The structure developed was in all probability as follows: 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 incarbon 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:

0.12%C0.25%C0.10% Ni -pearlite and<br/>ferrite0.9% Ni-pearlite0.9%10-12% Ni Martensite and<br/>ferriteAbove 15% Ni-poly-<br/>hedric7-10%12-27% Ni-Martensite<br/>Above 27% Ni-polyhedricAt

0.8%C O-5% Ni-ferrite 5-7% Ni-Martensite 7-10%Ni-Martensite 10-15% Ni-Martensite and polyhedric Abbve 15%-polyhedric.

1

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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 off plain-carbon steels.

No. 2:

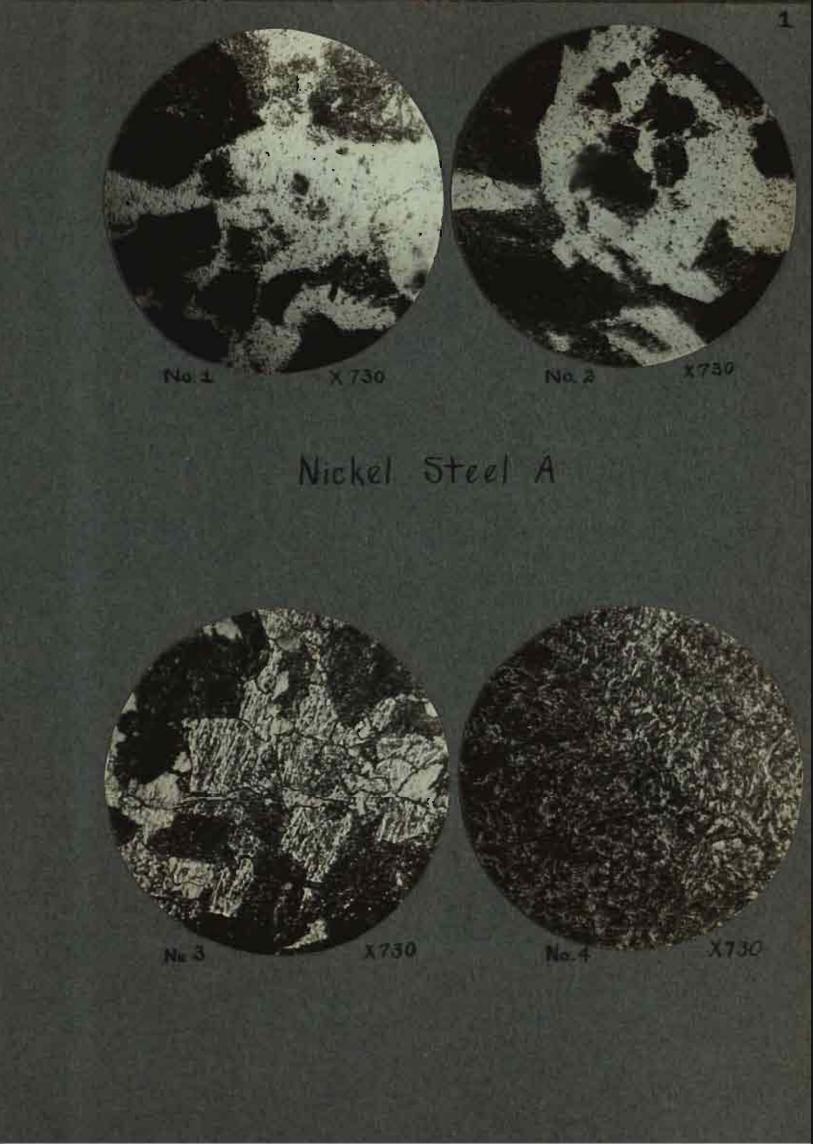
After heating to 700 degrees C and quanching in water. The micrograph shows no evidence of carbon diffusion or grain refinement. This due to the short length of time held at the tmmperature. Pearlite-ferrite structure.

No. 3:

After heating to 750 degrees C and quanching 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. 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.

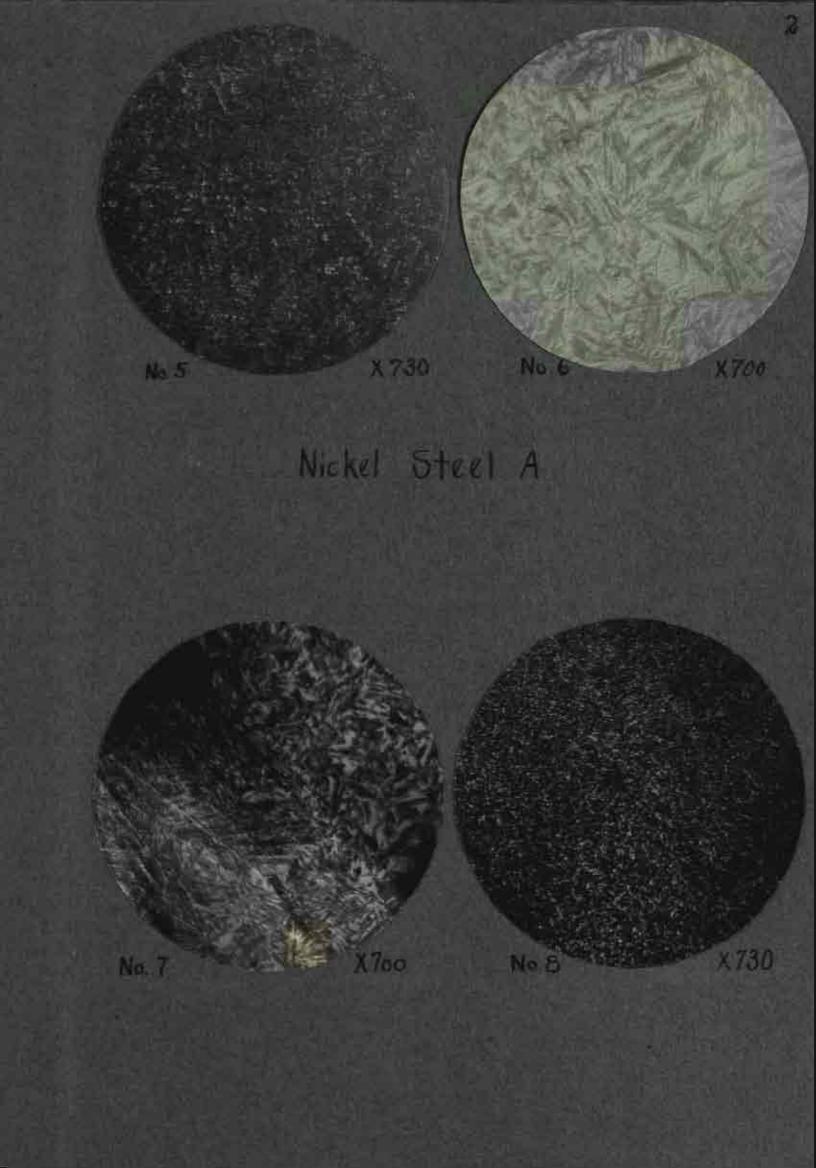
No. 7:

Quenched in water from 950 degrees C. Coarse Martensite.

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. 1-16:

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

No. 2-9:

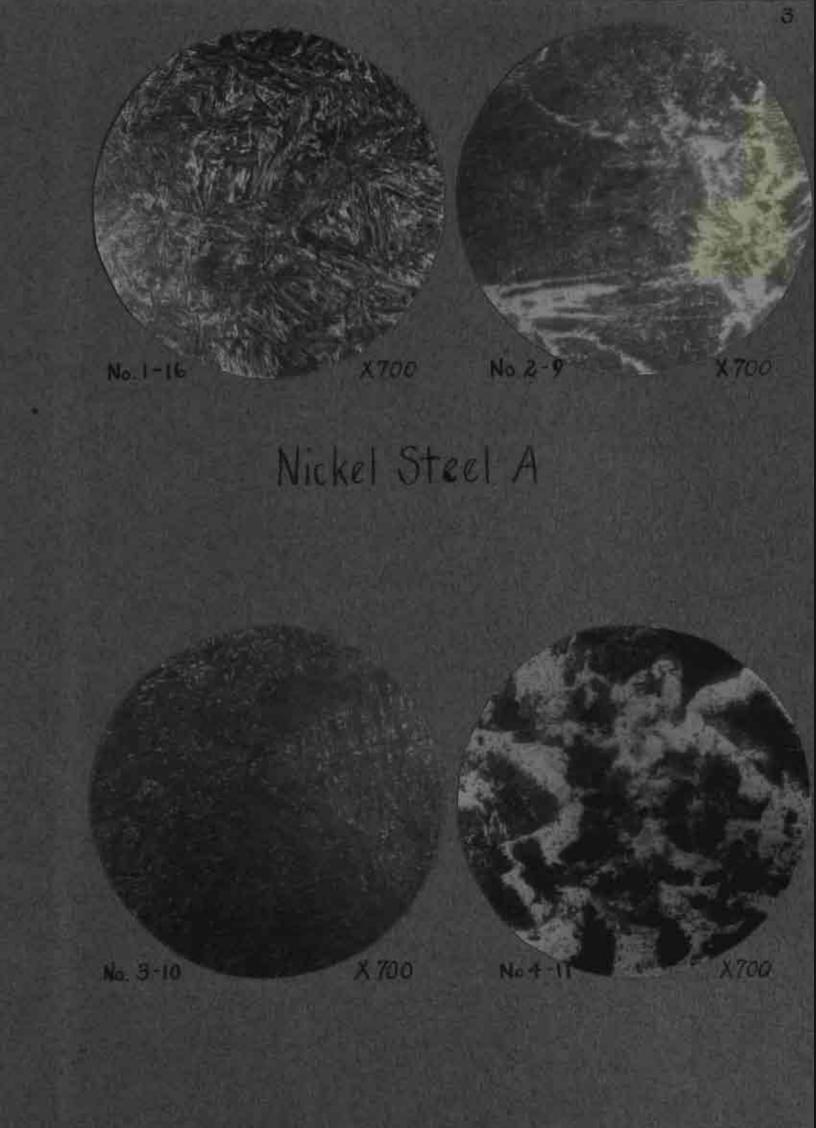
Cooled in air from 950 degrees C. Large pearlite grains with ferrite segregation along boundary lines.

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. 5-12:

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

No. 6-13:

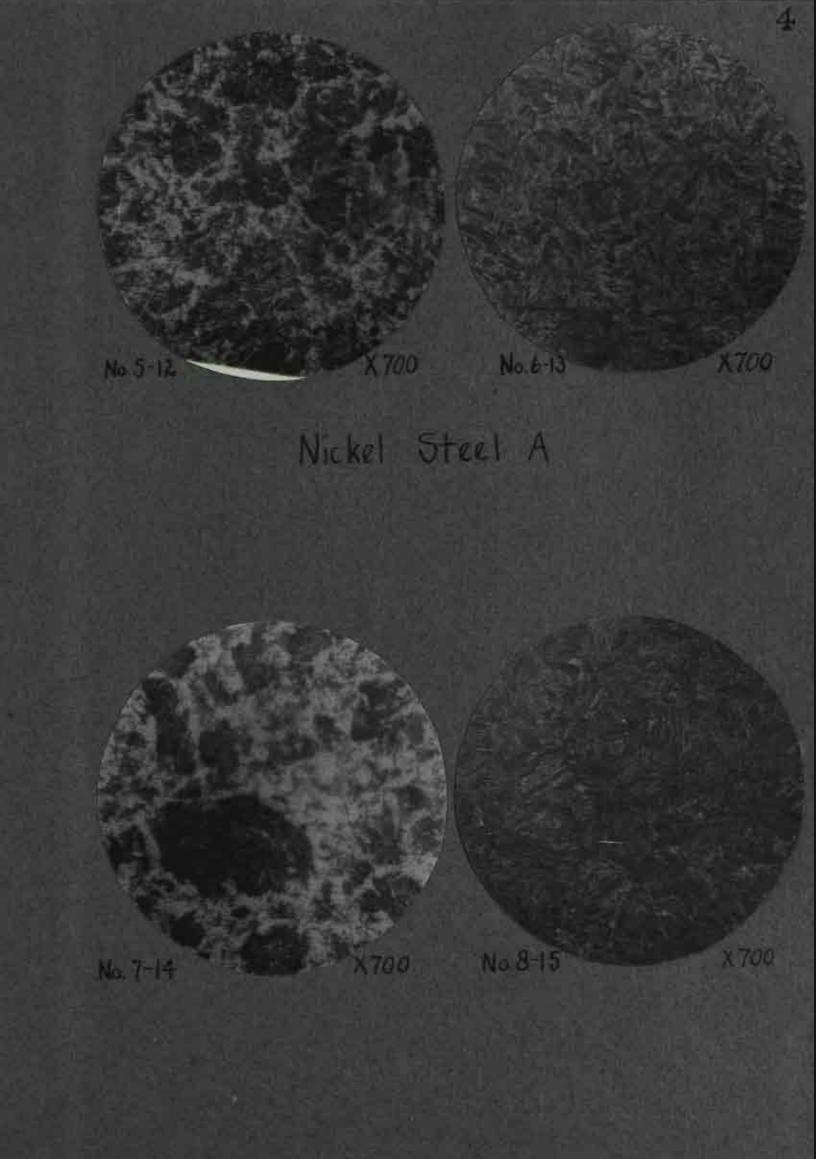
Quenched in ice brine from 900 degrees C, Coarse Martensite.

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. 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.

# Nickel Steel A

X125

125

No.10-26

No. II-3

Na.9-1

No 10-20

5

X700

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 minilar 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.

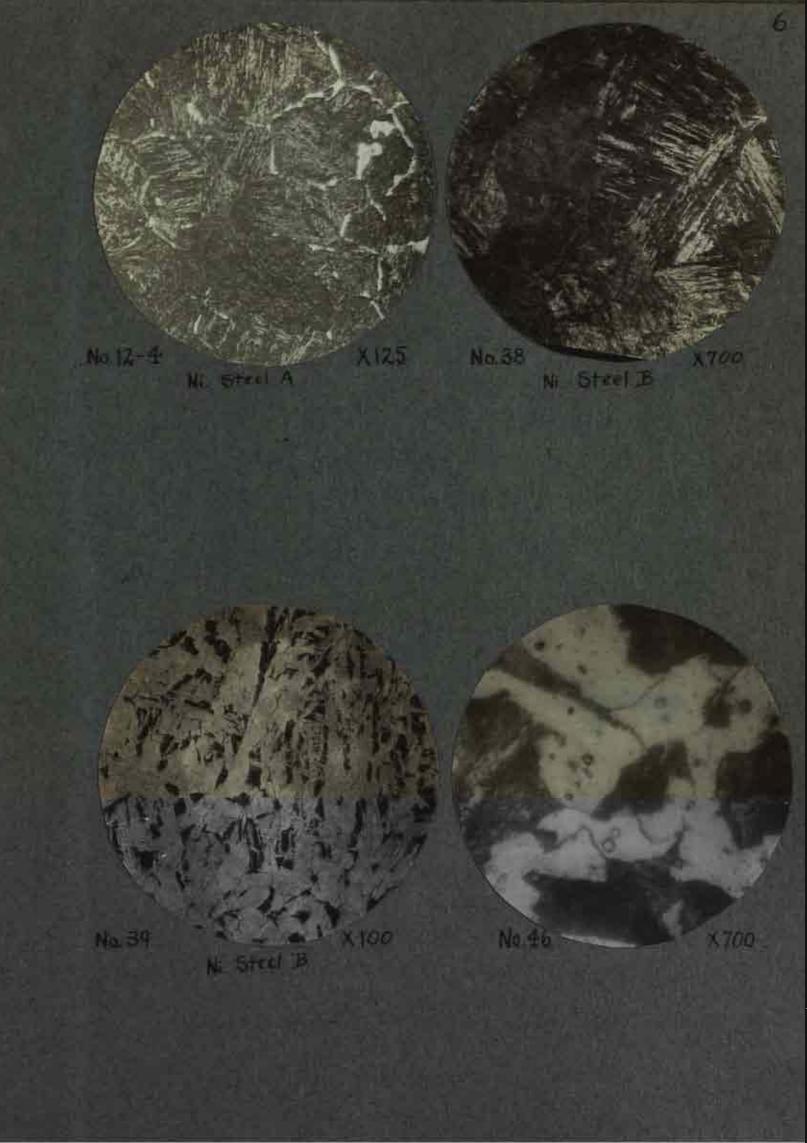
No. 39:

Furnace cooled from 1265 degrees C.Ferrite and pearlite.

No. 46:

Cast nickel steel, ferrite and pearlite.

13



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.

