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Final Report

1947

EFFECT OF NICKEL ON THE CRITICAL SHEAR AND NORMAL STRESSES FOR ALPHA IRON

to

Watertown Arsenal Laboratory
Watertown 72, Mass.

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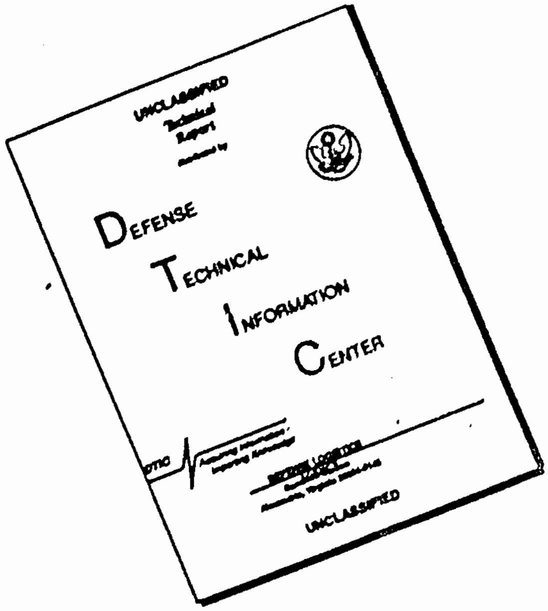

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EFFECT OF NICKEL ON THE CRITICAL SHEAR AND NORMAL STRESSES FOR ALPHA IRON

Final Report

by

I. Cadoff and E. Miller

to

Watertown Arsenal Laboratory
Watertown 72, Mass.

John P. Nielsen
Project Director

Harold K. Work
Director, Research Division

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EFFECT OF NICKEL ON THE CRITICAL SHEAR AND NORMAL STRESSES FOR ALPHA IRON*

Final Report

Object: To determine the variation of critical shear and normal stresses of alpha iron alloyed with 0-3.5% Ni at temperatures ranging from -180°C to +100°C.

Summary: Equipment and processes used in the experimental program concerned with production of the single-crystal specimen required for the analysis of critical stresses is described.

Conclusion: Single-crystal specimens were obtained for pure Iron. Nickel alloys did not respond to strain-anneal treatment for single-crystal growing.

* This work was performed under the technical supervision of Watertown Arsenal Laboratory, Watertown, Mass., and the cognizance of the New York Ordnance District.

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Final Report

TABLE OF CONTENTS

INTRODUCTION

The explanation of the mechanical behavior of metals at low temperatures presents a perplexing problem at present. In particular the loss of ductility, or "transition," which is found to occur in metals that are not face-centered cubic has become the subject of extensive research. In the case of body-centered-cubic ferritic steel, the brittle transition imposes a serious limitation on the application of plain-carbon steel for low-temperature service. Although the factors influencing this behavior are many, it has been reported[1] that nickel in ferritic steel lowers the temperature at which the transition occurs, as well as raising the overall impact resistance of the steel. Increasing the nickel content from 0 to 4% by weight not only increased the impact strength at 0°C and about -50°C but the low-temperature values increased at a faster rate and, for the upper limit of nickel alloying, approached the room-temperature values. For the alloy containing 4% Ni no loss in impact strength was observed at -46°C. This marked effect of nickel on the mechanical behavior of ferritic steel warranted further investigation of the basic nature of the deformation processes in iron-nickel alloys.

A deformation study of iron-nickel alloys for relative slip, twinning and cleavage mechanisms should provide the means of evaluating the role of nickel in the plastic processes in iron. The ductility of a metal is measured by the amount of plastic deformation (which at low temperatures is a result of slip and/or twinning) that the metal can undergo before it fractures. The amount of plastic flow possible is governed in part by the critical resolved stresses which are required to cause shear and cleavage. A qualitative representation of the relation of critical shear and cleavage stresses and the effect of temperature on these stresses is given in

Fig. 1. When the shear stress is less than the cleavage stress, as is the case at high temperature, plastic flow, which is accompanied by strain hardening, results. As shown in the illustration, the critical shear stress and critical cleavage stress both increase with decreasing temperature, the shear stress increasing at a faster rate. The intersection of these two curves would be equivalent to the "transition" point, for at this temperature and temperatures below it, fracture would occur without any plastic deformation.

The discussion presented in the preceding paragraph is very much idealized. Such factors as orientation, rate of loading, and triaxiality of stress were neglected. However, a careful study of single crystals in which these factors are controlled can yield data which will approach the ideal condition. A study of the type undertaken would show whether or not the effect of nickel on the impact resistance was a function of the normal deformation processes.

Since there are many factors involved in the brittle fracture of metals, the experimental program was designed to eliminate or carefully control these variables which may affect accurate measurement of the initial stress variation with nickel content. For this reason, high-purity iron and nickel were used; the stressing unit was designed to eliminate triaxiality and bending due to eccentric loading; and the loading rate was controlled so as to approach static loading.

In outline, the steps in the experimental program are (a) production of suitable single crystals of iron-nickel alloys containing up to 3.5%Ni (b) stressing of these crystals in controlled temperature baths until slip, twinning, and cleavage are observed; (c) correcting the loads at which deformation occurred for orientation relationships of the crystals to obtain the resolved stresses; (d) evaluation of the data obtained to determine the effect of nickel.

I. EXPERIMENTAL PROCEDURE

The experimental program was subdivided into two parts: (A) the preparation of suitable single crystals, and (B) the evaluation of the critical stresses at test temperatures. During the past year work was centered on Part A. Part B could not be carried out until specimens were available.

A. Preparation of Single-Crystal Specimens:

1. Materials:

High-purity iron and nickel were used for the alloy specimens in order to minimize the effects of impurities. Particular care was taken to obtain iron with a minimum of oxygen and carbon since it has been reported[2,3] that small amounts of these two elements cause marked reductions in the impact strength of iron at low temperatures. Brick's data,[4] however, does not confirm this effect for carbon in high-purity alloys. Table I contains a statement of purity of the iron and nickel used and the sources from which the metals were obtained. The purity of the as-received iron was considered high enough to permit its use without additional purification.

2. Melting:

Alloys were melted in a cold-electrode furnace of the type used for titanium melting. The design and operation of these furnaces has been reported on in detail[5] Ingots were prepared in the form of flat slabs as shown in Fig. 2.

Hardness measurements were used as a control guide for contamination during melting. No significant increases in hardness were observed.

The alloy slabs were spot checked for uniformity of nickel content by chemical analysis of samples taken from various points along the length of the slab. In general they were reasonably homogeneous.

3. Forming:

The arc-melted slabs were formed into two shapes suitable for tension testing: a reduced-section, round specimen and a tapered, flat specimen. Figures 3 and 4 illustrate the two types. The round cross-section specimen was obtained by cold forging the flat slab to a cross-section about 1/2 inch square and then turning it down on a lathe. Flat specimens were prepared by cold rolling the slab to a .275in. flat and then milling it to shape.

After machining to final shape, the specimens were annealed to remove any induced strains.

4. Crystal growing:

A survey of methods used for single-crystal production was made. Of the methods available, crystallization from the melt and high-temperature grain growth could not be conveniently used because of the transformations which take place on cooling to room temperature. Strain-annealing, which had been previously used with some success(6) for growing iron single crystals was selected. Cycling about the transformation temperature was considered as a supplementary technique.

Essentially, the strain-anneal method consists of producing a critical amount of plastic strain in the sample, controlled heating at a relatively slow rate to a temperature above the recrystallization range, and annealing at that temperature for the necessary time interval. The strain required, heating rates required, and other factors, as well as results obtained, are discussed in the following section under Data and Discussion.

The critical strain was usually obtained by pulling the specimen in a tensile tester. A photograph of the unit as set up for this test is shown in Fig. 5. The chain assembly is used to minimize eccentric loading and twisting during application of the load. The critical strain was measured by the percentage of reduction in area. In some experiments the strain was induced by bending or compression but the results obtained were not satisfactory.

To prevent oxidation, the strained specimens were sealed in argon-filled quartz capsules prior to annealing. Annealing was carried out in nichrome-wound tube furnaces built with a six-inch constant-temperature heating zone. Controlled heating was obtained by means of a dial-type on-off controller, the dial being rotated with a reduced gear motor drive so that heating rates of the order of 50°C per day were obtained.

Standard etching techniques were used to reveal the grain structure after the treatment was completed.

B. <u>Evaluation of Critical Stresses at Test Temperatures</u>:

The chamber shown in Fig. 7 was designed and built for the purpose of providing constant-temperature baths during testing. It was anticipated that the critical stresses would be determined at several points between 100°C and -180°C. This chamber is assembled on the tensile unit as shown in Fig. 6. The light and microscope ports are included so that the onset of slip can be observed and also to permit motion-picture recording of the progress of slip and fracture.

The stress value recorded by visual observation is resolved into components using the two-surface method of analysis to determine the crystallographic direction of slip. The orientation of the crystal is determined by Laue analysis.

II. DATA AND DISCUSSION

The experimental method used for growing single crystals of iron-nickel alloys was based on that used by Stone [6] for pure iron. His technique consisted of straining 3.5-3.8% (reduction in area produced by tension), heating from 450°C to 900°C at a rate of 50°C per day and holding at 900°C for 100 hours, followed by furnace cooling. The reported success was 30%. It was necessary to modify this procedure to compensate for factors introduced by the nature of the present investigation.

Table II is a summary of the pertinent data obtained for the single-crystal-growing experiments. As indicated, single crystals were obtained for pure iron only.

In general the probability of success of the strain-anneal method is increased if the starting grain size is small (ASTM#6 or smaller is desired), the critical strain is a minimum, and the annealing temperature is close to the melting point of the alloy. The physical nature of the alloys used for this investigation limited the likelihood of attaining these optimum conditions.

The grain size prior to straining could not be kept small, particularly in pure iron. Also, the grain size could not be readily controlled. The as-cast grain size in the ingot was generally greater than ASTM #1. The ingots were cold rolled and annealed for grain refinement, but a maximum of only 20% reduction in area was possible before the specimen became too thin to be used. This 20% reduction was too small to produce grain sizes much smaller than ASTM #2 in pure iron and ASTM #4-5 in alloys. Rapid cycling about the transformation temperature was used to refine the grain size, but the results were inconsistent and unacceptable. The difficulty in obtaining fine-grain specimens may be attributed to the high purity of the iron since grain growth takes place very rapidly in pure metals.

Since control of starting grain size was not feasible without additional processing, it was decided to continue with large-grain specimens. These specimens were found to require higher critical strains for recrystallization. A program for determining the variation of critical strain with grain size was carried out. Experiments in which strain was induced by bending, compression and tension were carried out but significant data were obtained only for the tension experiments. The results obtained from bending and compression experiments indicated that the induced strain was not uniform and the results could not be properly interpreted. The first successful results were obtained in a series of pure iron specimens (Nos. 47, 48, 49, 50) where single crystals were grown with 3.85 to 5.56% strain and starting grain size greater than ASTM #1 with a heating cycle of 50°C per day from 450°C to 890°C. Increasing the nickel content also resulted in an increase in the required critical strain. To determine the critical strain approximately, long tapered specimens were prepared so that on straining, the induced strain would vary along the length. For 3.5% Ni, recrystallization occurred at about 14% strain. For 1% Ni, recrystallization occurred at about 10% strain and for an alloy containing 2% Ni the critical strain was found to be about 11%. The high values of critical strain do not seem to be advantageous for single-crystal growing. In addition to raising the critical strain, the addition of nickel also lowers the annealing temperature. The upper limit of annealing temperatures is the transformation zone for iron-nickel alloys. The transformation process results in a recrystallization so that the strain-annealing treatment for single crystals cannot cross over into the austenite region. Figure 8 is a reproduction of the iron-nickel phase diagram in the region of interest. As shown, the slope of the A_3 line is quite pronounced, so that for 3.5%Ni the maximum annealing temperature available is only 600°C.

To overcome this difficulty several attempts were made to produce single crystals of high-nickel alloys by prolonged heating, alternately above and below the transformation range, but with little success.

The series of flat specimens, Fig. 3, did not prove as useful as had been hoped since the strain pattern produced was not uniform. The flat surface in contact with the grips had absorbed higher strain, and recrystallization was found to occur on the surface but not in toward the central portion. Round specimens give uniform stress distributions but require a machining time.

The lack of suitable single-crystal specimens has held up the work on low-temperature measurements.

III. SUMMARY

The experimental program aimed at producing single crystals for the study of the critical stresses has met with partial success. Single crystals were obtained for pure iron, but the addition of nickel adversely affects the factors favoring a low nucleation rate and rapid grain growth.

The addition of nickel to iron causes an increase in the critical strain required for recrystallization and in addition, lowers the temperature available for annealing.

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(6) Stone, F. G. "Production of Ferrite Single Crystals," Technical Note No. 3, Metals Technology, June 1948.

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LIST OF TABLES AND ILLUSTRATIONS

TABLE I

Analysis of Materials Used in the Production of Ingots

Impurities -- Weight Percent

Iron: Ferrovac-E

Heat No.	C	O_2	N_2	Al	Co	Cu	Mn	Ni	Pb	Si	Sn	Zn	Fe	S
1 P 231	.005	.0052	.00013											
1 P 515	.0031	.0072	.00051	.003	.006	.001	.001	.01	.01	.01	.005	.003		

Nickel: Mond Pellets

Heat No.	C	O_2	N_2	Al	Co	Cu	Mn	Ni	Pb	Si	Sn	Zn	Fe	S
	.040					.003							.025	.00[illegible]

TABLE II

SUMMARY OF EXPERIMENTAL DATA

Specimen Ident.No.	Type	Comp. Wt%Ni	Initial Grain Size	% Red. in Area	Heat-Treatment Cycle	Remarks
1	round	0	1-3	2.6	310 to 600°C at 50°C/day	Final Grain Size ~ ASTM #1
2	round	0	2	3.4	interrupted, 540 to 890°C	After final anneal specimen
3	round	0	1-4	2.5	at 50°C/day, to 42 hr at 890°C, F.C.	had varying grain size indicating non-uniform load. Appeared very fine grained.
4	wire	0	6	3	96 hr at 880°C, F.C.	Strain not homogeneous-due to bending of wire.
5	wire	0	6	3	96 hr at 880°C, F.C.	Strain not homogenous-due to bending of wire.
6	round	2	6-7	2.84	430 to 700°C at 50°C/ day, F.C.	Grain Size varied throughout specimen ~ ASTM #6 - #2
7	comp.	0	1-2	6.9	16 hr at 650°C, F.C.	fine grained
8	comp.	0	1-2	2.59	16 hr at 650°C, F.C.	fine grained
9	comp.	0	1-2	2.44	16 hr at 650°C, F.C.	fine grained
10	comp.	0	1-2	1.43	16 hr at 650°C, F.C.	unchanged
11	comp.	0	1-2	1	16 hr at 650°C, F.C.	unchanged
12	round	0	1	2	80 hr at 880°C, A.C.	unchanged
13	round	1	2-4	2.2	485°C to 805°C, at 50°C/	unchanged
14	round	1	?	2.4	day, 96 hr at 805°C, F.C.	unchanged
15	round	1	2-4	4.1	96 hr at 800°C, F.C.	unchanged
16	round	1	4-5	4.98	96 hr at 800°C, F.C.	unchanged
17	round	2	3-4	3.3	72 hr at 700°C, F.C.	Small grained except for large grained section near a shoulder
18	round	0	1	3.38	450°C to 875°C at 50°C/day, 72 hr at 875°C, F.C.	Large grained at section between center of gage section and shoulders. Rest of Specimen ~ #1.
19	comp.	0	1-2	3.27	54 hr at 890°C, F.C.	Most of specimen #1, with some very large grains.

SUMMARY OF EXPERIMENTAL DATA

TABLE II (continued)

Specimen Ident. No.	Type	Comp. Wt %Ni	Initial Grain Size	% Red. in Area	Heat-Treatment Cycle	Remarks
20	comp.	0	1-2	3.71	54 hr at 890°C, F. C.	Mainly #1 grain size, some very large grains.
21	comp.	0	1-2	3.84	54 hr at 890°C, F. C.	Slightly smaller grain size than 19 and 20. ASTM #2, no very large grains.
22	comp.	0	1-2	2.31	54 hr at 890°C, F.C.	Mixed grain size. Some huge grains, some #3-4
23	comp.	0	1-2	2.17	54 hr at 890°C, F.C.	Most of specimen #1, some very large grains
24	comp.	0	1-2	4.02	54 hr at 890°C, F.C.	Most of specimen #1, some very large grains
25	comp.	1	5	2.8	54 hr at 800°C, F.C.	Specimen had elongated grains #3-4-grains grew
26	comp.	1	5	1.28	54 hr at 800°C, F.C.	Equiaxed structure #5, unchanged.
27	comp.	1	5	4.23	54 hr at 800°C, F.C.	#4, unchanged
28	comp.	1	5	4.7	54 hr at 800°C, F.C.	Elongated grains #3-4, grain growth.
29	comp.	1	5	10.56	54 hr at 800°C, F.C.	Very large grained compared to others #2
30	comp.	2	5	1.9 - 3.5	54 hr at 710°C, F.C.	unchanged
31	comp.	2	5	3.34	54 hr at 710°C, F.C.	unchanged
32	comp.	2	5	3.36-12.8	54 hr at 710°C, F.C.	unchanged
33	comp.	3.5	5	3.73	54 hr at 620°C, F.C.	unchanged
34	comp.	3.5	5	2.35-3.57	54 hr at 620°C, F.C.	unchanged
35	comp.	3.5	5	2.15-3.8	54 hr at 620°C, F.C.	unchanged
36	round	3.5	6-7	4.4	96 hr at 620°C, F.C.	unchanged
37	round	3.5	6-7	4.2	96 hr at 620°C, F.C.	unchanged
38	sheet	3.5	8	3.4	50 hr at 600°C, F.C.	unchanged
39	sheet	3.5	8	2.7	50 hr at 600°C, F.C.	unchanged
40	round	3.5	6-7	6.15	50 hr at 600°C, F.C.	unchanged
41	round	3.5	6-7	4.55	50 hr at 600°C, F.C.	unchanged
42	sheet	0	4-5	5.02	50 hr at 900°C, F.C.	Same grain size after annealing but grains appeared elongated in direction of rolling.

SUMMARY OF EXPERIMENTAL DATA

TABLE II (continued)

Specimen Ident No.	Type	Composition Wt% Ni	Initial Grain Size	% Red in Area	Heat Treat. Cycle	Remarks
43	sheet	0	3-5	3.19	50 hr at 900°C, F.C.	All specimens appeared same grain size after annealing as before, but grains to have elongated in the direction of rolling.
44	sheet	0	5	4.55	50 hr at 900°C, F.C.	"
45	sheet	0	3-6	3.3	50 hr at 900°C, F.C.	"
46	tapered to gage length	0	1 at 50x mag.	3.3	450 to 890°C at 50°C/day, then 48 hr at 890°C, F. C.	unchanged
47	round	0	"	6.04		very large grained
48	round	0	"	3.84		Single crystal in 1/2" gage section.
49	round	0	"	5.56	96 hr at 890°C, F.C.	Single crystal in section between center of gage section and shoulder
50	round	0	"	5	96 hr at 890°C, F.C.	Very large grains.
51	round	3.5	5	6.5	96 hr at 600°C, F.C.	unchanged
52	round	2	1	6.1	96 hr at 720°C, F.C.	unchanged
53	tapered	1	5	0-11	1 hr at 800°C, A.C.	Large grains appeared in region where strain estimated to be 7-11%. Rest of specimen remained unchanged.
54	"	1	5	0-8	1 hr at 800°C, A.C.	Specimen necked down beyond 8-1/2% strained region. Specimen did not appear recrystallized.

[illegible] (Continued)

Specimen Ident. No.	Type	Composition Wt % Ni	Initial Grain Size	% Red in Area	Heat Treatment Cycle	Remarks
55	tapered	3.5	7	0 20	24 hr at 600°C, F.C.	Specimen recrystallized between 10-15%. Remainder of specimen fine grained.
56	"	2	5	0 9	16 hr at 700°C, F.C.	Specimen necked down rapidly at about 9% reduction, unrecrystallized.
57	square	1	2	8.8	595 to 790°C at 50°C/ day then 48 hr at 790°C, F.C.	Unrecrystallized
58	"	1	1	9	"	"
59	"	1	1	10.5	"	Large grains in gage lengths all four sides.
60	"	1	1	12.3	"	"
61	"	1	1 at 50x	11.5	"	Small grained center gage section, large grained in tapered section.
62	"	1	2	8.5	"	Large grained on ungripped sides, fine grained on gripped sides of specimen.
63	"	2	5	11.7	96 hr at 700°C, A.C.	Large grained on ungripped sides.
64	"	2	1 at 50x	7.4	"	Unrecrystallized
65	"	2	3	12.7	"	Fine grained. Specimen appeared bent and was not axially loaded.
66	"	2	2	14.4	540 to 700°C at 50°C/ day, then 72,hrs at 700°C, F.C.	Fine grained with several larger grains in non-gripped surface. Tested in non-axial grips.
67	"	2	5	14	"	Fine grained section with large grains in tapered section of gripped surface.
68	"	2	3-4	12.4	"	Large grained on ungripped sides.
69	"	2	4-5	10	"	Center section large grained on all four sides.

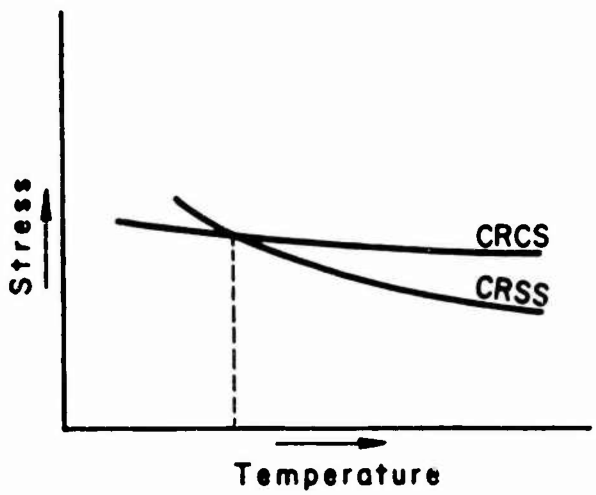
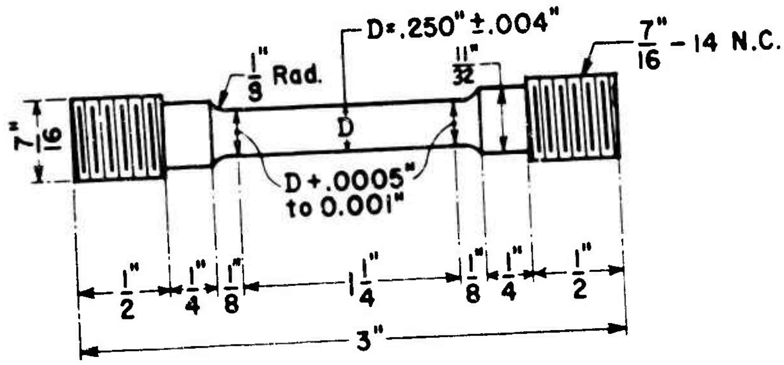


Fig. 1 Qualitative Representation of Critical Stresses Versus Temperature.

Fig. 2 Ingot Produced by Arc Melting



ROUND TENSILE SPECIMEN

Fig. 3 Scale Drawing and Photograph of Round Tensile Specimen.

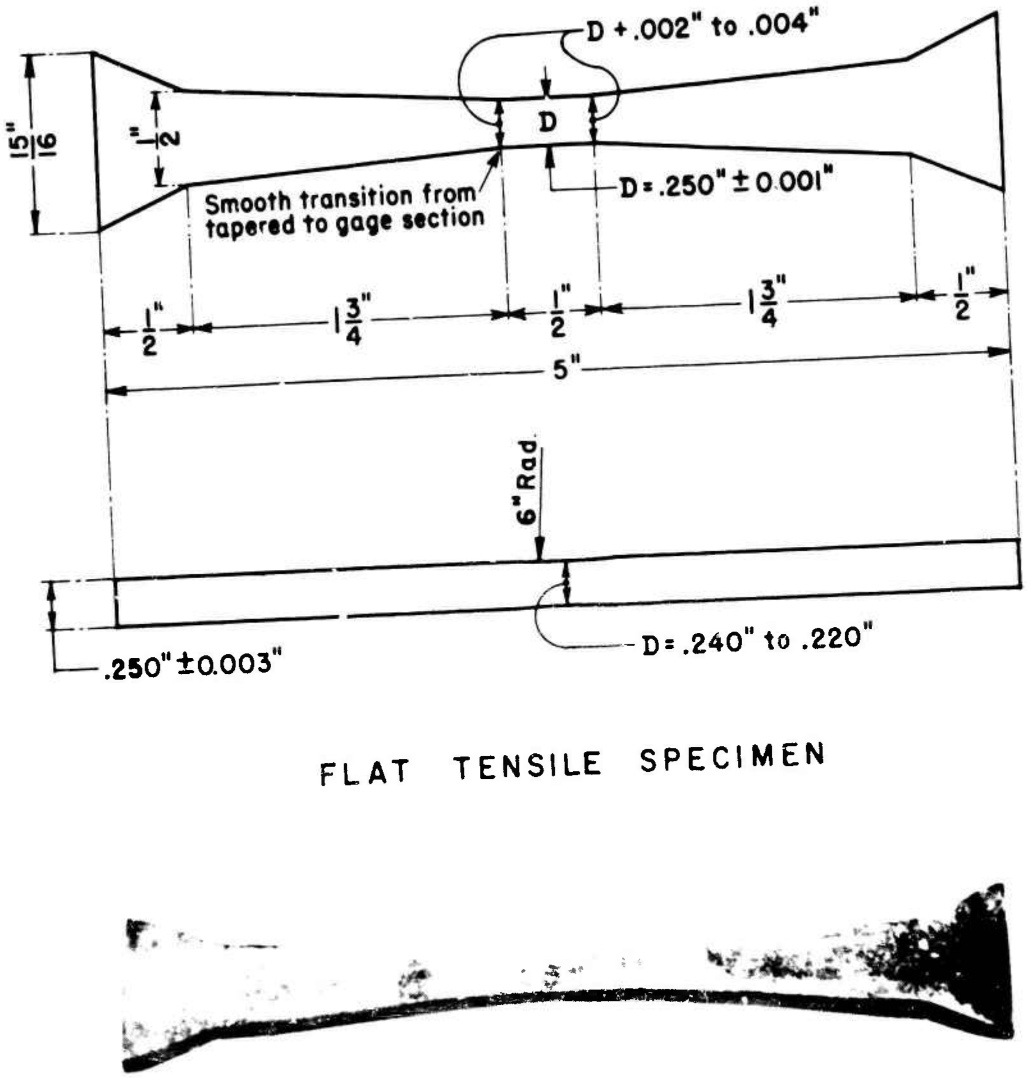


Fig. 4 Scale Drawing and Photograph of Flat Tensile Specimen.

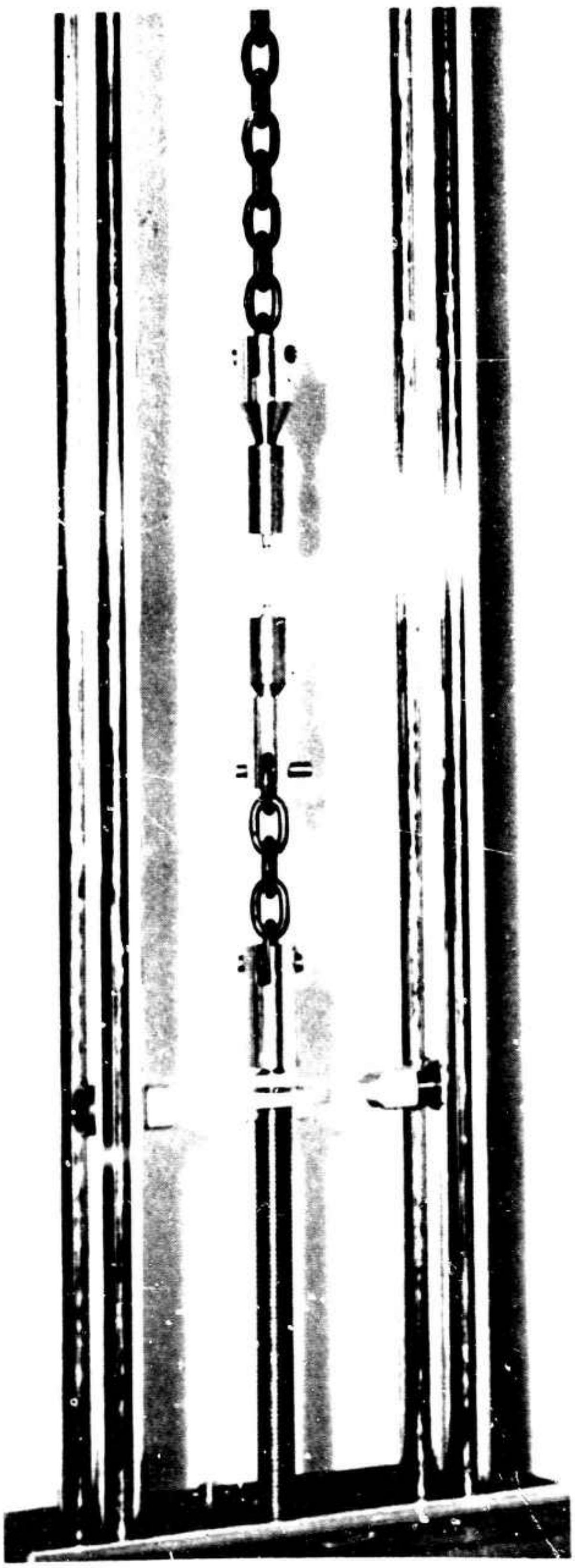


Fig. 5 Round Specimen Mounted in Tensile Machine Prior to Critical Straining

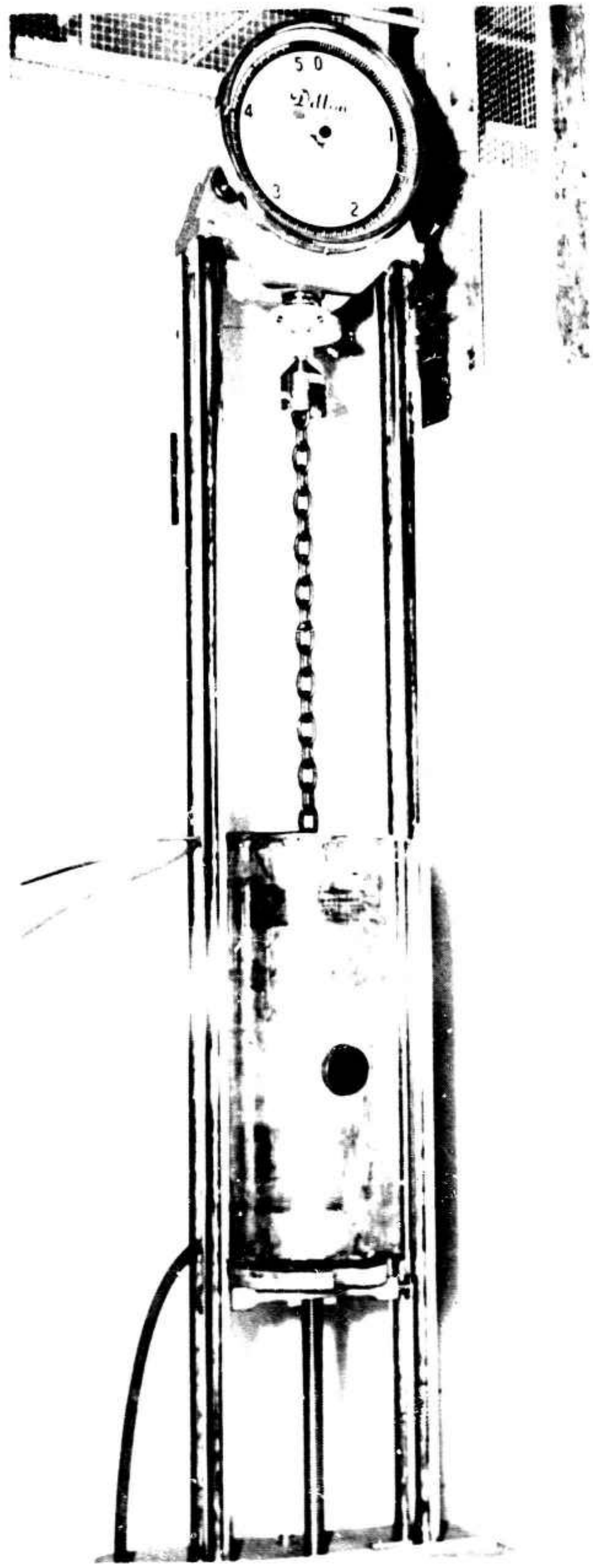


Fig. 6 Low-Temperature Tensile-Test Apparatus

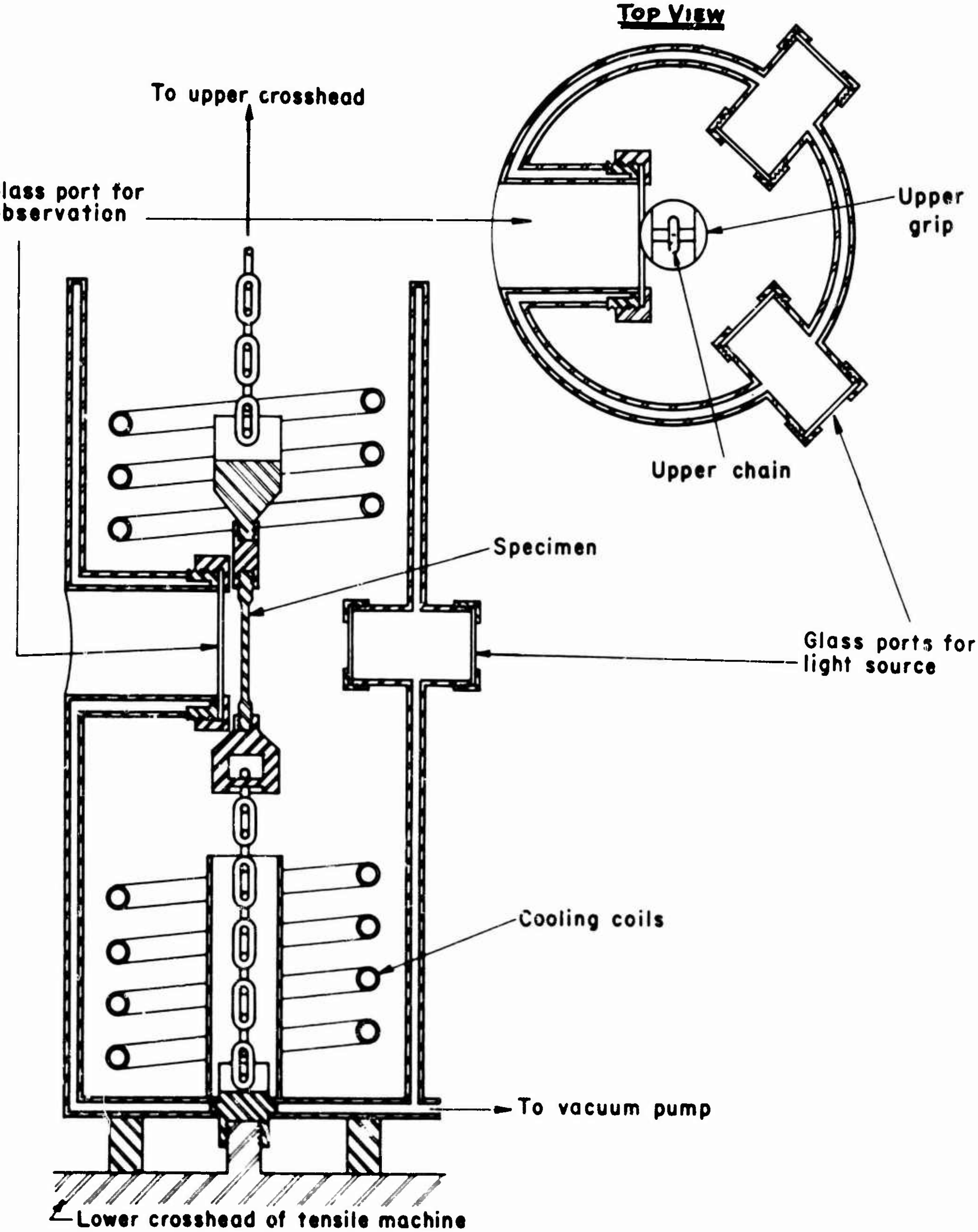


Fig. 7 Schematic Diagram of Low-Temperature Tensile Test Apparatus

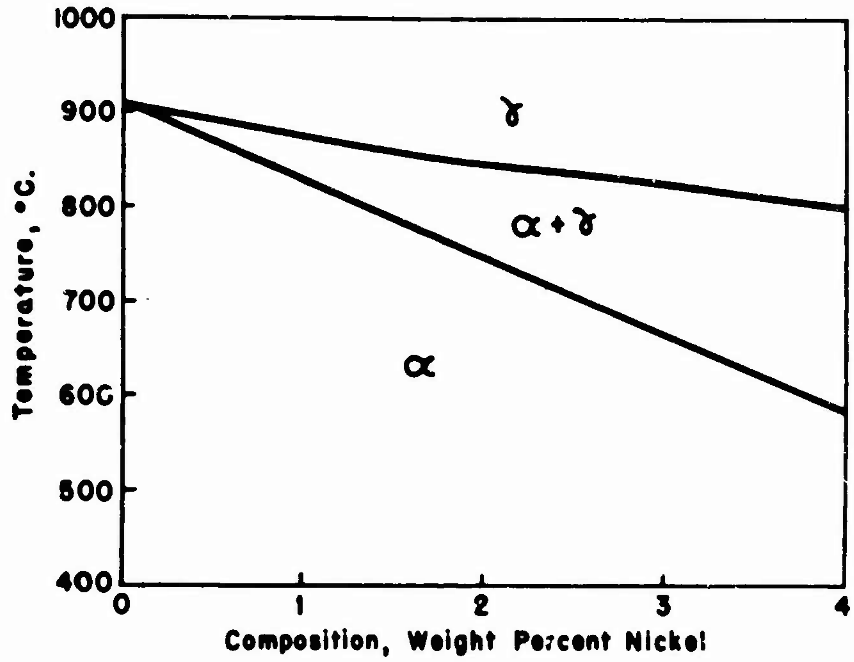


Fig. 8 Iron-Nickel Phase Diagram (Partial)
(Metals Handbook 1948 Edition)