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TECHNICAL REPORT

EVALUATION OF THE EFFECT OF COLD WORK ON DISPERSION-STRENGTHENED NICKEL-BASE ALLOYS

by

R. C. Nelson and R. Widmer

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-7265

TELEDYNE MATERIALS RESEARCH COMPANY

A TELEDYNE COMPANY

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SUMMARY REPORT

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April, 1967

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EVALUATION OF THE EFFECT OF COLD WORK ON DISPERSION-STRENGTHENED NICKEL-BASE ALLOYS,

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ABSTRACT

An evaluation of the effect of cold work on nickel-base alloys containing thorium dioxide was undertaken.

Drawing and swaging techniques were used to introduce the cold work to these 80% nickel/20% chromium-base alloys. Swaging was performed with 10%, 15% and 20% reductions of area per cycle. Intermediate anneals at 1600°F and 2000°F were used to complete the working cycle. Sound material was obtained after extensive amounts of working.

Elevated temperature tensile strengths were generally improved by introducing cold work.

FOREWORD

This report covers the work performed under NASA Contract NAS 3-7265 during the period from 13 June 1966 to 13 December 1966.

This contract was initiated between the NASA Lewis Research Center, Cleveland, Ohio and the New England Materials Laboratory, Inc., (now Teledyne Materials Research Co.) for the "Evaluation of the Effect of Cold Work on Dispersion-Strengthened Nickel-Base Alloys." Technical direation was supplied by the Project Manager, Mr. F. H. Harf of the Lewis Research Center, Airbreathing Engines Division, Dr. T. P. Herbell of the Materials and Structures Division was the NASA Research Advisor.

Mr, Richard C. Nelson of the New England Materials Laboratory (now Teledyne Materials Research Co.) was Project Engineer in charge. Dr. Robert Widmer was the Program Manager. Technical advice and assistance was provided by Drs. N. J. Grant and Allan S. Bufferd. Messrs. Allan R. Runge, David Kushinsky and Miss Ursula Jahnigen assisted in the execution of the experimental work.

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EVALUATION OF THE EFFECT OF COLD WORK ON DISPERSION-STRENGTHENED NICKEL-BASE ALLOYS.

by

R. C. Nelson and R. Widmer

TELEDYNE MATERIALS RESEARCH CO.

SUMMARY

Studies of the 80% nickel - 20% chromium - thorium dioxide system to determine the effect of cold work on mechanical properties were undertaken. Two alloys containing 2.5 v/o thorium dioxide and one containing 5.0 v/o of the same oxide were evaluated.

Swaging was the most successful method of introducing cold work. Tensile strength at 1800°F for a 2.5 v/o thoria alloy was increased from 6,060 psi to 12,900 psi by swaging 4 times with 10% reduction steps. The alloy containing 5.0 v/o thoria had a maximum tensile strength of 10,600 psi at 1800°F after eight passes of 20% reduction steps versus 7,190 psi in the as extruded condition. Yield strength and ductility were generally adversely affected by cold work.

There was no correlation between mechanical properties and the reduction of area per cycle in the swaging operations. The mechanical properties of the alloys were not affected by varying the annealing temperature 1600°F or 2000°F between swagings.

Improvements in stress rupture properties were marginal.

Hardness measurements of the swaged alloys after exposure at 1500°F, 1800°F and 2200°F indicated that the alloys were stable under these conditions since no appreciable hardness drop occurred.

Oxidation resistance at 1800°F, 2000°F, 2200°F and 2400°F of the alloys in the as extruded condition was determined and compared with Nichrome. The alloys are significantly more oxidation-resistant than commercial Nichrome at all temperatures.

I. INTRODUCTION

The mechanical properties of several metal- metal oxide systems have been evaluated after the introduction of cold work to extruded structures.[1,2,3,4,5] An improvement in properties was observed by most of the above-referenced investigations. The improvement wag achieved by various working cycles. A cycle is defined as a reduction followed by an anneal. The annealing temperature is generally higher than the recrystallization temperature of the matrix but less than the recrystallization temperature of the metal- metal oxide alloy. The properties of the alloys are dependent on the number of working cycles, the amount of cold work per cycle, and the annealing temperature.

The effect of cold work on elevated tensile properties has been shown by Fraser et al of Sherritt Gordon Mines Limited.[4] The material was nickel-3 volume percent thorium dioxide. After 20 cycles of 10% cold work per cycle with intermediate anneals at 2200°F, the 1600°F tensile strength increased from 17,000 psi in the as-extruded condition to 27,000 psi.

However, it should be pointed out that Inman, M. C, et al [7] report only marginal changes in properties through cold work.

The present evaluation was carried out with three 89% nickel-20% chromium (Nichrome) alloys containing thorium dioxide as a dispersoid. These alloys were prepared under a previous program at New England Materials Laboratory (now Teledyne Materials Research Co.) and are identified as 86, #6A and f7. Alloy #6 and #6A contain 2.5 $^{V}/o$ thorium dioxide while Alloy #7 contains 5 $^{V}/o$ thorium dioxide. The oxide was introduced into these alloys by the thermal decomposition of thorium nitrate onto 1-micron Nichrome powder.

Alloys #6 and 87 were treated with carbon to reduce the matrix oxygen content, Alloy #6A was consolidated without any treatment to remove oxygen. Pertinent data of the alloys in the as-extruded conditions are recorded in Table I.

II. EXPERIMENTAL WORK

Cold Drawing

It was first attempted to introduce cold work into the alloys by drawing. Only Alloy #7 was used in these experiments.

The rods were drawn at a speed of one inch per minute through a series of tungsten carbide dies that permitted 20% reductions between successive steps. Molybdenum disulfide, both in aerosol and flake form, was used as a lubricant. The disulfides were obtained from Bemol, Inc. of Newton, Massachusetts. Following each drawing the rod was annealed at 1600°F for one hour.

In the two attempts to draw Alloy f7, the rods broke during the second 20% reduction cycle. Apparently this reduction was too severe. Therefore further drawing was abandoned.

It was thought that 10% reductions would yield sound material. But dies of the appropriate sizes were not available and would have required several months for delivery.

Cold Swaging

20% reduction

Swaging was used as a second method of introducing cold work into the alloys. Alloy #7 was the first alloy of the three to be subjected to cold work by swaging. The alloy was reduced 20% per pass and was annealed 1/2 hour at 1600°F between swagings. Eleven swaging steps were performed and resulted in sound material. The core diameter was reduced from 0.39 to 0.076 inches.

Nuclear Metals, Inc., a division of Whittaker Corp., Concord, Mass. performed this swaging. Rockwell A hardness values are recorded in Table III and shown in Figure 1. The measurements were made after each swaging and after each annealing cycle to give an indication of the stability of the alloy following the cold work.

15% reductions

Alloy #7 was also subjected to cold work by swaging in which the cross-section area was reduced 15% per pass. The effect of intermediate anneal temperature was studied. Part of the alloy was subjected to anneals at 1600°F; another portion at 2000°F. Swaging of the alloys in 15% reduction steps was performed by Metalonics, Inc., a division of Kawecki Chemical Co., South Boston, Massachusetts. The hardness values are listed in Table III and shown in Figure 2. The material was cracked after seven swagings, regardless of annealing temperature.

Alloy #6 was subjected to swaging with 15% reductions per cycle. A portion was annealed at 1600°F, while another was annealed at 2000°F. The hardness values obtained in the course of these experiments are reported in Table IV and Figure 3. This alloy cracked after seven swagings, under both sets of annealing conditions.

10% reductions

At Nuclear Metals swaging of the alloys in 10% reductions per cycle was accomplished on alloys #6, #6A and #7. A 1/2 hour anneal at 1600°F was used. Sixteen swagings were performed on alloy #7, while only four were possible with alloys #6 and #6A. At this point alloys 116 and #6A cracked. Hardness values are recorded in Tables V and VI.

III. EVALUATION OF PROPERTIES

A. Microstructure,

The microstructure of the alloys in the as-extruded condition was examined at 100X, 1000X and 20,000X by optical and electron microscopy techniques. The microstructures of longitudinal sections of alloy #6 are shown at 100X and 1000X in Figure 4. The same alloy at 20,000X is shown in Figure 5, while transverse sections at 100X, 1000X, and 20,000X are shown in Figure 6 and 7. The structures of alloys #6A and #7 are shown in Figures 8-15 with the same magnifications. All alloys have a rather non-uniform structure. The size of the oxide particles varies between several tenths of a micron and 1 micron. The interparticle spacing is within the range of 1-5 microns. Alloy #6A appears to have a finer oxide dispersion. This alloy was not vacuum-carbon reduced, and some of the oxide particles present are probably chromium oxide.

B. Oxidation Resistance

Oxidation resistance of the alloys as well as of Nichrome was determined by exposing samples in air at various temperatures. Weight changes after 1, 10 and 100 hours at 1800°F, 2000°F, 2200°F and 2400°F were determined. Specimens were placed in platinum boats, exposed at the temperatures for 1 hour,. cooled, and weighed. The same specimens were then similarly treated for a total of 10 hours at the same temperatures and subsequently for 100 hours. A different specimen of each alloy was used for each temperature.

The depth of penetration was measured by mounting in bakelite the above specimens after the 100-hour exposures. The specimens were polished and a measuring eye piece was used to determine the depth of oxide penetration.

The changes in weight are recorded in Table VII while the penetrations of oxide are reported in Table VIII and represented in Figures 16–20.

The oxidation resistance of the alloys compares favorably with that of Nichrome. The data indicate that commercial Nichrome was generally more susceptible to oxidation at all conditions of testing. The Nichrome used in these experiments was obtained from Driver–Harris Corp., Harrison, New Jersey and it had the following composition:

Chromium	19.5 %
Silicon	1.45%
Carbon	0.03%
Nickel	balance

C. Stability Tests

Hardness measurements of the swaged alloys were made after various amounts of cold work and subsequently after exposure of the alloys for 1 hour and 5 hours at 1500°F, 1800°F and 2200°F in air. These measurements, recorded in Table IX, indicate that the alloys were stable under the conditions tested, as no appreciable hardness drop occurred.

D. Tensile Tests

The tensile properties of the swaged alloys were evaluated at room temperature, 1800° and 2000°F and are recorded in Table X.

The beneficial effect of cold work on the elevated temperature tensile properties of these alloys is shown if one examines, for example, the 1800°F data on alloy #6A. The tensile strength has increased from 6,000 to

13,000 psi as a result of 4 swaging cycles of 10% reduction steps with 1600°F intermediate anneals.

A similar, but less dramatic, improvement in tensile properties at room temperature and at 1800°F was observed with alloy f7 when 20% reduction steps were used. However, as the number of cycles increased beyond eight, the tensile strength decreased, and after eleven swagings it was essentially that of the as-extruded alloy.

E. Stress Rupture Tests

Stress rupture properties of the alloys after varying amounts of cold work were determined and are presented in Table XI.

The data indicate less improvements in stress rupture strength than in tensile strength.

IV. CONCLUSIONS AND RECOMMENDATIONS

The results illustrate that a dispersion-strengthened structure such as alloys #6, #6A and #7 cannot, or at least not easily, be brought to a high creep strength level through thermomechanical treatment. A material with better structural parameters in the as extruded condition responds very much better to the same type of swaging and annealing cycles. (This was demonstrated with some alloys prepared on an in-house budget. See Appendix.) For these reasons it would be inappropriate to make generalized conclusions on the basis of the results on the three alloys discussed in this report. However, the following recommendations can be made for future work:

1. Several alloys (among the ones listed below) should be selected. Enough material should be prepared by the process briefly described in the appendix of this report (Halide diffusion process) and a thorough evaluation of the influence of the thermomechanical treatments on the mechanical properties of the materials should be made. Answers to the following questions should be obtained:

   a. If swaging is used, what reduction steps are most efficient?

   b. What is the number of swaging cycles for optimum properties?

   c. Should annealing be applied after each cold work cycle and at what temperature and what time?

   d. Can cold swaging, plus annealing, be replaced by warm working through forging, warm swaging or extrusion? It is, of course, of particular interest to decrease the number of cycles now used in thermomechanical treatments of dispersion-strengthened alloys.

2. Matrices to be considered for this type of an effort are for instance:

60Ni	20Cr	20W		
74Ni	20Cr	4Mo	2Nb	
57Co	18Cr	15W	10Ni	
59Ni	16Cr	16Mo	5Fe	4W
62Ni	15Cr	18Co	5Mo	
58Ni	20Cr	14Mo	8W	
65Co	20Cr	15W		
80Ni.	20Cr			

3. A better correlation should be established between structural parameters, mechanical properties and processing variables. Transmission electron microscopy will be required for this effort.

4. Attempts should be made to obtain high density powder compacts with a large diameter, which would allow more reduction via cold and/or warm work. It is proposed that a study in isostatic hot pressing be done with this goal in mind. The influence of the pertinent variables - temperature, pressure, dimensions - on the structural parameters has to be studied.

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TABLE I

DATA OF ALLOYS IN THE AS EXTRUDED CONDITION

Alloy	Length, Inches	Core Diameter, Inches	$\%O_2$ [a]	% C	1800°F Tensile Data			
					UTS, ksi	0.2%Y.S., ksi	e, %	R.A.%
#6	29	0.39	0.53	0.03	8.0	7.5	5.3	5.3
#6A	12	0.36	1.00	0.10	6.1	5.1	4.4	5.1
#7	08	0.34	0.94	0.04	7.2	6.5	8.6	10.1

[a] Vacuum fusion technique
Oxygen in 2.5 v/o thorium oxide is 0.367.%

TABLE II

HARDNESS OF ALLOY #7 AFTER DRAWING

Condition	Hardness, Rockwell A[a]	
	Bar 1	Bar 2
As extruded	63	63
20% reduction	68	68
Anneal 1 hr. @ 1600°F	61	60
20% reduction	66	67
Anneal 1 hr. @ 1600°F	60	59

[a] Average of at least nine determinations

TABLE III

HARDNESS OF ALLOY #7 AFTER SWAGING

(20% and 15% reductions per cycle)

Condition	Hardness, Rockwell A [a]		
	20% Reduction 1600°F Anneal	15% Reducfion 1600°F Anneal	15% Reduction 2000°F Anneal
As extruded	63.0	63.0	63.0
First swage	68.3	64.8	64.5
Anneal 1/2 hour	60.9	63.9	62.9
Second swage	67.0	67.9	67.7
Anneal 1/2 hour	59.7	60.1	60.2
Third swage	66.7	67.4	67.7
Anneal 1/2 hour	61.0	60.0	60.0
Fourth swage	66.4	66.5	66.6
Anneal 1/2 hour	60.4	65.0	60.0
Fifth swage	65.9	67.9	67.4
Anneal 1/2 hour	61.1	60.0	59.9
Sixth swage	66.6	68.1	67.7
Anneal 1/2 hour	61.6	61.2	60.6
Seventh swage	67.6	67.7	68.6
Anneal 1/2 hour	62.2	61.0	58.7
Eighth swage	67.2	-	-
Anneal 1/2 hour	61.1	-	-
Ninth swage	65.3	-	-
Anneal 1/2 hour	61.6	-	-
Tenth swage	67.4	-	-
Anneal 1/2 hour	60.4	-	-
Eleventh swage	68.3		

[a] Average at least three determinations

TABLE IV

HARDNESS OF ALLOY #6 AFTER SWAGING
(15% reduction per cycle)

Condition	Hardness, Rockwell A [a]	
	1600°F Anneal	2000°F Anneal
As extruded	62.0	62.0
First swage	67.1	66.1
Anneal 1/2 hour	58.1	59.0
Second swage	66.6	66.6
Anneal 1/2 hour	57.1	56.8
Third swage	65.5	65.6
Anneal 1/2 hour	60.0	57.7
Fourth swage	65.8	64.8
Anneal 1/2 hour	62.7	58.6
Fifth swage	66.2	66.0
Anneal 1/2 hour	57.6	56.8
Sixth swage	66.3	67.1
Anneal 1/2 hour	58.7	59.3
Seventh swage	66.1	66.0
Anneal 1/2 hour	59.1	57.4

[a] Average at least three determinations

TABLE V

HARDNESS OF ALLOY #7 AFTER SWAGING
(10% reduction per cycle)

Condition	Hardness, Rockwell A[a] 1600°F Anneal
As extruded	63.0
First swage	62.9
Anneal 1/2 hour	64.1
Second swage	67.8
Anneal 1/2 hour	63.7
Third swage	65.4
Anneal 1/2 hour	62.5
Fourth swage	67.4
Anneal 1/2 hour	58.9
Fifth swage	65.9
Anneal 1/2 hour	63.2
Sixth swage	64.3
Anneal 1/2 hour	63.2
Seventh swage	65.3
Anneal 1/2 hour	62.0
Eighth swage	64.6
Anneal 1/2 hour	63.2
Ninth swage	66.0
Anneal 1/2 hour	61.0
Tenth swage	66.2
Anneal 1/2 hour	63.1
Eleventh swage	68.1
Anneal 1/2 hour	60.6
Twelfth swage	64.7
Anneal 1/2 hour	63.1
Thirteenth swage	65.5
Anneal 1/2 hour	62.9
Fourteenth swage	65.3
Anneal 1/2 hour	60.4
Fifteenth swage	66.1
Anneal 1/2 hour	60.0
Sixteenth swage	64.6
Anneal 1/2 hour	-

[a] Average of at least three determinations

TABLE VI

HARDNESS OF ALLOY #6 AFTER SWAGING

(10% reduction per cycle)

Condition	Hardness, Rockwell A[a] 1600°F Anneal
As extruded	64.8
First swage	-
Anneal 1/2 hour	65.3
Second swage	66.9
Anneal 1/2 hour	64.3
Third swage	63.2
Anneal 1/2 hour	64.9
Fourth swage	65.6

[a] Average of at least three determinations

TABLE VII

WEIGHT CHANGE AFTER EXPOSURE IN AIR [a]

Material	Weight Change, mg/cm^2											
	1800°F			2000°F			2200°F			2400°F		
	1 hr	10 hrs	100 hrs	1 hr.	10 hrs.	100 hrs.	1 hr.	10 hrs.	100 hrs.	1 hr.	10 hrs.	100 hrs.
Nichrome	+0 54	+1.09	+0 98	+0 74	+1 38	+1.10	+1 31	+2.03	-0.36	+1 87	+1.91	-2.67
Alloy #6	0 00	+0.23	+0 11	0.00	+0 09	0.10	0 0	+0.29	-0.39	0 00	+0.50	-7.54
Alloy #6A	0.00	+0.15	0 00	0.00	+0 07	0.10	0 0	+0.14	-0 48	+0.25	+1.20	-5.99
Alloy #7	0.00	+0.23	0 00	0 00	+0 18	0.10	0 0	+0.54	-0.75	0.00	0.00	-6.48

[a] Same sample was weighed after 1, 10, 100 hours.
Different samples were used for the different temperatures.

TABLE VIII

OXIDE PENETRATION AFTER EXPOSURE IN AIR [a]

Material	Penetration, inches after 100 hours exposure at			
	1800°F	2000°F	2200°F	2400°F
Nichrome	0.0007	0.00084	0.0014	0.0014
Alloy #6	0.00035	0.00028	0.00014	0.00028
Alloy #6A	0.00014	0.00028	0.00014	0.0017
Alloy #7	0.00014	0.00028	0.00014	0.00042

[a] Specimens were mounted in bakelite, polished and the oxide penetration determined with a measuring eye piece.

TABLE IX

HARDNESS OF ALLOYS #6, #6A and #7 AFTER COLD WORKING BY SWAGING AND HEAT TREATMENTS IN A

Material	Hardness, Rockwell A[a]								
	As Swaged	After Treatment at 1500°F for		After Treatment at 1800°F for		After Treatment at 2000°F for		After Treatment at 2200°F for	
		1 hr.	5 hrs.	1 hr.	5 hrs.	1 hrs.	5 hrs.	1 hr.	5 hrs.
Alloy #6 (15% reductions)									
As swaged 8 times	67.5	61.5	58.5	59.9	59.1	60.1	59 0	59.8	59.5
Alloy #6A (10% reductions)									
As swaged 3 times	67.1	67.0	64.7	64.4	62.3	62.3	61 4	60 8	60.2
Alloy #7 (20% reductions)									
As swaged 6 times	60.5	61.7	61.3	61.8	60.9	61.5	61.0	61.5	58.6
" " 8 "	60.9	60.8	60.0	61.1	58.4	60.4	59.5	60.2	56.3
" " 10 "	61.0	60.1	60.3	59.5	57.8	61.1	59.9	60.0	56.5
" " 11 "	67.0	61.0	58.6	60.2	58.2	58.7	51.2	58.8	42.6

[a] Average of at least three determinations

TABLE X

TENSILE PROPERTIES OF ALLOYS #6, #6A AND #7 AFTER COLD WORKING THE ALLOYS BY SWAGING

Alloy	Composition	Test Temp. °F	0.2% Y.S., psi	UTS, psi	e, %	RA, %	Condition
#6	80 Ni 20 Cr + 2.5 v/o ThO_2	R.T.	83,250	116,650	10.9	13.7	As extruded
		1800	7,470	7,970	5.3	5.3	" "
		2000	1,990	5,870	3.0	6.4	As swaged 7 times, annealed at 2000°F between swagings, 15% reduction steps
#6A	80 Ni 20 Cr + 2.5 v/o ThO_2	R.T.	89,500	123,000	7.7	11.8	As extruded
		1800	5,050	6,060	4.4	5.1	" "
		1800	10,200	12,900	1.0	1.0	As swaged 4 times, annealed at 1600°F between swagings, 10% reduction steps
		2000	4,810	9,440	2.0	1.0	
#7	80 Ni 20 Cr + 5.0 v/o ThO_2	R.T.	82,500	116,500	8.5	10.1	As extruded
		1800	6,490	7,190	8.6	10.1	" "
		R.T.	149,000	149,000	2.0	6.0	As swaged 11 times, annealed at 1600°F between swagings, 20% reduction steps

TABLE X CONTINUED

TENSILE PROPERTIES OF ALLOYS #6, #6A AND #7 AFTER COLD WORKING THE ALLOYS BY SWAGING

Alloy	Composition	Test Temp. °F	0.2% Y.S., psi	UTS, psi	e, %	RA, %	Condition
#7	80 Ni 20 Cr + 5.0 v/o ThO_2	1800	5,470	10,300	2.0	3.0	As swaged 6 times, annealed at 1600°F between swagings, 20% reduction steps
		1800	6,670	10,600	2.0	3.0	As swaged 8 times, annealed at 1600°F between swagings, 20% reduction steps
		1800	5,730	8,720	2.0	2.0	As swaged 10 times, annealed at 1600°F between swagings, 20% reduction steps
		1000 2000	5,750 3,175	7,025 5,860	3.0 2.0	3.9 5.8	As swaged 11 times, annealed at 1600°F between swagings, 20% reduction steps
		1800 2000	7,500 4,590	7,500 4,590	5.2 -	0 -	As swaged 6 times, annealed at 1600°F between swagings, 10% reduction steps

TABLE XI

STRESS RUPTURE PROPERTIES AT 2000°F OF ALLOYS #6, #6A,

AND #7 AFTER COLD WORKING THE ALLOYS BY SWAGING

Material	Stress, psi	Rupture Life, hours	e, %	R.A. %
Alloy #6				
15% reductions				
After 8 cycles	2,000	0.3	4	7
Alloy #6A				
10% reductions				
After 4 cycles	4,000	1.0	9	0
	3,000	8.0	5	0
Alloy #7				
10% reductions				
After 6 cycles	5,000	0.05	5	4
After 11 cycles	4,000	0.03	4	0
After 16 cycles	3,000	0.1	1	0
After 16 cycles	2,000	0.6	1	0

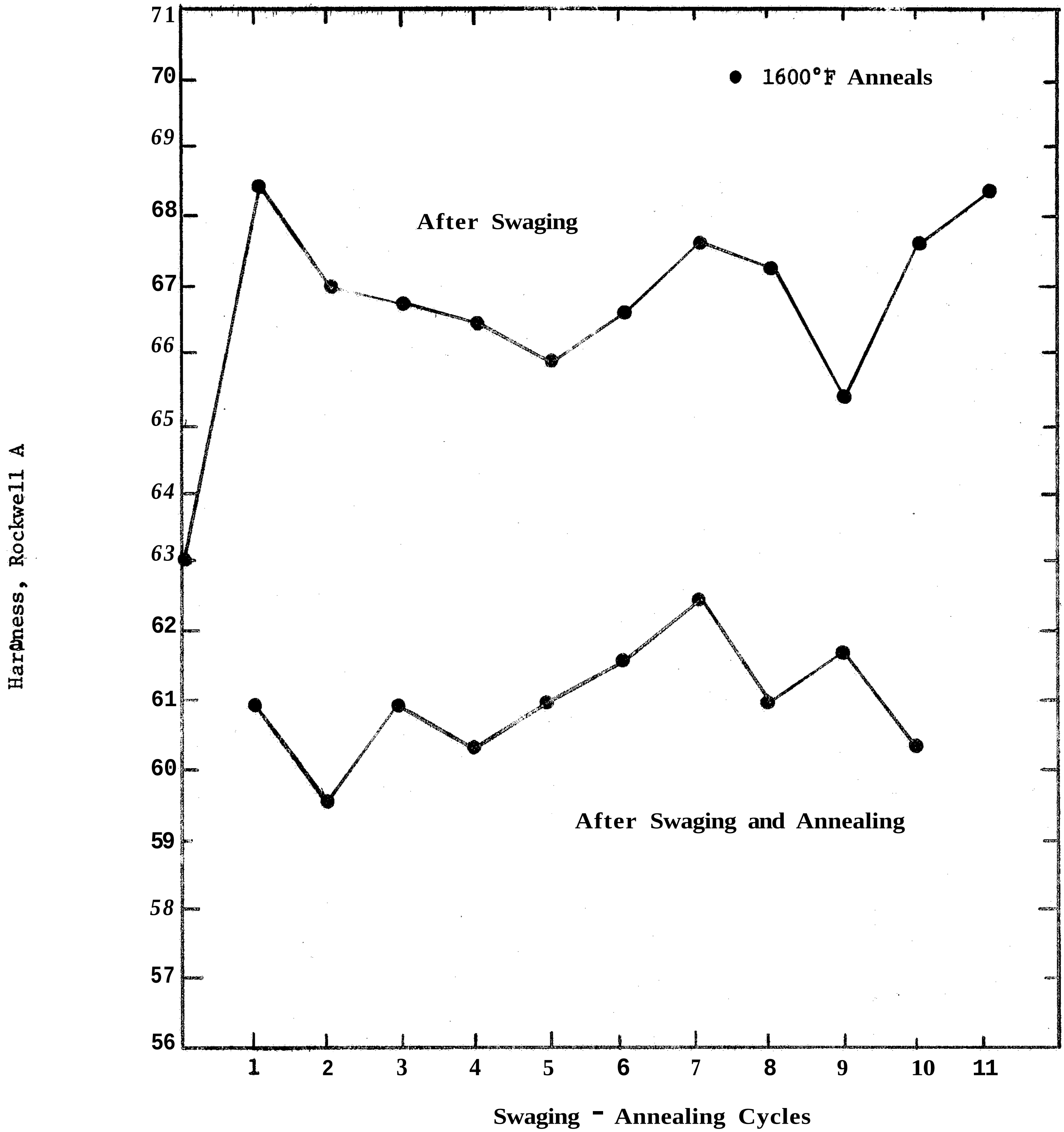


Figure 1: Effect of Cold Work (Swaging by 20% Reduction per cycle) on Alloy #7, as measured by hardness changes.

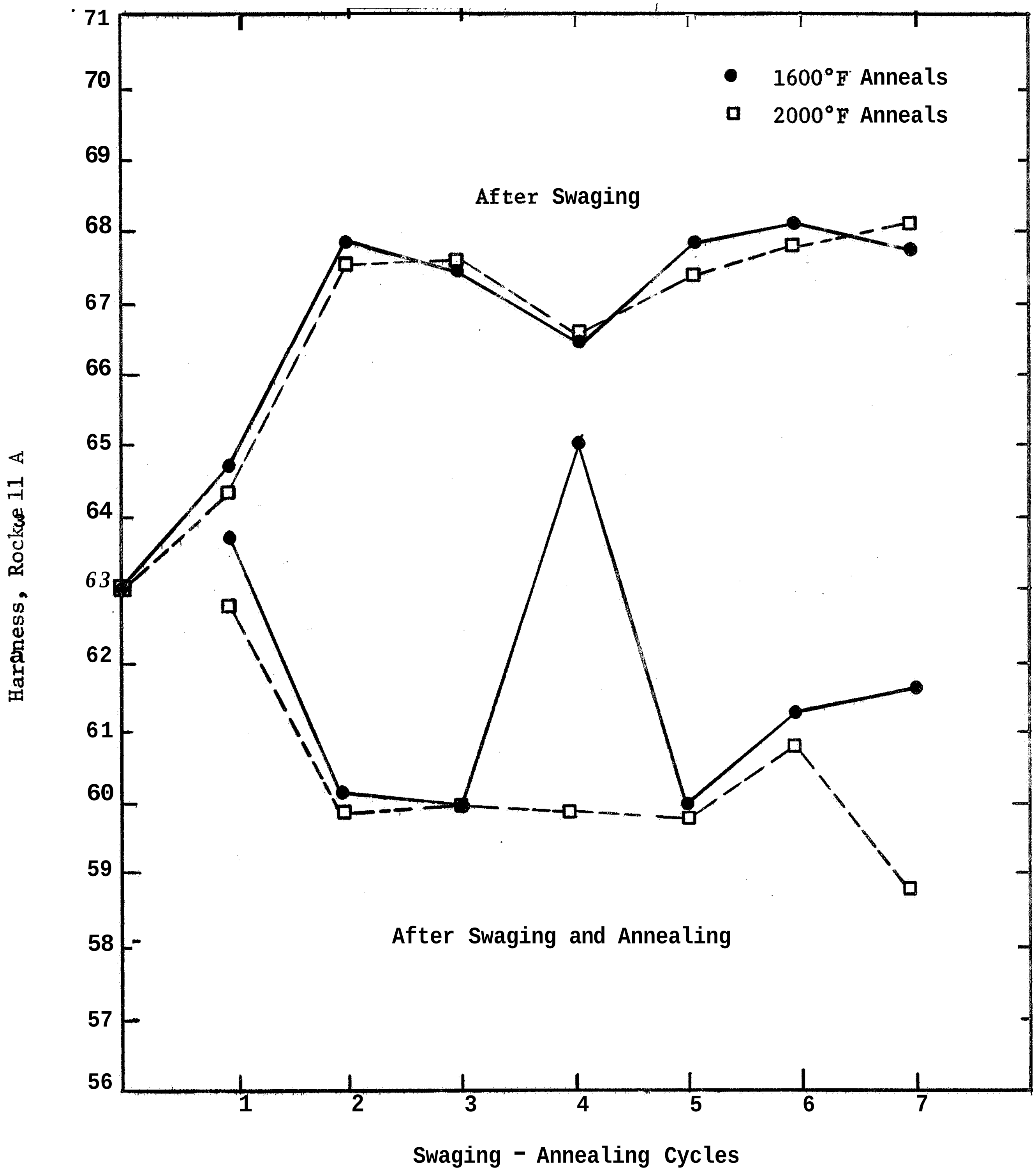


Figure 2: Effect of Cold Work (Swaging by 15% Reduction per cycle) on Alloy #7, as measured by hardness changes.

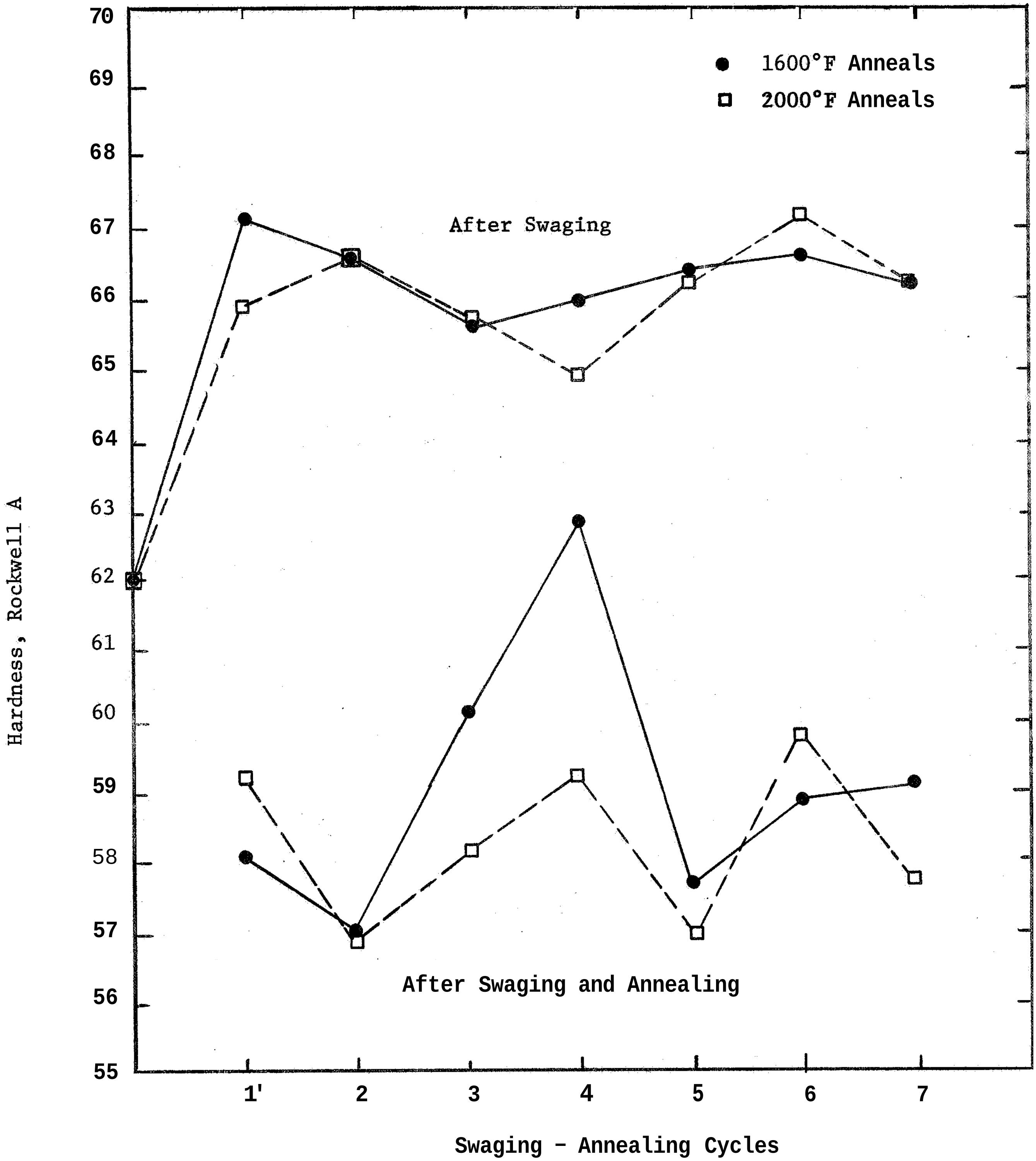
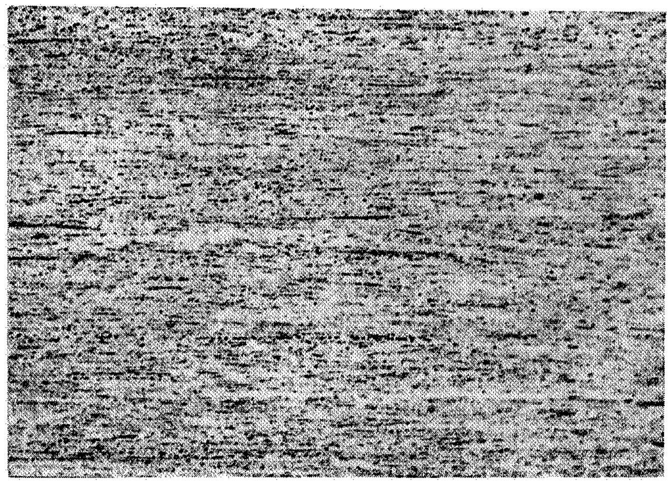


Figure 3: Effect of Cold Work (Swaging of 15% Reduction per cycle) on Alloy #6, as measured by hardness changes.



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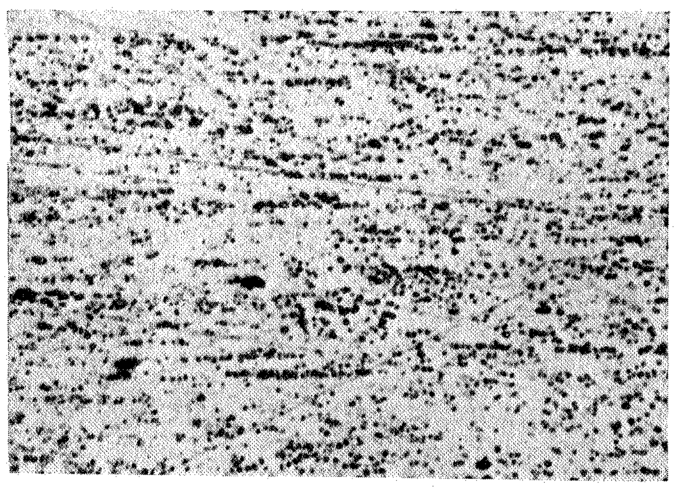

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Figure 4: Microstructure of longitudinal section of Alloy #6 (80 nickel-20 chromium + 2.5 v/o ThO_2), as extruded.

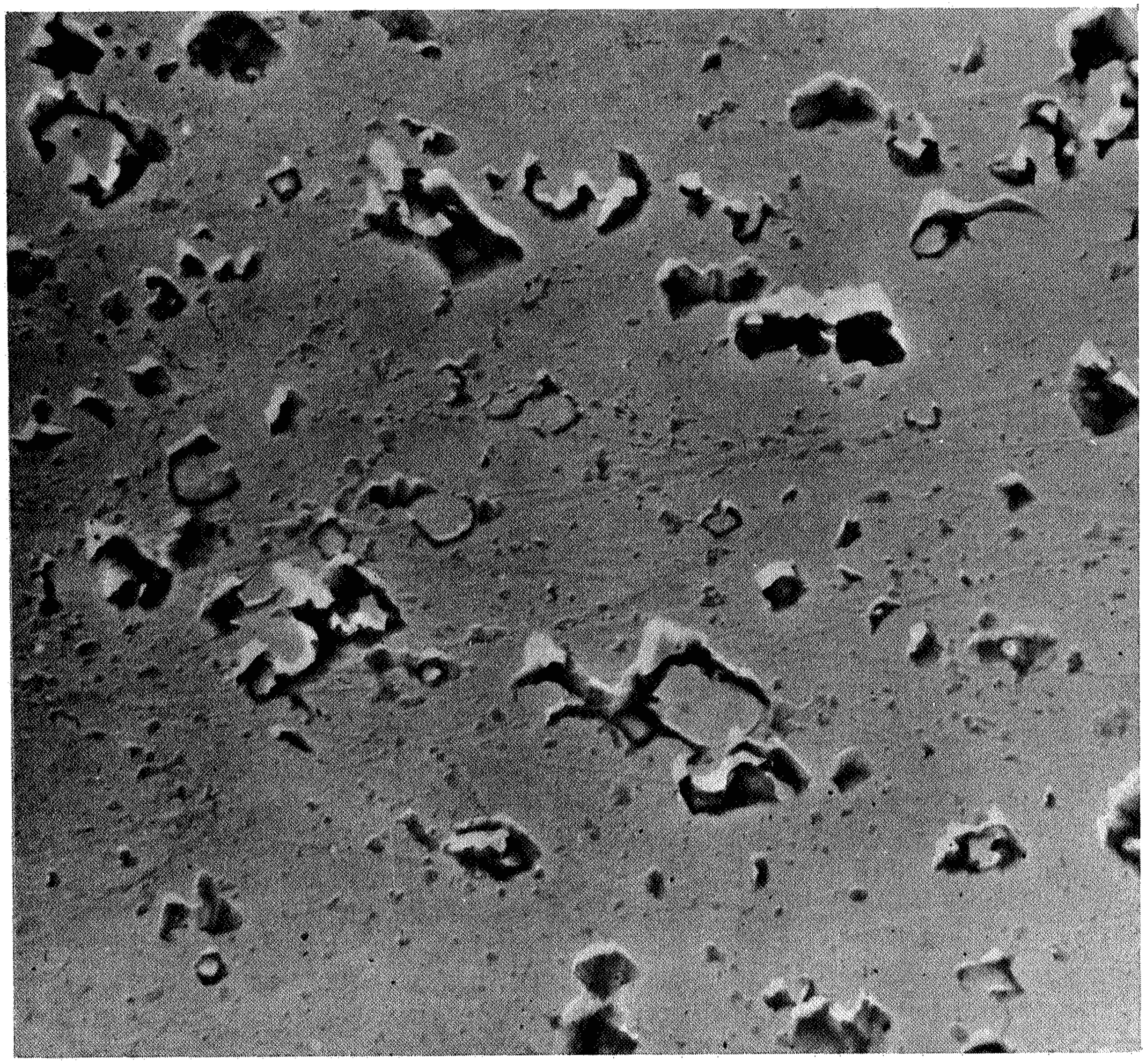

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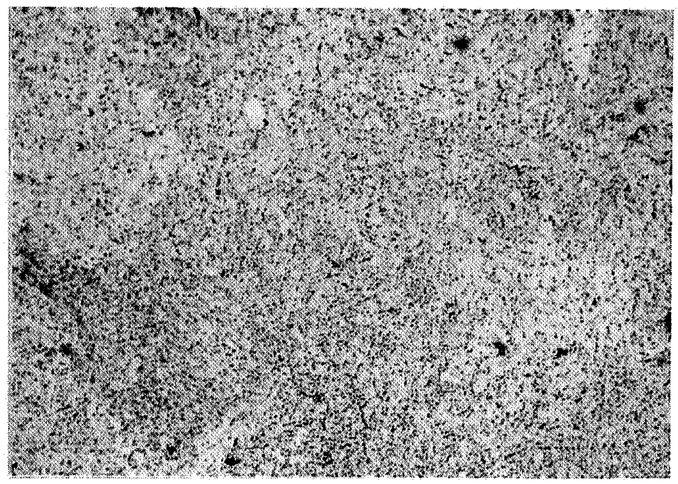
Figure 5: Electron micrograph of a longitudinal section of Alloy #6, (80 nickel-20 chromium + 2.5 v/o ThO_2), as extruded.



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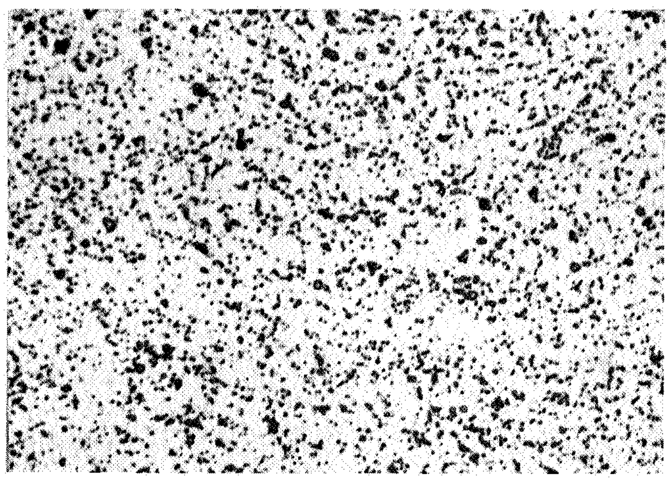

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Figure 6: Microstructure of transverse section of Alloy #6 (80 nickel-20 chromium + 2.5 v/o ThO_2), as extruded.

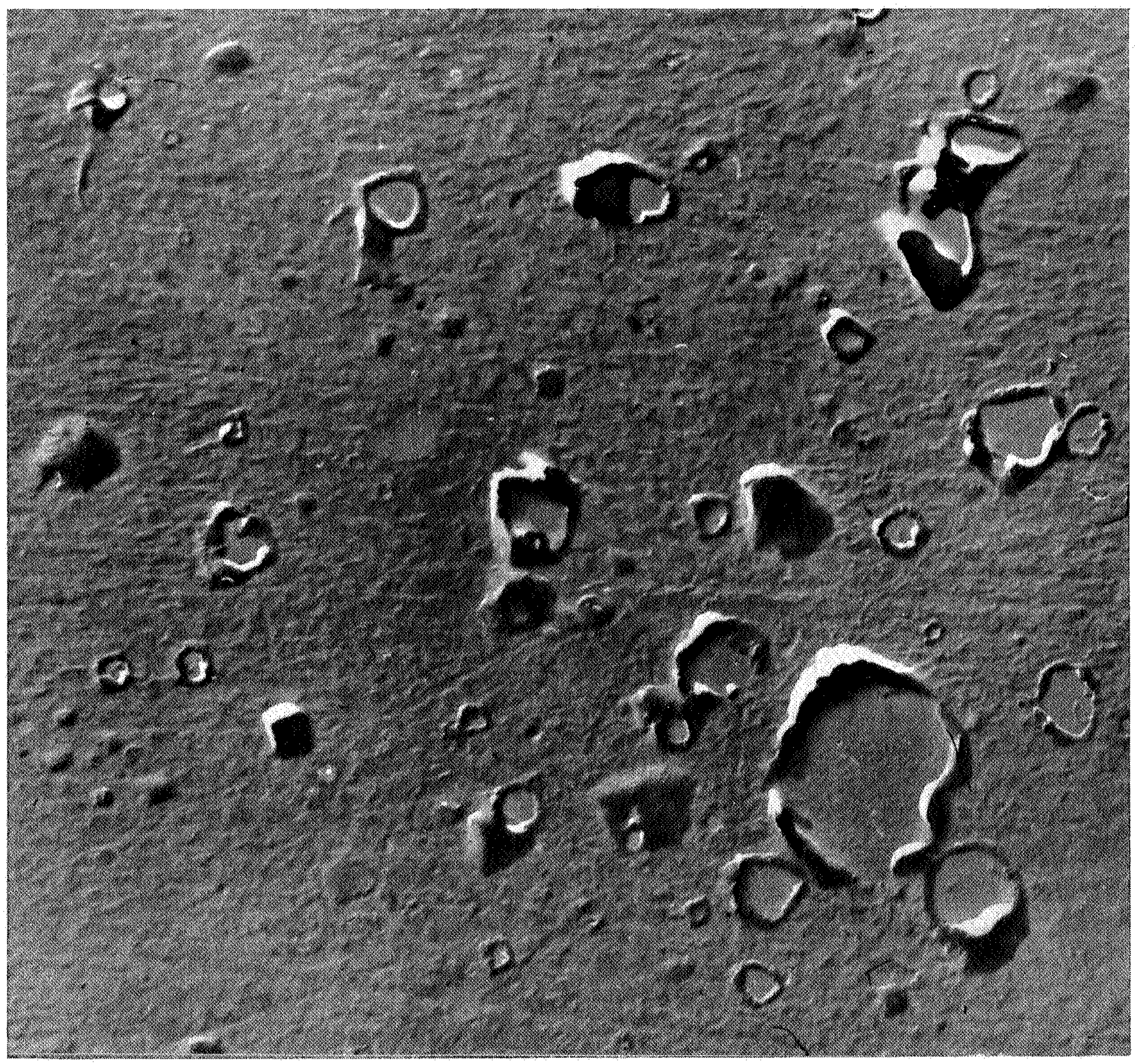

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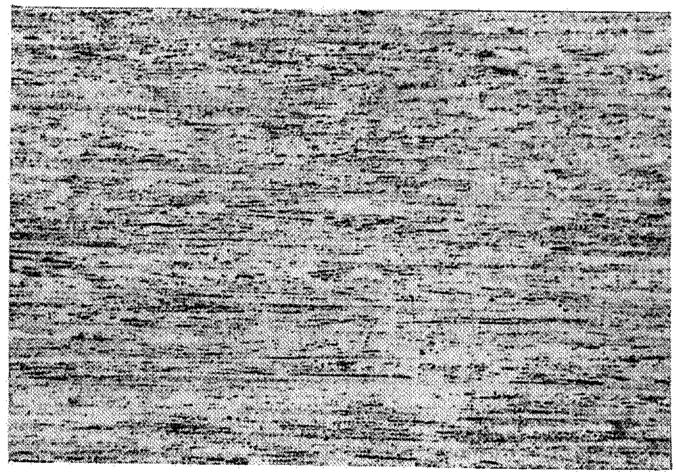
Figure 7: Electron micrograph of a transverse section of Alloy 86, (80 nickel–20 chromium + 2.5 v/o ThO_2), as extruded.



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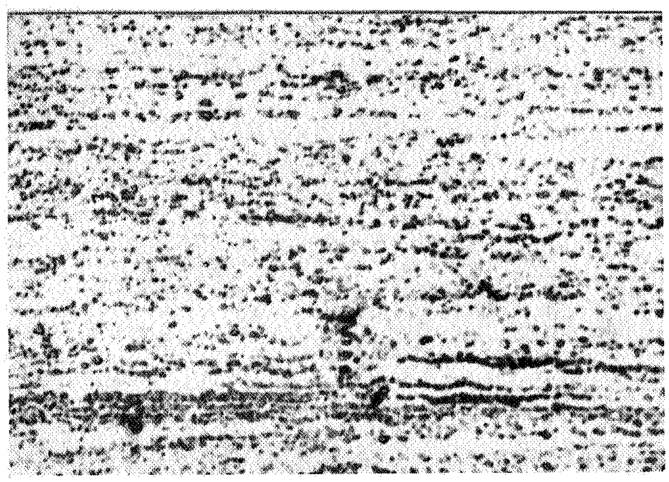

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Figure 8: Microstructure of longitudinal section of Alloy #6A (80 nickel-20 chromium + 2.5 v/o ThO_2), as extruded.

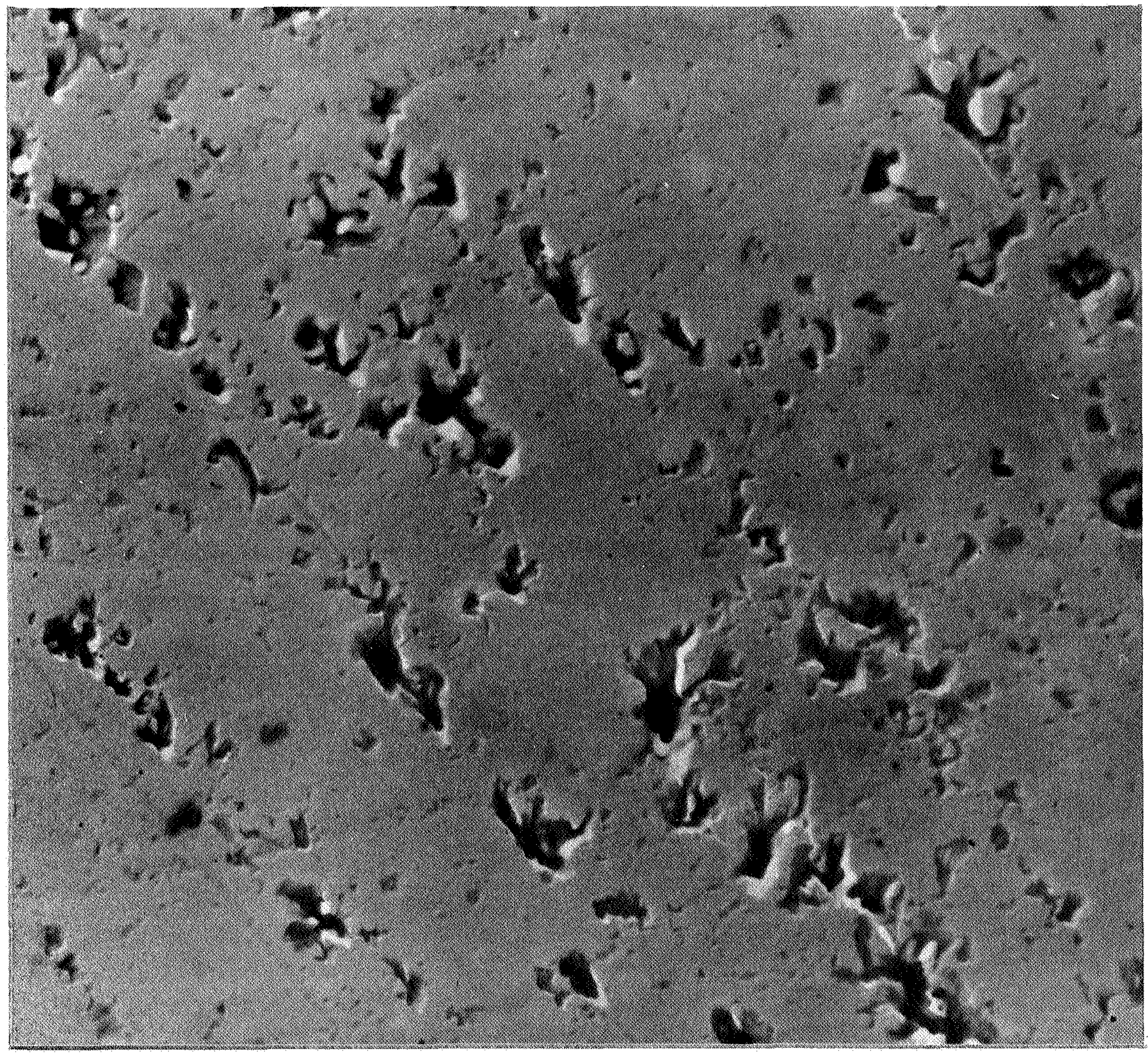

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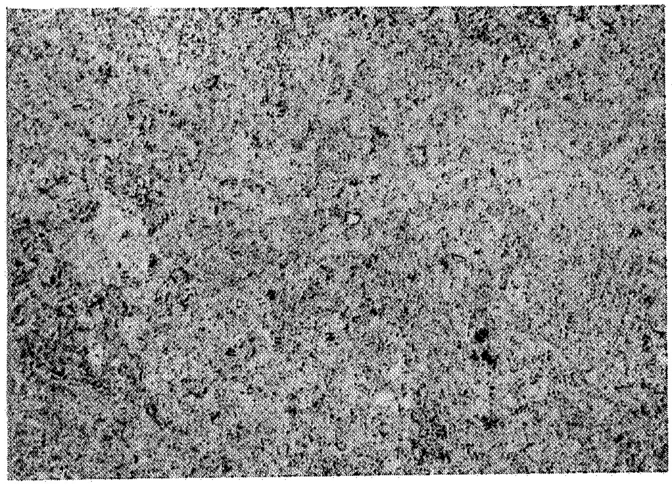
Figure 9: Electron micrograph of a longitudinal section of Alloy #6A, (80 nickel-20 chromium + 2.5 v/o ThO_2), as extruded.



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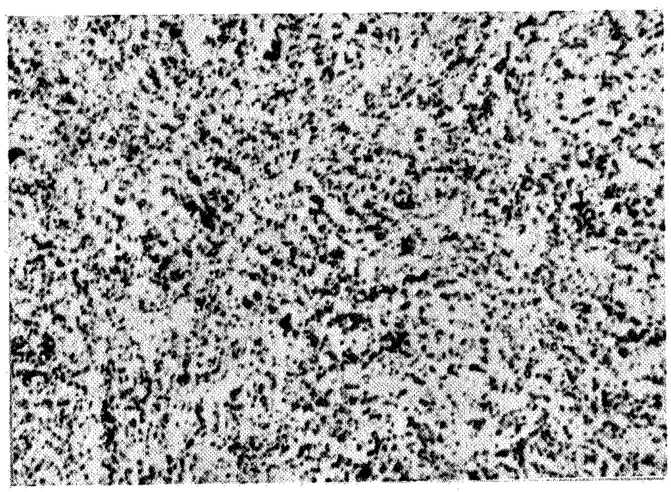

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Figure 10: Microstructure of transverse section of Alloy #6A (80 nickel-20 chromium + 2.5 v/o ThO_2), as extruded.

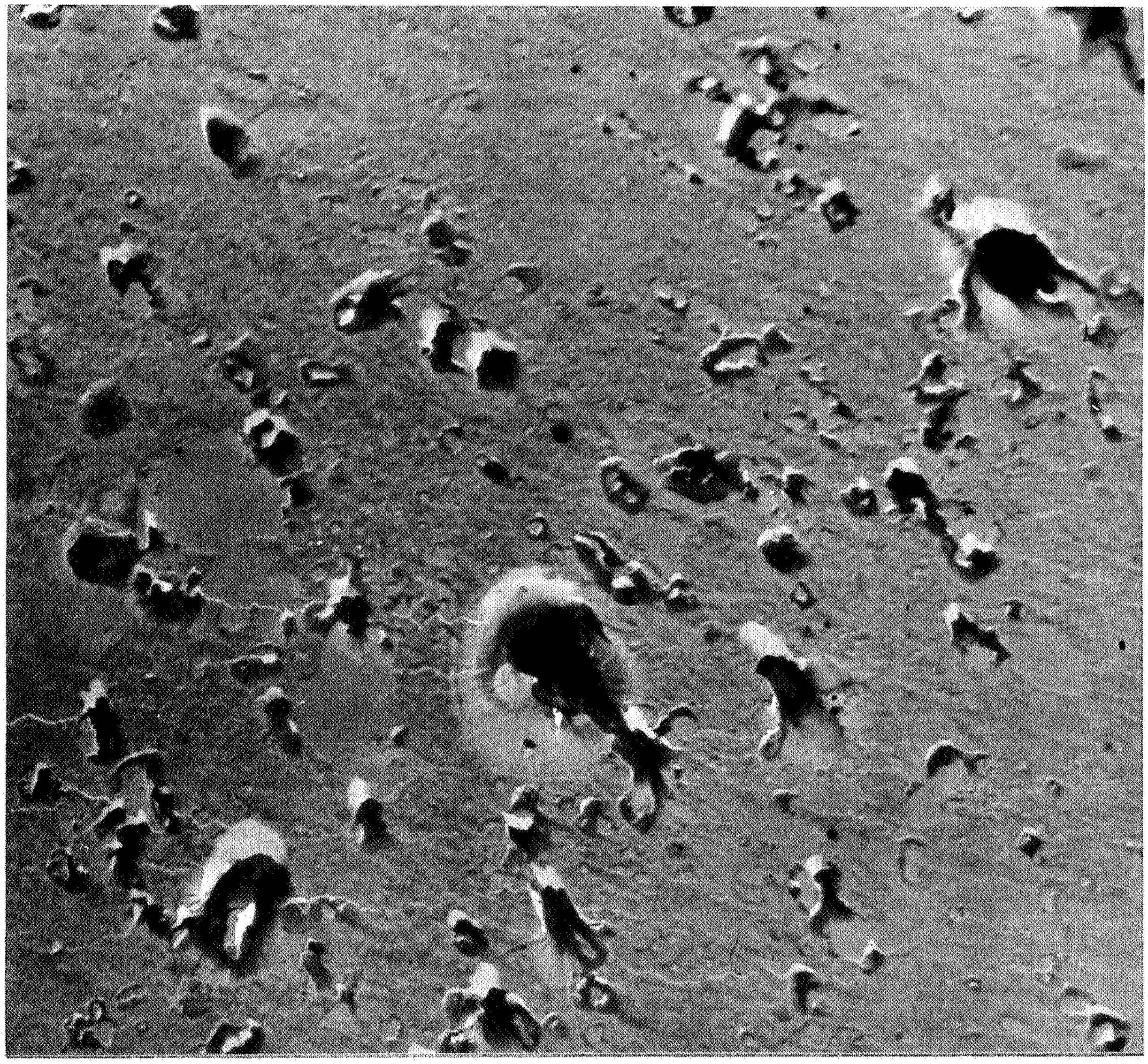

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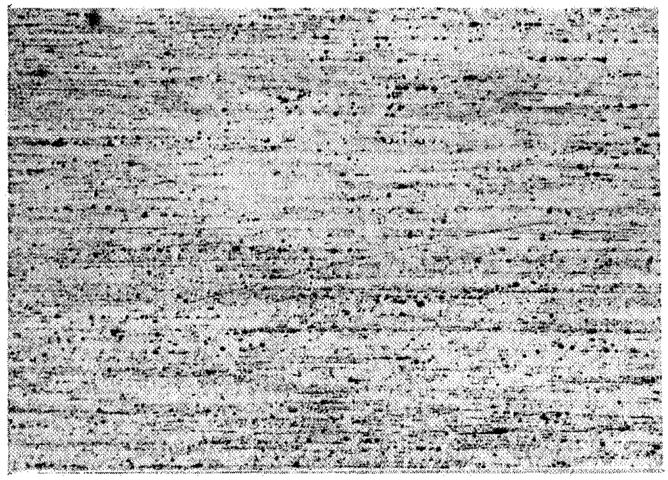
Figure 11: Electron micrograph of a transverse section of Alloy #6A, (80 nickel-20 chromium + 2.5 v/o ThO_2), as extruded.



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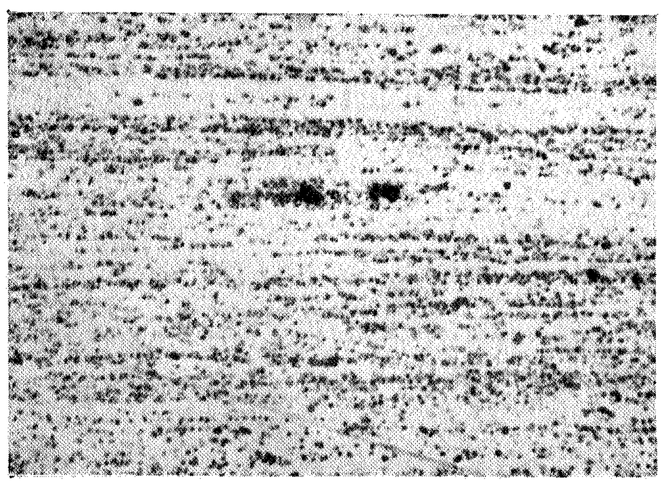

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Figure 12: **Microstructure of longitudinal section of Alloy #7 (80 nickel-20 chromium + 5 v/o ThO_2), as extruded.**

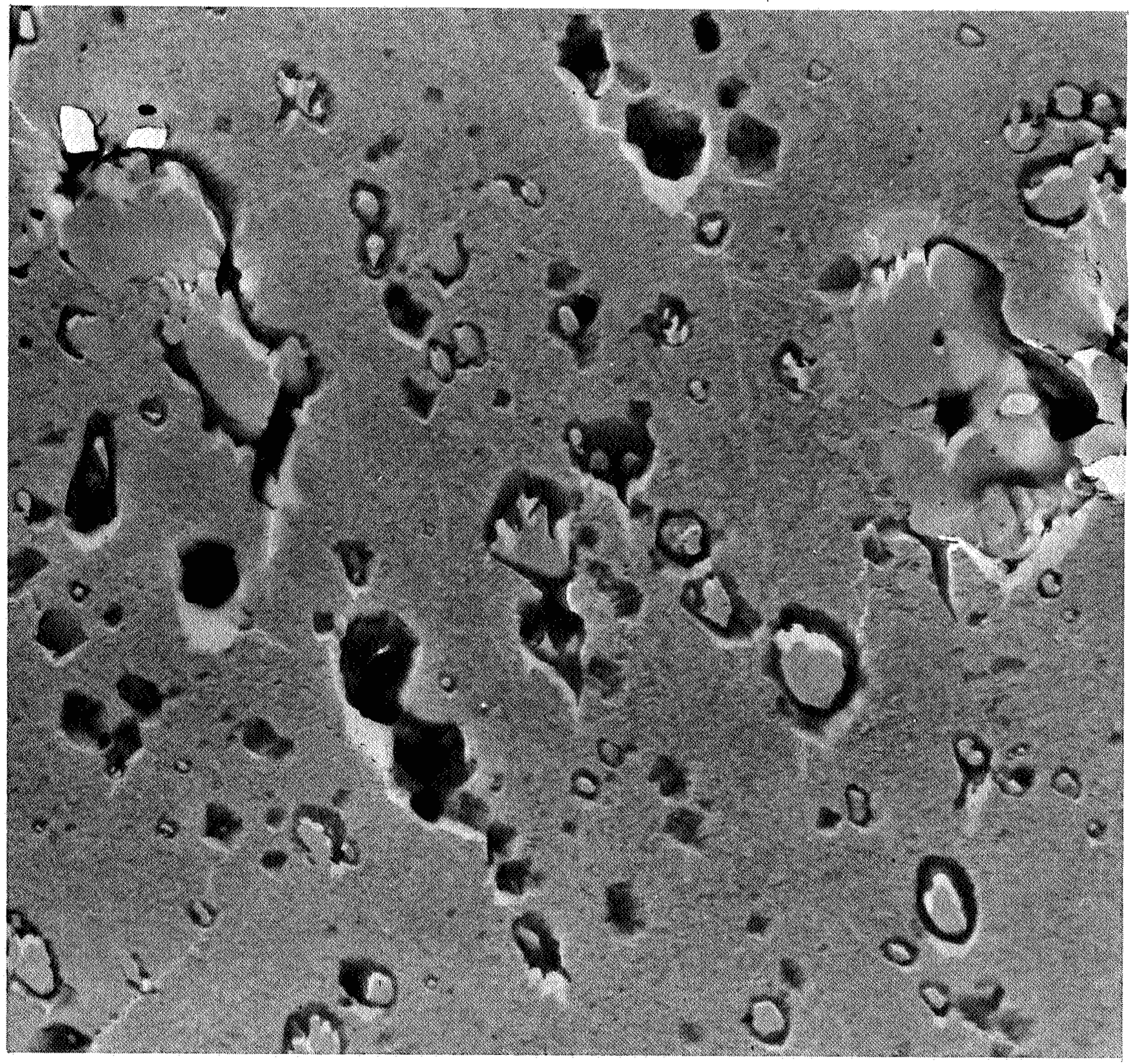

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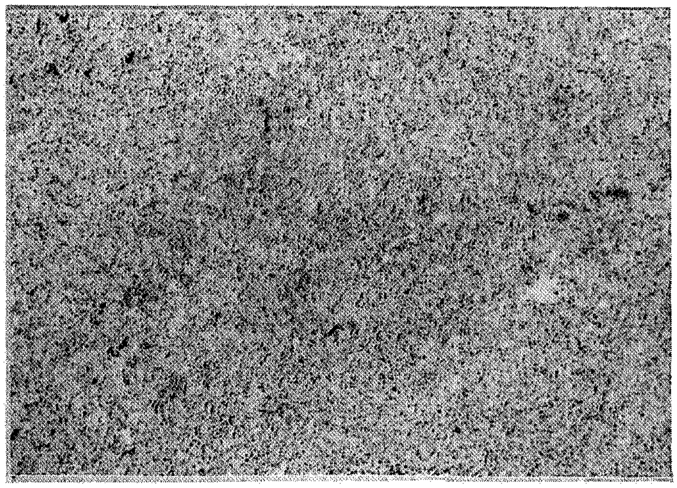
Figure 13: Electron micrograph of a longitudinal section of Alloy #7, (80 nickel-20 chromium + 5 v/o ThO_2), as extruded.



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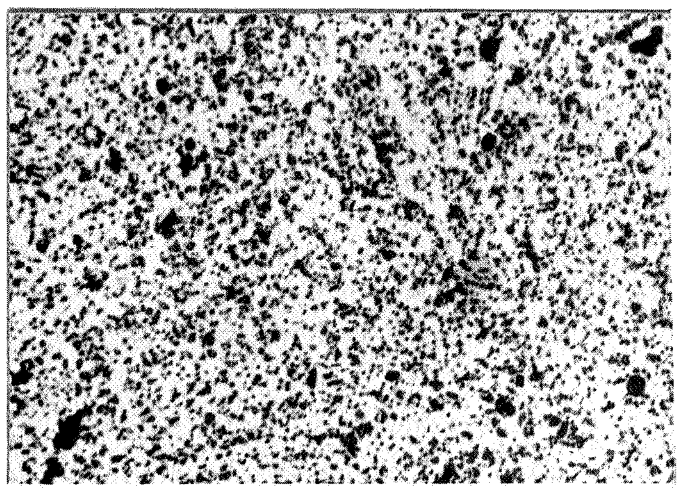

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Figure 14: Microstructure of transverse section of Alloy 87 (80 nickel–20 chromium + 5 v/o ThO_2), as extruded.

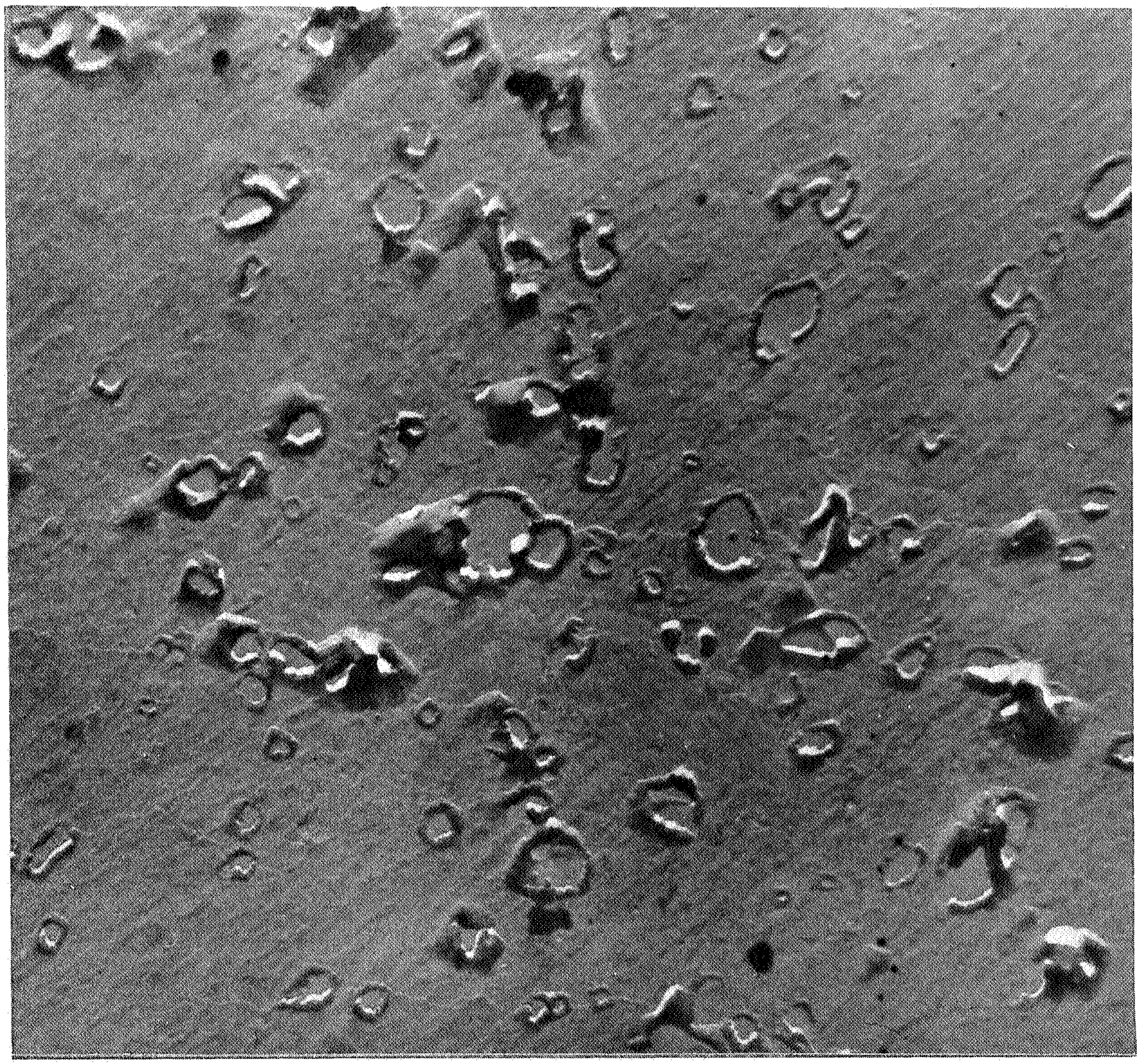

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Figure 15: Electron micrograph of a transverse section of Alloy #7, (80 nickel-20 chromium + 5 v/o ThO_2), as extruded.

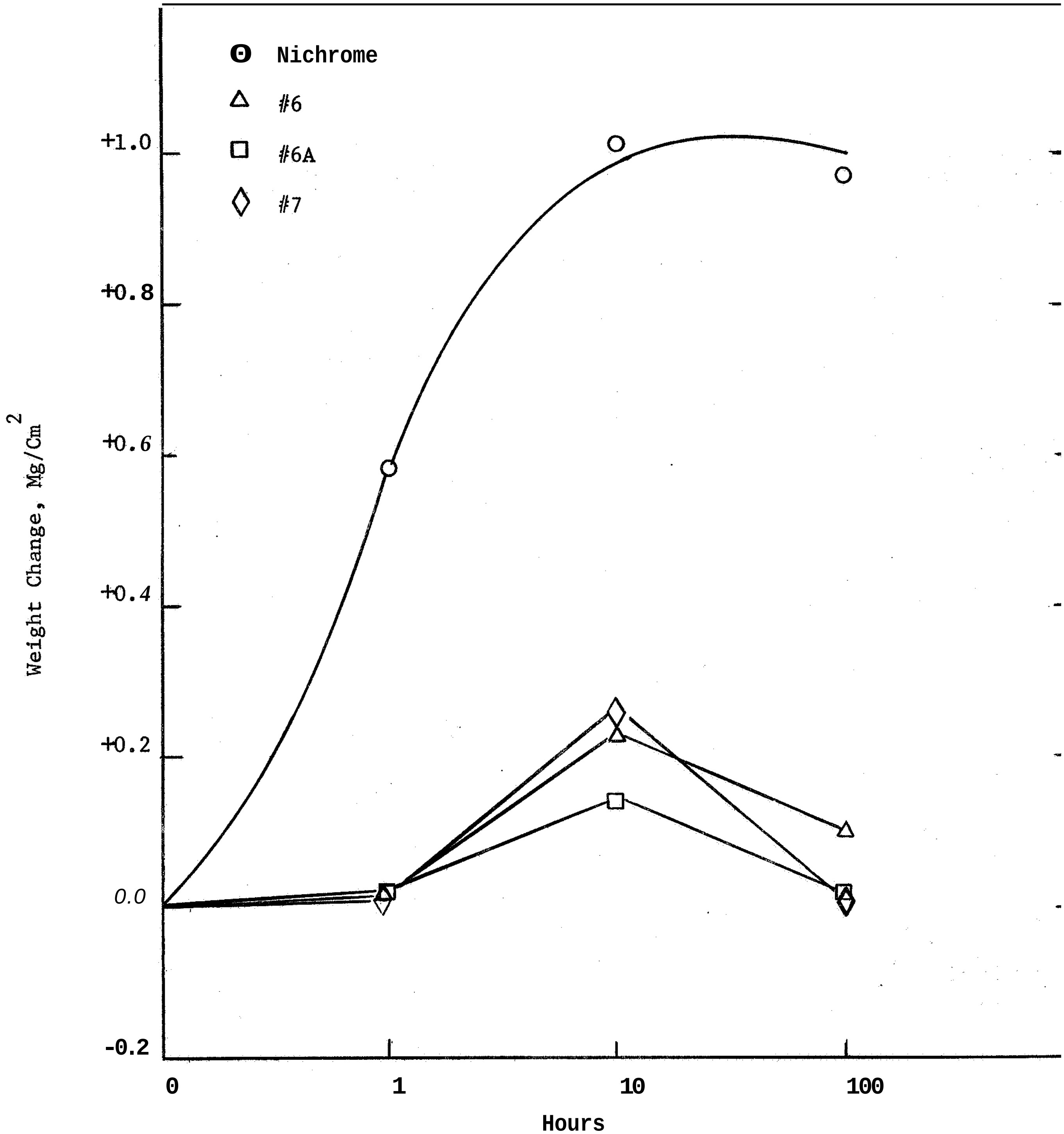


Figure 16: Oxidation Characteristics of Various Nickel-Base Alloys After Exposure in Air for Various Times at 1800°F (as measured by weight change)

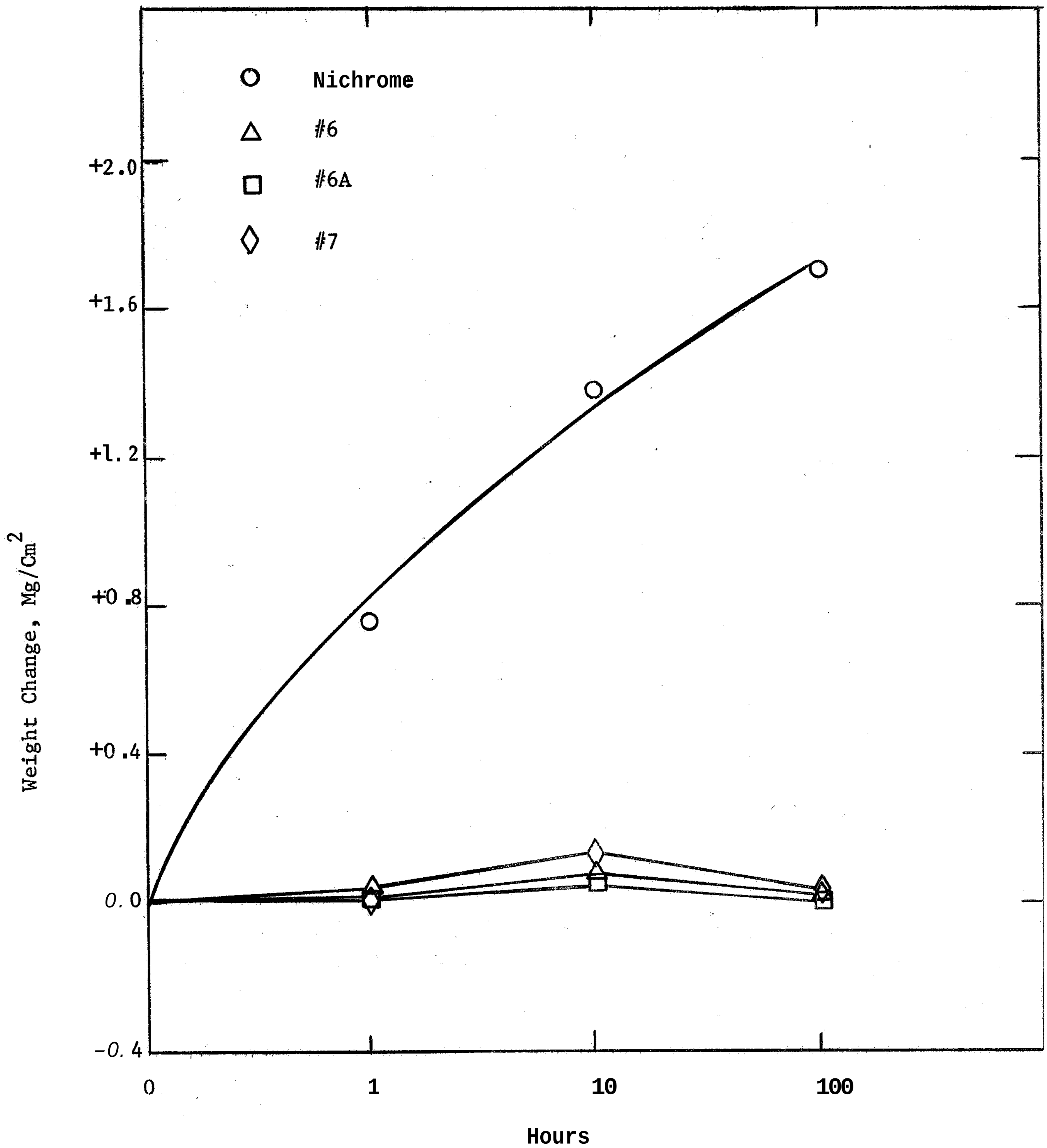


Figure 17: Oxidation Characteristics of Various Nickel-Base Alloys After Exposure in Air for Various Times at 2000°F. (as measured by weight change)

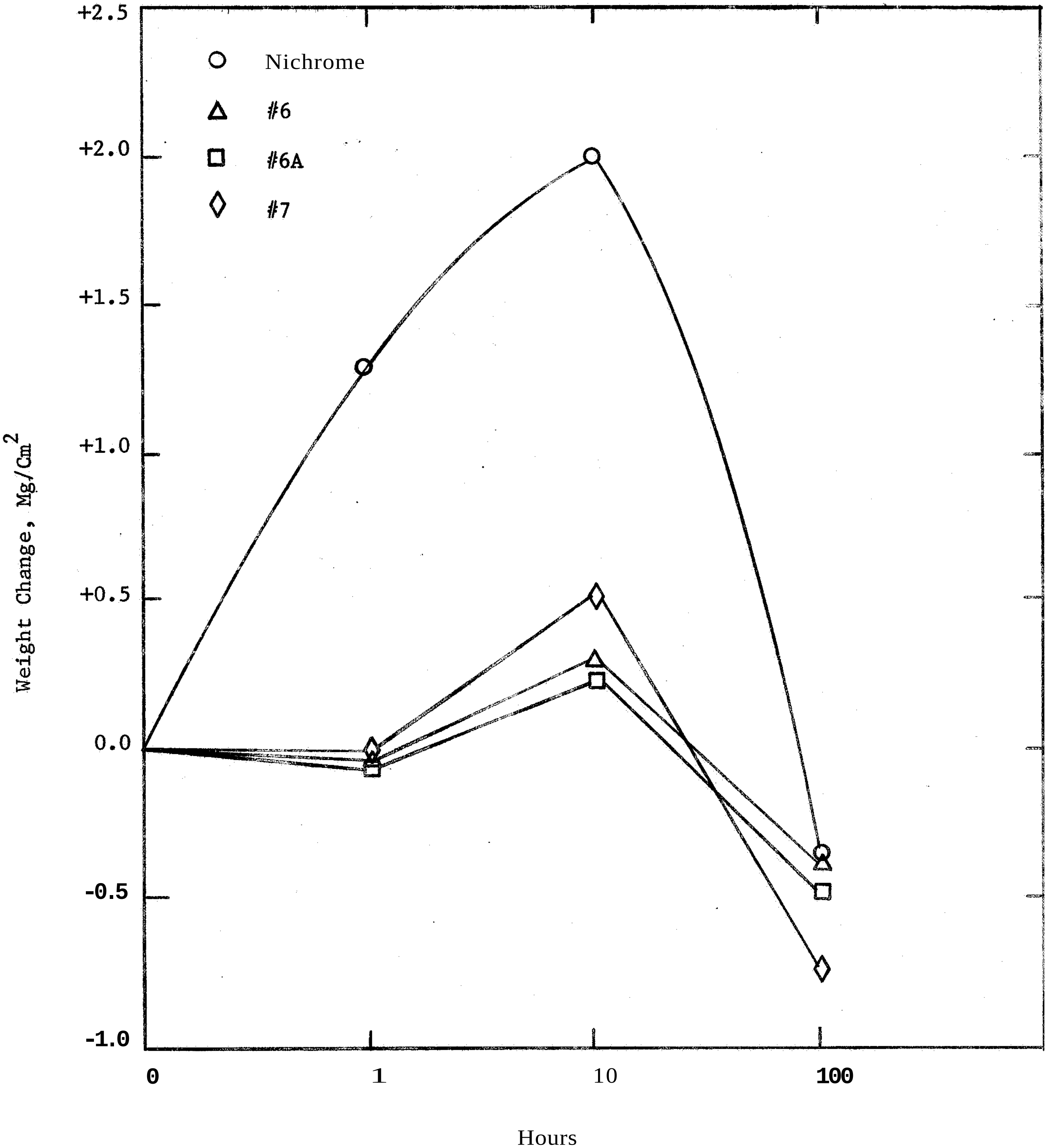


Figure 18: Oxidation Characteristics of Various Nickel-Base Alloys After Exposure in Air for Various Times at 2200°F (as measured by weight change)

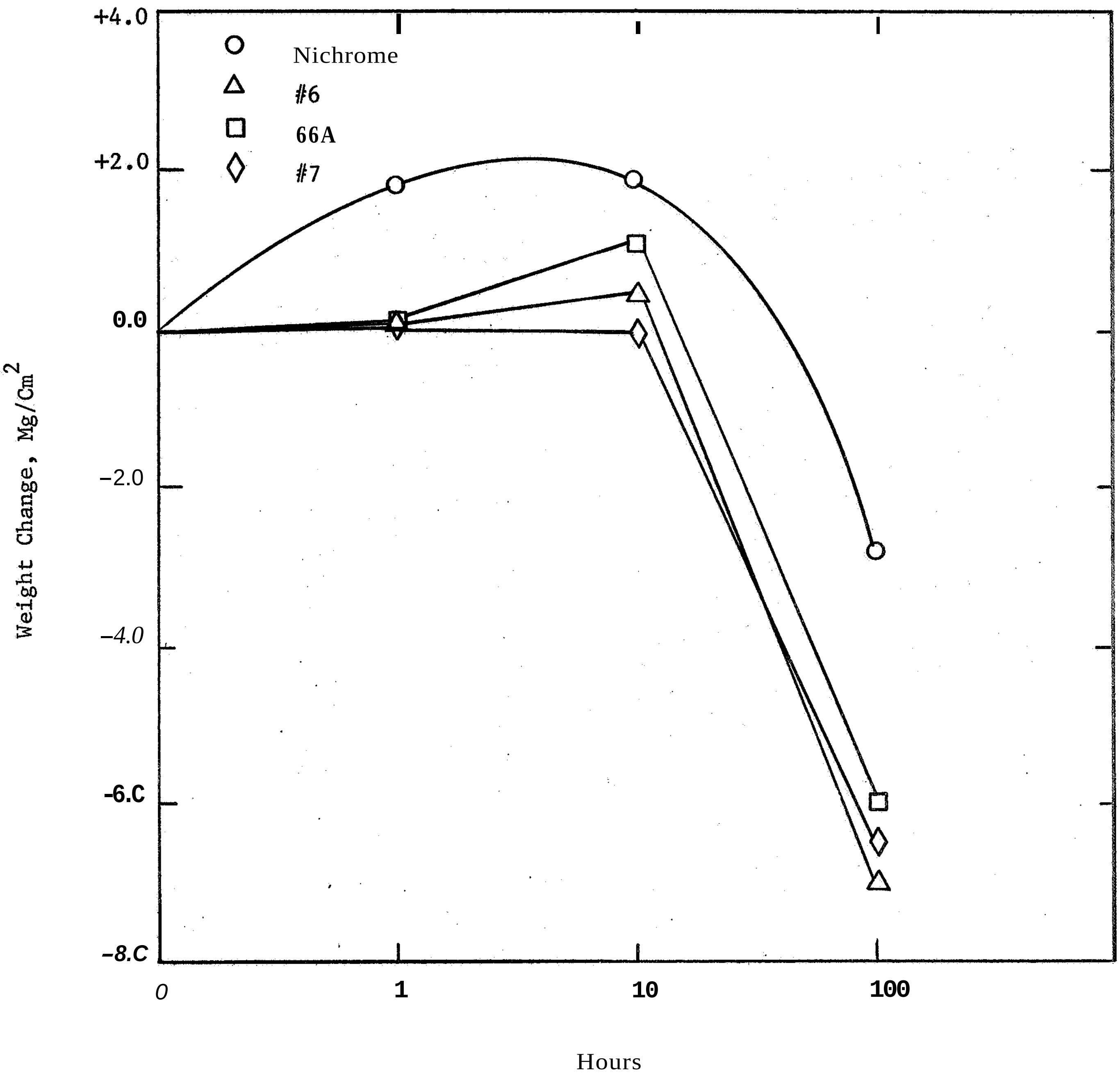


Figure 19: Oxidation Characteristics of Various Nickel–Base **Alloys** After Exposure in Air for Various Times at 2400°F (as measured by weight change)

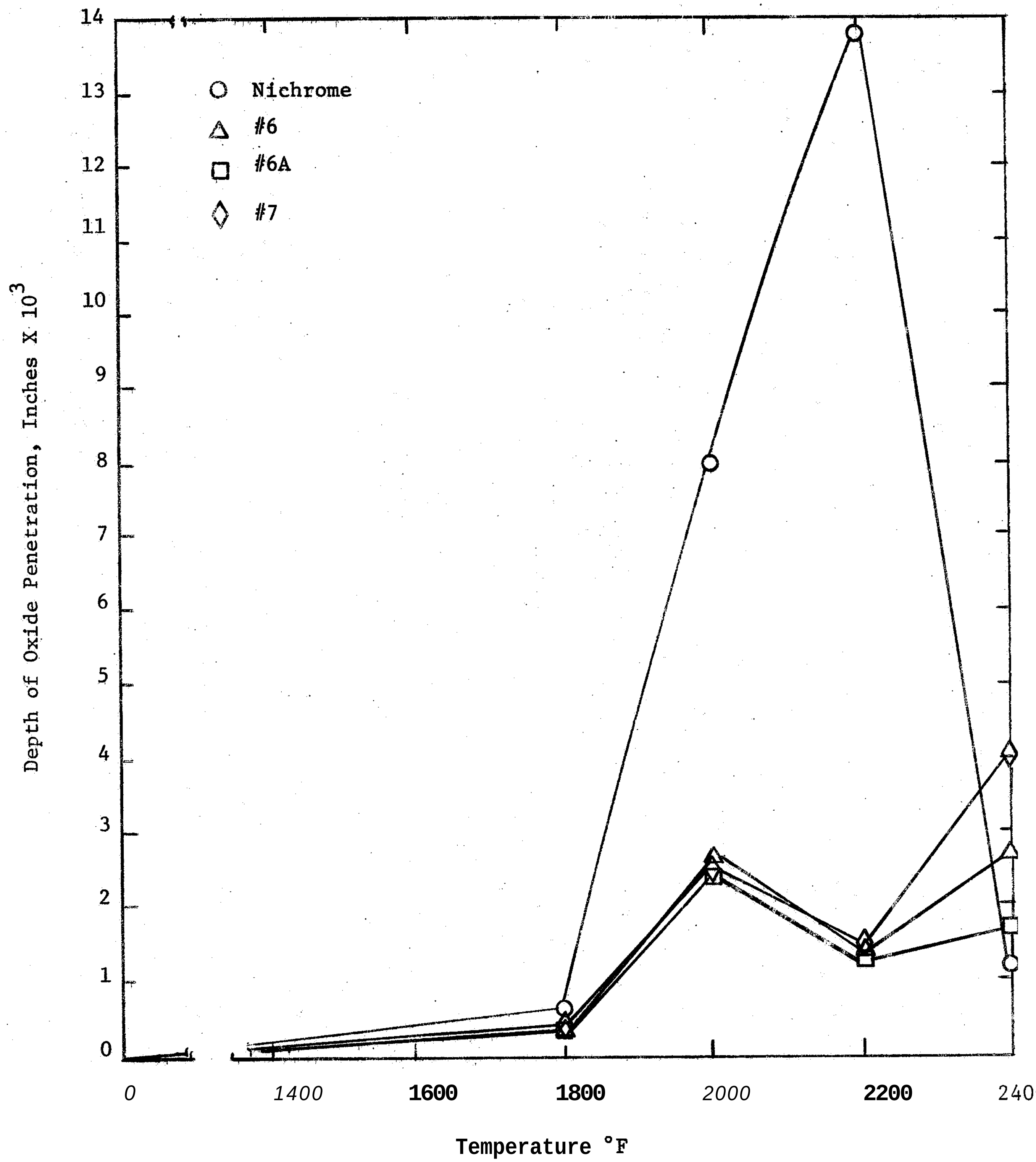


Figure 20: Oxidation Characteristics of Various Nickel-Base Alloys After 100 Hours Exposure in Air, at Various Temperatures. (as measured by depth of oxide penetration)

APPENDIX: Experiments with Dispersion-Strengthened Nickel/Chromium-Base Alloys

Experiments with dispersion-strengthened nickel/chromium-base matrices were conducted in cooperation with a French group using know-how of both companies. The processing techniques used can be applied to the dispersion strengthening of almost any solid solution matrix containing elements such as iron, nickel, chromium, cobalt, molybdenum, tungsten, manganese, platinum metals, and niobium. Excluded are the more reactive metals. The process is particularly attractive as it lends itself to the design of "tailor-made" alloys because of the applicability to various solid solutions containing chromium. The processing variables used in preparing these alloys are shown schematically in Figure A 1.

Samples that were prepared had a very uniform and fine dispersion in the extruded condition, an oxide particle size of around 0.1 micron and an interparticle spacing below 1 micron.

The tensile and stress rupture properties of some of the alloys were evaluated in the as-extruded and the extruded and swaged condition. The results for the 2000°F tests are listed in Table A 1. In the extruded condition the material had moderate to low strength coupled with good ductility. After various swaging and annealing treatments the high temperature stress rupture strength was increased with best results around 6,000 psi for 100 hours' rupture life.

These preliminary results indicate that the process chosen here can indeed be applied to the dispersion strengthening of a wide variety of superalloy compositions. Stable structures can be obtained. The first attempts to improve the strength properties of extruded alloys by swaging were successful, although the stress rupture data are still far from being optimized.

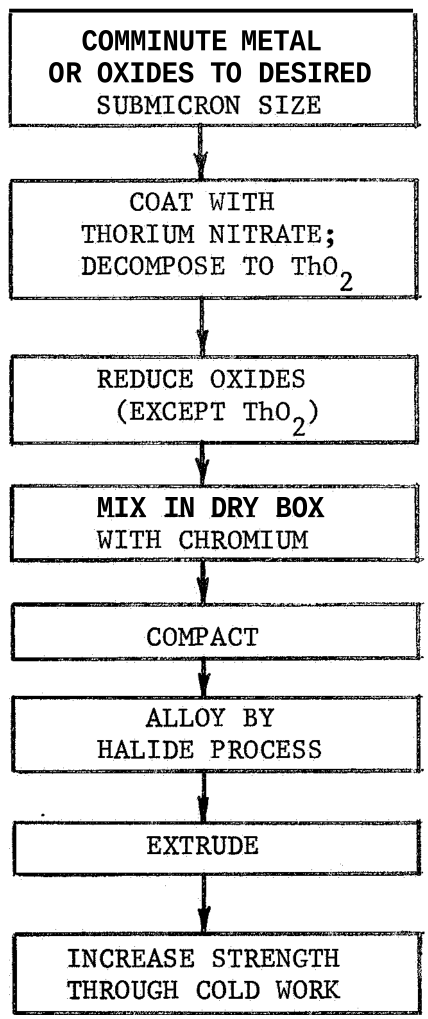
TABLE A 1

MECHANICAL PROPERTIES OF DISPERSION-STRENGTHENED NICHROME ALLOYS

Alloy	Nominal Composition	Hot/Cold Work	2000°F Tensile Properties				2000°F Stress Rupture Properties	
			UTS, psi	0.2% Y.S.psi	e,%	R.A. %	psi	Life, hours
1A	80%Ni 20%Cr + 5 v/o ThO_2	As extruded	2,430	1,570	18.5	12.0	-	-
		As extruded & swaged	5,200	5,200	2.1	0.0	4,000 5,000	32.1 0.6
		As extruded & swaged	8,300	7,800	3.4	1.0	4,000	791 discontinued
		As extruded & swaged	9,000	9,000	3.4	4.0	4,000 6,000	1026 (Disc) 2.0
1B	80%Ni 20%Cr + 5 v/o ThO_2	As extruded	3,910	-	7.4	1.4	-	-
		As extruded & swaged	5,100	4,500	3.5	2.0	4,000 6,000	393.9 0.2
		As extruded & swaged	-	-	-	-	6,000	2.4
		As extruded & swaged	-	-	-	-	6,000	103

FIGURE A 1

SCHEMATIC FLOW DIAGRAM OF PROCESSING VARIABLES USED IN PREPARING DISPERSION-STRENGTHENED SWER ALLOYS



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