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INFLUENCE OF SEVERAL METALLURGICAL VARIABLES

ON THE TENSILE PROPERTIES OF HASTELLOY N

H. E. McCoy

OAK RIDGE NATIONAL LABORATORY
operated by
UNION CARBIDE CORPORATION
for the
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METALS AND CERAMICS DIVISION

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ABSTRACT

The tensile properties of Hastelloy N have been evaluated after various heat treatments. One vacuum-melted and four air-melted heats were studied. It was found that the vacuum-melted material exhibited good ductility after all heat treatments. Annealing the air-melted material to temperatures in excess of 2150°F brought about significant reductions in the minimum fracture strain exhibited by the alloy. Holding at temperatures of about 1600°F for an extended period recovered the fracture ductility. Aging material in the 1100 to 1200°F range that had been previously annealed at 2150°F brought about a significant reduction in the ductility. These changes in ductility occurred with very small changes in tensile strength.

It is felt that these effects can be explained in terms of the formation of a brittle grain boundary layer along which a crack can propagate easily at elevated temperatures. Interrupting the continuity of this layer by overaging or cold working recovers good fracture ductility. The formation of this layer is associated with the presence of trace alloying elements.

INTRODUCTION

Modern technology depends in many ways upon materials which have the capability of sustaining loads at elevated temperatures in corrosive environments. Because of the variety of service conditions of interest, a number of alloys have been developed with various properties and capabilities. However, because of the complexity of most of these alloys, it is necessary that extensive metallurgical investigations be carried out to determine whether the properties of the alloy remain suitable under the proposed service conditions. Although these studies are costly and time-consuming, it is only after such studies that the alloy can be safely and efficiently utilized.

One such alloy that was developed for a specific application is Hastelloy N. This alloy is nickel-base and was chosen for use in the MSR because it offered good resistance to corrosion by molten-fluoride salts and possessed moderate mechanical strength.[1] The basic properties of this alloy have been investigated and reported previously.[2] However, the use of numerous heats of this alloy under various sets of circumstances has revealed potential problem areas. In order that this alloy might be better utilized for its intended purposes and for other future applications, further studies have been conducted. These studies have been concerned with the influence of the following variables on the properties: solution annealing temperature, specimen orientation, aging, cold working, and carbon content. Tensile tests and metallographic studies have been the principal techniques used to evaluate the influence of the above variables.

EXPERIMENTAL DETAILS

The chemical analyses of several heats of the Hastelloy N used in this investigation are indicated in Table 1. Four of the heats of material were produced by the Stellite Division of Union Carbide Corporation and were received in the form of 1/2-in.-thick plates. One heat of material was obtained from the Allvac Metals Company in the form of 1/2-in.-diam rod. The geometry of the test specimen used is shown in Fig. 1.
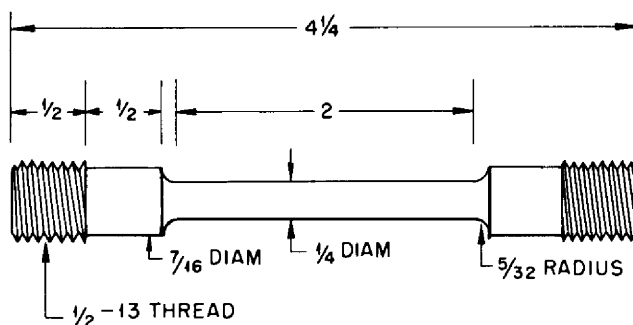
ALL DIMENSIONS ARE IN INCHES

Fig. 1. Geometry of Test Specimen.

Table 1. Chemical Composition (by Weight Percent)
of Several Heats of Hastelloy N

Heat	Vendor	C	S	Mn	Si	Cr	Mo	Co	Ti
2477	Allvac	0.057	0.003	0.04	0.015	7.05	16.32	0.14	0.10
5073	Stellite Division	0.06	0.008	0.47	0.59	6.73	16.09	0.07	0.01
5074	Stellite Division	0.06	0.006	0.45	0.58	6.76	16.28	0.07	0.01
5075	Stellite Division	0.07	0.007	0.50	0.62	6.87	15.95	0.06	0.01
SP-25	Stellite Division	0.05	0.011	0.30	0.21	6.81	16.58	0.49	0.03

Heat	Vendor	Al	B	Fe	Cu	P	W	V
2477	Allvac	0.055	0.0008	4.25	0.10	0.008	0.47	
5073	Stellite Division	0.01	0.006	3.89	0.01	0.004	0.045	0.30
5074	Stellite Division	0.02	0.001	4.05	0.01	0.003	0.04	0.28
5075	Stellite Division	0.01	0.008	3.84	0.01	0.002	0.04	0.26
SP-25	Stellite Division	0.01	0.003	4.10		0.011		

Most of the tensile tests were run in a hydraulic Baldwin testing machine at a crosshead speed of 0.05 in./min or a strain rate of 2.5%/min. A limited number of tests were run in an Instron testing machine at various strain rates. All specimens were tested in air. The furnace used was of the clamshell type and was preheated before being closed around the test specimen. A standard equilibrating time of 1/2 hr was used for all specimens to reach the desired test temperature.

All heat treatments prior to testing were carried out in an argon atmosphere. Unless otherwise indicated, the specimens were cooled from the annealing temperature by pulling them from the hot zone into the

water-cooled end of the furnace tube. Thermocouples were attached to several of the specimens, and the average cooling rate down to 500°F was 200 to 500°F/min.

EXPERIMENTAL RESULTS

Influence of Solution Annealing Treatment

The tensile properties of heat 5075, after annealing 1 hr at 2150 and 2300°F, are given in Table 2 and depicted graphically in Figs. 2 and 3. Both the tensile and yield strengths were lower for the material annealed at 2300°F than for the material annealed at 2150°F except at test temperatures of 1800°F where the reverse was noted. After annealing at 2150°F, the material exhibits a ductility minimum in the temperature range of 1200 to 1400°F. A 2300°F anneal shifts the ductility minimum to 1600°F and reduces the minimum ductility significantly. However, the ductility below 1200°F is not very different for material annealed at 2150 and 2300°F.

The tensile properties of heat 5074 after annealing at 2150 and 2300°F are given in Table 3. Duplicate sets of specimens were run on the Baldwin and Instron testing machines. The strength values obtained on the Instron (hard) machine were consistently higher than those obtained on the Baldwin (soft) machine for the same heat of material. However, the differences are very small and are probably because of slight variations in strain rate. The fracture ductilities did not show any consistent variations. The tensile and yield strengths of heat 5074 are comparable with those of heat 5075. However, the fracture ductility exhibits a different behavior. After annealing at 2150°F, the ductility minimum of heat 5074 occurs between 1400 and 1600°F. After annealing at 2300°F, the fracture ductility is still decreasing with temperature at 1800°F. However, the ductility below 1200°F is not significantly different for material annealed at 2150 and 2300°F.

The properties of heat 5073 in longitudinal and transverse orientations (with respect to rolling direction) are given in Table 4. The strength variations with test temperature and heat treatment are comparable with those just discussed for heats 5074 and 5075. However, the fracture ductility shows a significant difference. After annealing at 2150°F, the

Table 2. Tensile Properties of Hastelloy N

Heat 5075

(Strain Rate: 2.5%/min)

Heat Treatment	Test Temperature (°F)	Yield Strength (psi)	Ultimate Tensile Strength (psi)	Elongation (%)	Reduction in Area (%)
a	75	43,500	113,800	50.0	53.05
a	800	31,300	99,000	55.0	51.00
a	1200	31,500	79,100	30.0	23.00
a	1400	30,400	61,600	25.0	23.36
a	1600	29,700	37,300	30.0	32.22
a	1800	21,400	21,600	41.0	36.50
b	75	40,100	109,900	58.0	51.77
b	800	26,900	93,600	59.5	54.12
b	1200	25,000	71,100	29.5	31.32
b	1400	25,100	50,300	16.0	13.15
b	1600	24,300	38,100	7.5	3.97
b	1800	22,600	22,800	26.0	35.25

[a]Annealed 1 hr at 2150°F in argon, fast cooled.
[b]Annealed 1 hr at 2300°F in argon, fast cooled.

ductility continues to decrease with test temperature through 1800°F. After annealing at 2300°F, the fracture ductility at lower temperatures is not changed significantly, but it decreases very rapidly above 1400°F. The ductility continues to decrease through the highest temperature investigated, 1800°F, where a reduction in area of only 1.6% was obtained.

The tensile properties of heat SP-25 after various heat treatments are given in Table 5. The strength of this heat is slightly less than that of heats 5073, 5074, and 5075. Heat treatments at 2000 and 2300°F resulted in comparable low-temperature rupture ductilities. Both heat treatments also resulted in decreasing fracture ductilities with increasing test temperature with the 2300°F heat treatment yielding lower
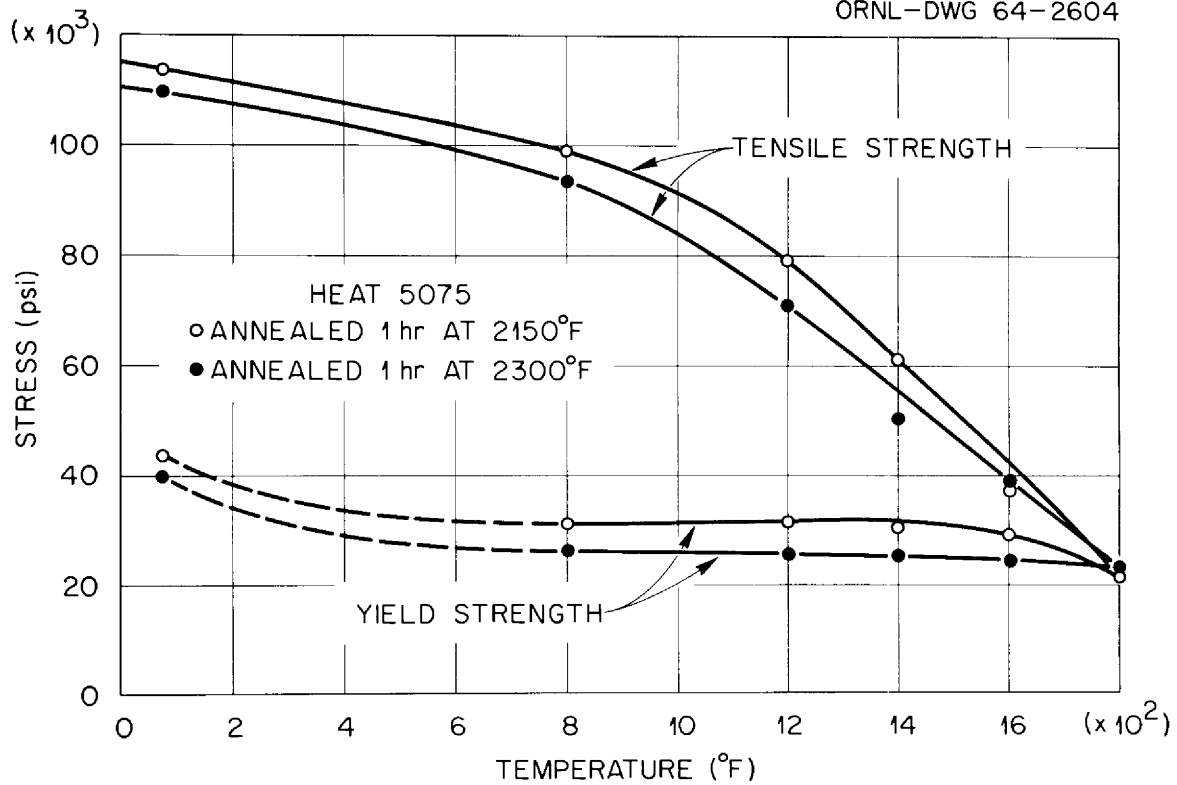
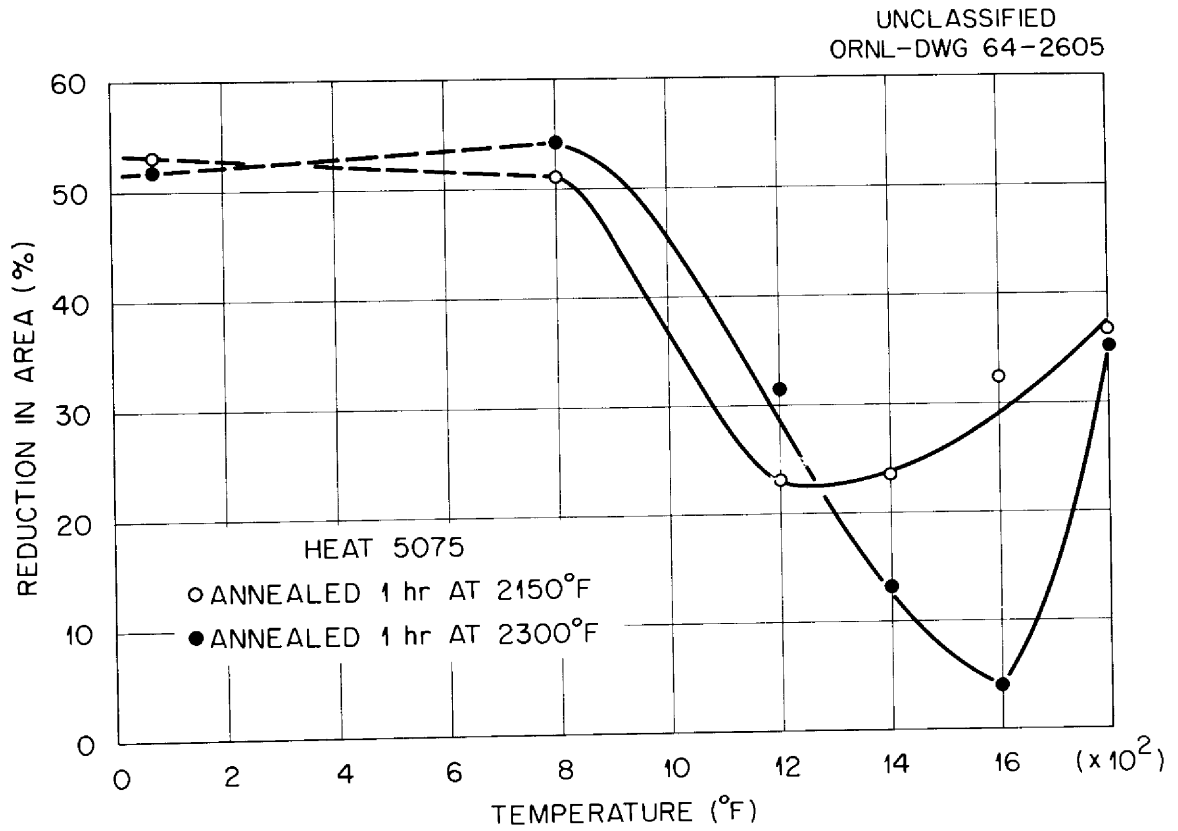Fig. 2. Tensile and Yield Strengths of Hastelloy N (Heat 5075) at Various Test Temperatures.

Fig. 3. Ductility of Hastelloy N (Heat 5075) at Various Test Temperatures.

Table 3. Tensile Properties of Hastelloy N

Heat 5074

Heat Treatment	Test Temperature (°F)	Yield Strength (psi)	Ultimate Tensile Strength (psi)	Elongation (%)	Reduction in Area (%)
		Baldwin Machine			
a	75	47,900	117,100	50.0	48.16
a	800	32,600	98,600	50.0	48.75
a	1200	27,900	75,200	32.5	34.08
a	1400	25,600	56,400	26.0	29.44
a	1600	25,900	36,000	26.0	20.52
a	1800	20,800	20,800	50.5	42.25
b	75	39,500	106,100	62.0	48.16
b	800	29,100	91,700	62.0	51.01
b	1200	25,700	70,200	33.0	41.77
b	1400	25,500	54,800	25.5	23.98
b	1600	24,700	36,100	8.5	4.77
b	1800	21,900	21,900	7.5	3.97
		Instron Machine			
a	75	45,100	124,100	45.4	48.90
a	800	37,600	108,600	48.5	49.47
a	1200	31,700	79,900	28.2	37.29
a	1400	33,700	63,900	32.9	32.39
a	1600	26,300	37,400	36.0	34.88
a	1800	21,700	21,800	57.9	51.38
b	75	40,800	110,200	54.7	50.37
b	800	30,400	96,300	58.1	49.55
b	1200	26,400	76,300	32.9	34.19
b	1400	26,500	61,100	21.7	22.62
b	1600	27,300	37,500	7.27	7.11
b	1800	23,700	24,100	4.64	4.77

[a]Annealed 1 hr at 2150°F in argon, fast cooled.
[b]Annealed 1 hr at 2300°F in argon, fast cooled.

Table 4.   Tensile Properties of Hastelloy N

Heat 5073

Orientation	Heat Treatment	Test Temperature ($^\circ$F)	Yield Strength (psi)	Ultimate Tensile Strength (psi)	Elongation (%)	Reduction in Area (%)
Longitudinal	a	75	43,100	112,100	50.0	44.79
Longitudinal	a	800	31,800	100,000	56.0	39.93
Longitudinal	a	1200	27,900	76,000	36.5	34.20
Longitudinal	a	1400	27,500	60,000	26.0	27.50
Longitudinal	a	1600	27,700	36,300	21.0	20.50
Longitudinal	a	1800	19,800	19,800	10.0	9.41
Longitudinal	b	75	41,100	107,000	61.0	50.05
Longitudinal	b	800	30,400	91,800	61.5	51.18
Longitudinal	b	1200	24,600	71,100	40.0	39.93
Longitudinal	b	1400	24,800	52,300	21.5	24.75
Longitudinal	b	1600	26,100	34.400	8.5	6.30
Longitudinal	b	1800	21,200	21,200	3.0	1.60
Transverse	a	75	43,500	113,800	53.5	50.60
Transverse	a	800	32,000	100,400	54.0	50.60
Transverse	a	1200	27,500	79,700	37.5	36.00
Transverse	a	1400	29,000	62,000	27.5	27.50
Transverse	a	1600	27,500	36,700	27.5	26.14
Transverse	a	1800	20,600	20,600	20.0	13.19
Transverse	b	75	41,100	107,400	54.5	50.60
Transverse	b	800	29,000	90,700	53.5	51.90
Transverse	b	1200	25,500	73,100	37.5	40.54
Transverse	b	1400	26,700	53,800	19.0	24.75
Transverse	b	1600	26,600	35,700	5.8	5.55
Transverse	b	1800	22,400	22,400	6.0	2.40

[a] Annealed 1 hr at 2150°F in argon, fast cooled.
[b] Annealed 1 hr at 2300°F in argon, fast cooled.

Table 5. Tensile Properties of Hastelloy N

Heat SP-25

Heat Treatment	Test Temperature (°F)	Yield Strength (psi)	Ultimate Tensile Strength (psi)	Elongation (%)	Reduction in Area (%)
a	75	42,900	116,700	56.8	48.63
a	800	28,000	99,700	58.0	45.73
a	1200	26,400	73,300	31.6	32.12
a	1400	26,100	61,000	24,0	29.18
a	1600	23,500	36,500	28.0	24.79
a	1800	18,100	18,700	24.8	20.24
b	75	37,600	101,000	64.8	47.56
b	800	24,000	85,700	65.6	43.96
b	1200	20,400	66,800	50.8	37.50
b	1400	21,000	57,100	32.0	25.45
b	1600	24,400	34,800	6.4	6.60
b	1800	20,000	20,000	4.8	3.14
c	75	40,400	112,200	60.0	46.90
c	800	28,000	98,700	62.0	45.45
c	1200	27,200	77,500	40.0	34.08
c	1400	25,700	62,300	50.8	38.51
c	1600	25,200	35,000	60.8	44.55
c	1800	19,500	19,500	28.8	23.73
d	75	38,900	96,600	59.6	36.63
d	800	25,000	79,900	56.8	40.67
d	1200	22,500	61,900	44.8	36.94
d	1400	21,000	49,900	32.0	30.51
d	1600	25,500	35,600	9.60	17.02
d	1800	19,600	19,600	3.20	7.37

[a]Annealed 1 hr at 2000°F in argon, fast cooled.
[b]Annealed 1 hr at 2300°F in argon, fast cooled.
[c]Annealed 100 hr at 2000°F in argon, fast cooled.
[d]Annealed 100 hr at 2300°F in argon, fast cooled.

ductility values. Prolonged heating at 2000 and 2300°F significantly increased the fracture ductility at test temperatures above 800°F. These treatments resulted in very minor changes in strength.

The tensile properties of a heat of vacuum-melted Hastelloy N, heat 2477, are given in Table 6. The first two tests listed in the table indicate that the mill anneal probably did not actually reach 2150°F. This material has strength comparable with that observed for the other heats of material melted in air. The ductility of the mill-annealed material goes through several fluctuations as a function of test temperature but the reduction in area never goes below 35%. A 1-hr anneal at 2300°F slightly reduces the ductility at high temperatures but the minimum reduction in area observed is 26%

Table 6. Tensile Properties of Hastelloy N

Heat 2477

Heat Treatment	Test Temperature (°F)	Yield Strength (psi)	Ultimate Tensile Strength (psi)	Elongation (%)	Reduction in Area (%)
a	75	38,400	109,100	55.5	62.25
b	75	61,900	121,100	45.0	59.42
b	800	46,400	103,500	49.0	54.66
b	1200	43,300	84,500	29.0	35.28
b	1400	42,300	68,600	41.5	39.93
b	1600	31,200	37,100	83.0	78.51
b	1800	20,200	20,900	45.0	50.60
c	75	38,700	104,300	75.0	61.99
c	800	28,000	90,900	70.0	60.93
c	1200	24,400	73,300	50.0	41.03
c	1400	23,400	60,800	29.5	29.54
c	1600	29,300	38,800	45.0	38.95
c	1800	21,200	21,400	33.5	26.23

[a]Annealed 1 hr at 2150°F in argon, fast cooled.
[b]Mill annealed.
[c]Annealed 1 hr at 2300°F in argon, fast cooled.

The individual stress-strain curves for Hastelloy N show several interesting features. Tensile curves at 75°F are quite smooth and appear normal. Curves at 800 to 1600°F exhibit serrations equivalent to as much as 4000 psi and occur at frequencies as rapid as 1 serration/0.025% strain. These serrations vary in magnitude and frequency in the course of a single test or from one test temperature to another. However, the characteristics of the serrations at a given strain rate and temperature are quite reproducible. At 1800°F, the curves are usually quite smooth; the material shows some strain hardening, reaches its maximum load at a very low strain, and then the load continues to decrease during the rest of the test.

Since air-melted Hastelloy N exhibits good ductility after annealing at 2150°F and much lower ductility after annealing at 2300°F, several tests were run to determine how rapidly the ductility decreased with increasing annealing temperature. The results of these tests are given in Table 7. Specimens were annealed 1 hr at temperatures between 2150 and 2400°F and were tested at 1600°F, a temperature in the minimum ductility range. As shown in Table 7, the ductility is significantly reduced by an anneal at 2200°F over that observed after a 2150°F anneal. Annealing at 2250°F brings about a further reduction in ductility, but increasing the annealing temperature to 2300 and 2400°F does not result in further embrittlement. These changes in ductility occur with only small changes in tensile strength.

Table 7. Influence of Annealing Temperature on the
Tensile Properties of Hastelloy N[a]

Heat 5075

Annealing Temperature (°F)	Yield Strength (psi)	Ultimate Tensile Strength (psi)	Elongation (%)	Reduction in Area (%)
2150	29,700	37,300	30.0	32.20
2200	28,100	36,800	11.0	9.33
2250	26,900	35,800	7.5	5.54
2300	27,300	38,100	7.5	3.97
2400	26,300	39,500	6.0	4.78

[a]Annealed 1 hr at indicated temperatures, rapidly cooled; test temperature: 1600°F.

Since the fracture ductility of the air-melted Hastellloy N was rendered quite low by pretest annealing at 2300°F, several notched specimens were tested to determine the influence of a sharp notch on the fracture ductility. The results of these tests are summarized in Table 8. The notch was 0.030-in. deep and had a root radius of 0.001 to 0.0015 in. and an included angle of 30° at the base. These test results, as well as the photomicrograph in Fig. 4, indicate that, after the material has been annealed at 2300°F, fracture can occur at a notch with no measurable plastic strain.

Table 8.   Influence of Notching on the Properties of Hastelloy N
Heat 5073
(Notch Radius is 0.001 to 0.0015 in.)

Heat Treatment	Notched	Test Temperature (°F)	Yield Strength (psi)	Ultimate Tensile Strength (psi)	Elonga-tion (%)	Reduction in Area (%)
a	No	75	43,500	113,800	53.5	50.6
a	No	1400	29,000	62,000	27.5	27.5
a	No	1600	27,500	36,700	27.5	26.1
a	Yes	75	57,800	124,400		15.3
a	Yes	1400	42,700	68,200		8.4
a	Yes	1600	41,400	59,100		4.2
b	No	75	41,100	107,400	54.5	50.6
b	No	1400	26,700	53,800	19.0	24.8
b	No	1600	26,600	35,700	5.8	5.6
b	Yes	75	49,400	115,400		22.8
b	Yes	1400	42,200	58,600		6.4
b	Yes	1600	41,500	44,400		0.0

[a]Annealed 1 hr at 2150°F in argon, fast cooled.
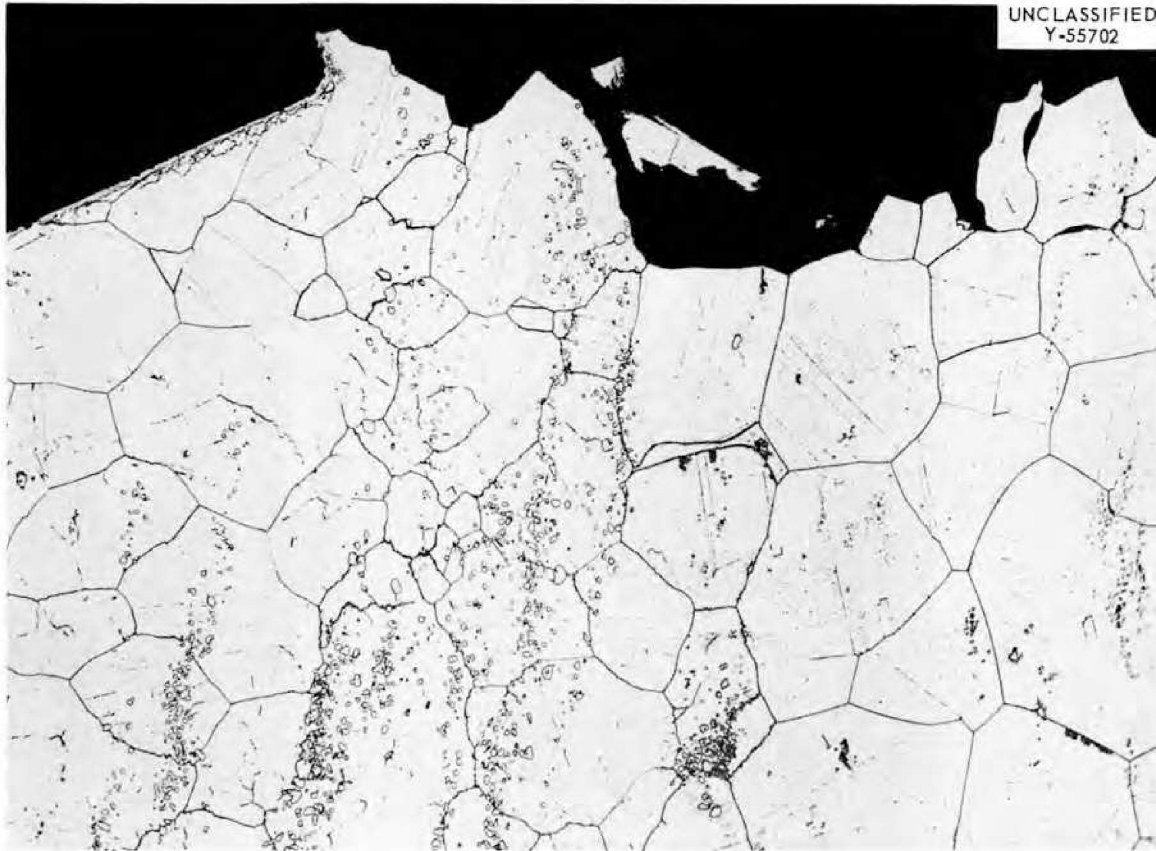[b]Annealed 1 hr at 2300°F in argon, fast cooled.

Fig. 4. Fracture of Notched Hastelloy N Specimen Tested at 1600°F, Annealed 1 hr at 2300°F Prior to Testing. Heat 5073. Etched in glyceria regia. 100×

In an effort to determine whether the changes in ductility brought
about by solution annealing were associated with microstructural changes,
several specimens were examined metallographically. Figures 5, 6, 7, 8,
9, and 10 illustrate that microstructure of heat 5075 after annealing 1 hr
at 2050, 2200, 2250, 2300, 2400, and 2500°F, respectively. The stringers
of precipitate have been tentatively identified by electrolytic extraction
as carbides of the M_6C type. These stringers do not appear to dissolve
at an appreciable rate at temperatures less than 2400°F. At 2500°F, the
discrete precipitate particles have dissolved, but a lamellar product is
present in the grain boundaries. There is also some evidence of melting
during the 2500°F anneal and the inhomogeneous distribution of the molten
areas illustrates the inhomogeniety of this material. The grain sizes
are equivalent after anneals at 2150 and 2200°F. The grain size is in-
creased by an anneal at 2250°F although the grain growth is reduced sig-
nificantly in areas where the stringers are present. Annealing at 2300°F
does not result in a significantly larger grain size than that obtained
at 2250°F. The influence of the stringers on the grain growth has disap-
peared at 2400°F and the grain size is quite large. However, numerous
individual precipitate particles are present which retard the motion of
the grain boundaries and cause them to be quite irregular (see Fig. 9b).
The boundaries also etch rapidly and appear quite broad, indicating that,
possibly, an impurity layer is present. Annealing at 2500°F does not
result in additional grain growth, but the grain boundary layer and pre-
cipitate particles appear to be converted to an intergranular lamellar
product (Fig. 10).

The diamond-pyramid hardness (DPH) of the material after each anneal
is listed with the photomicrographs in Figs. 5 through 10. It is quite
surprising that the hardness decreases with increasing annealing temperature.

Heats 5074, 5073, and SP-25 were also annealed at various temperatures.
The resulting structures were quite similar to those just described for
heat 5075. However, the structure of heat 2477 was quite different.
Figures 11, 12, 13, 14, and 15 illustrate the structure of this material
after annealing 1 hr at 2150, 2200, 2300, 2400, and 2500°F, respectively.
After annealing at 2150°F, the structure contains randomly dispersed
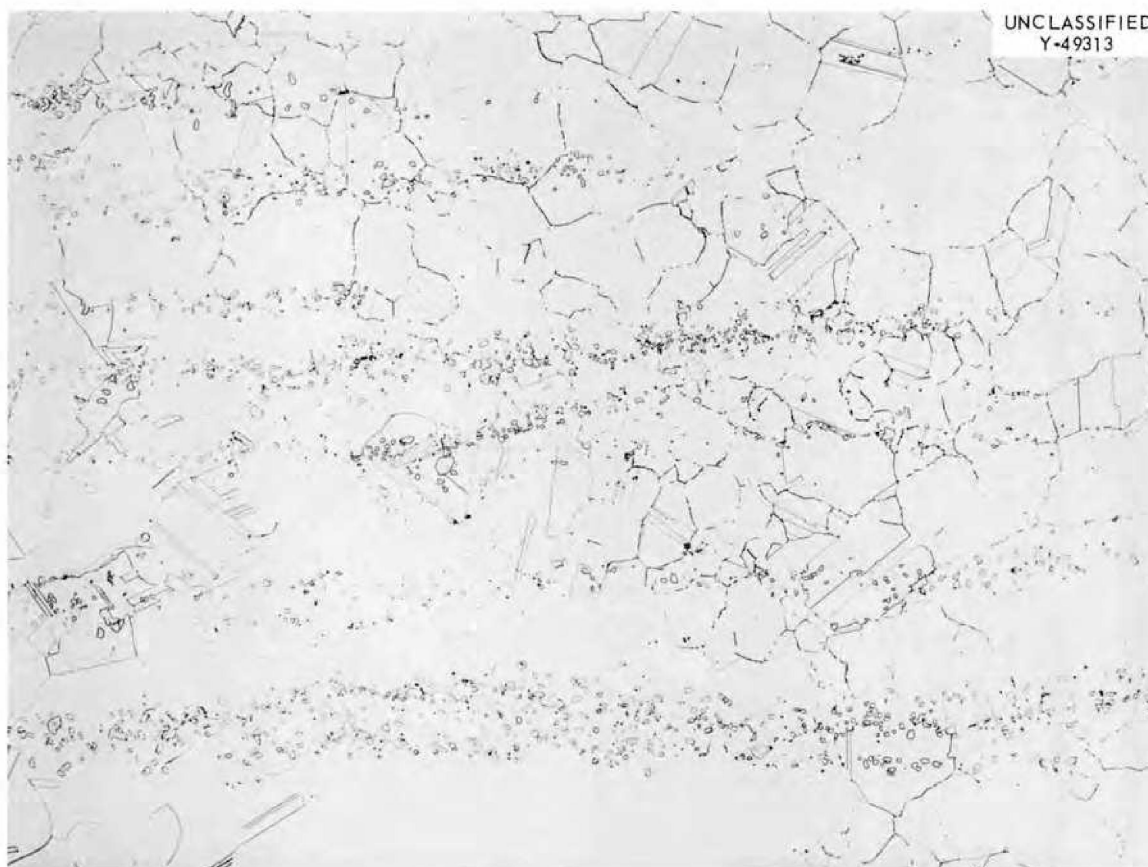precipitates rather than the stringers found in heats 5073, 5074, 5075,

Fig. 5. Photomicrograph of Hastelloy N After Annealing 1 hr at 2150°F in Argon and Rapidly Cooling. Heat 5075. Hardness (DPH) average 178; range 162–193. Etched in glyceria regia. 100x

Fig. 6. Photomicrograph of Hastelloy N Annealed 1 hr at 2200°F in Argon and Rapidly Cooled. Heat 5075. Hardness (DPH) average 185; range 174–213. Etched in glyceria regia. 100×
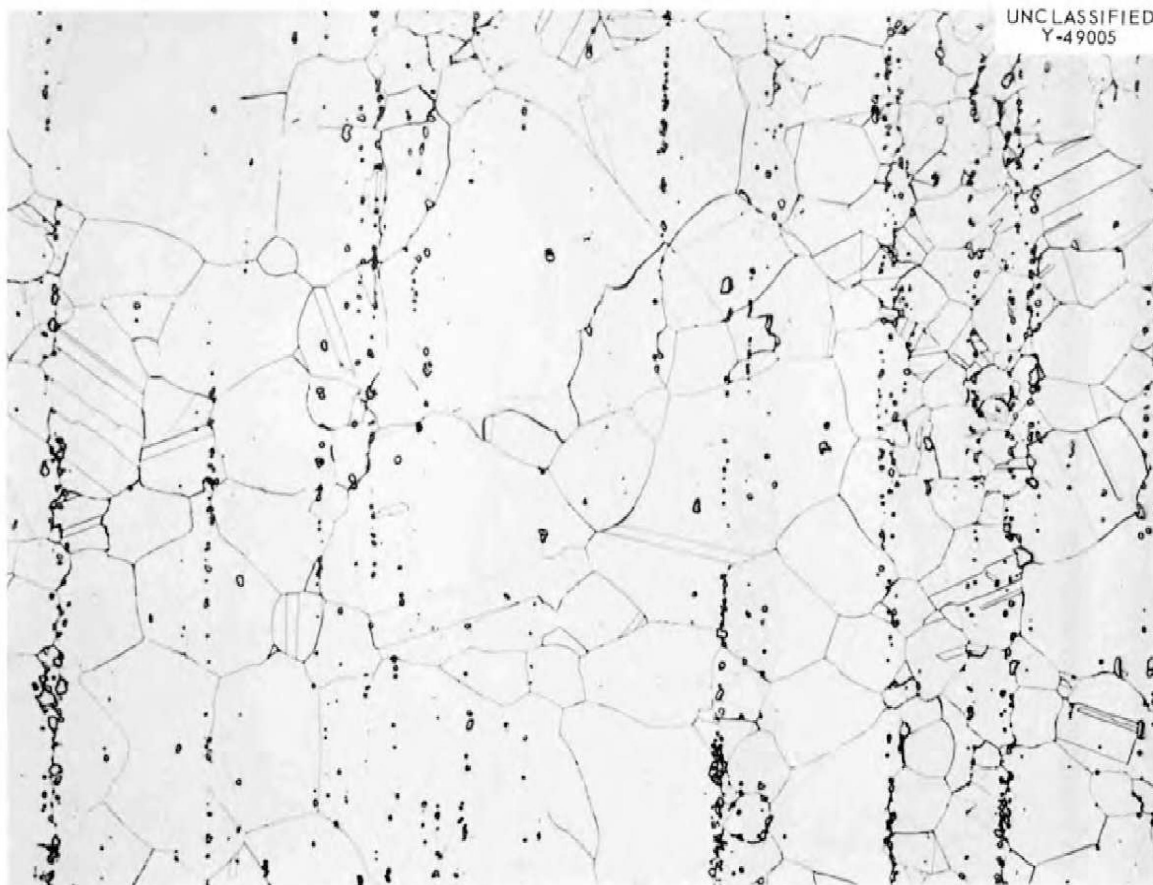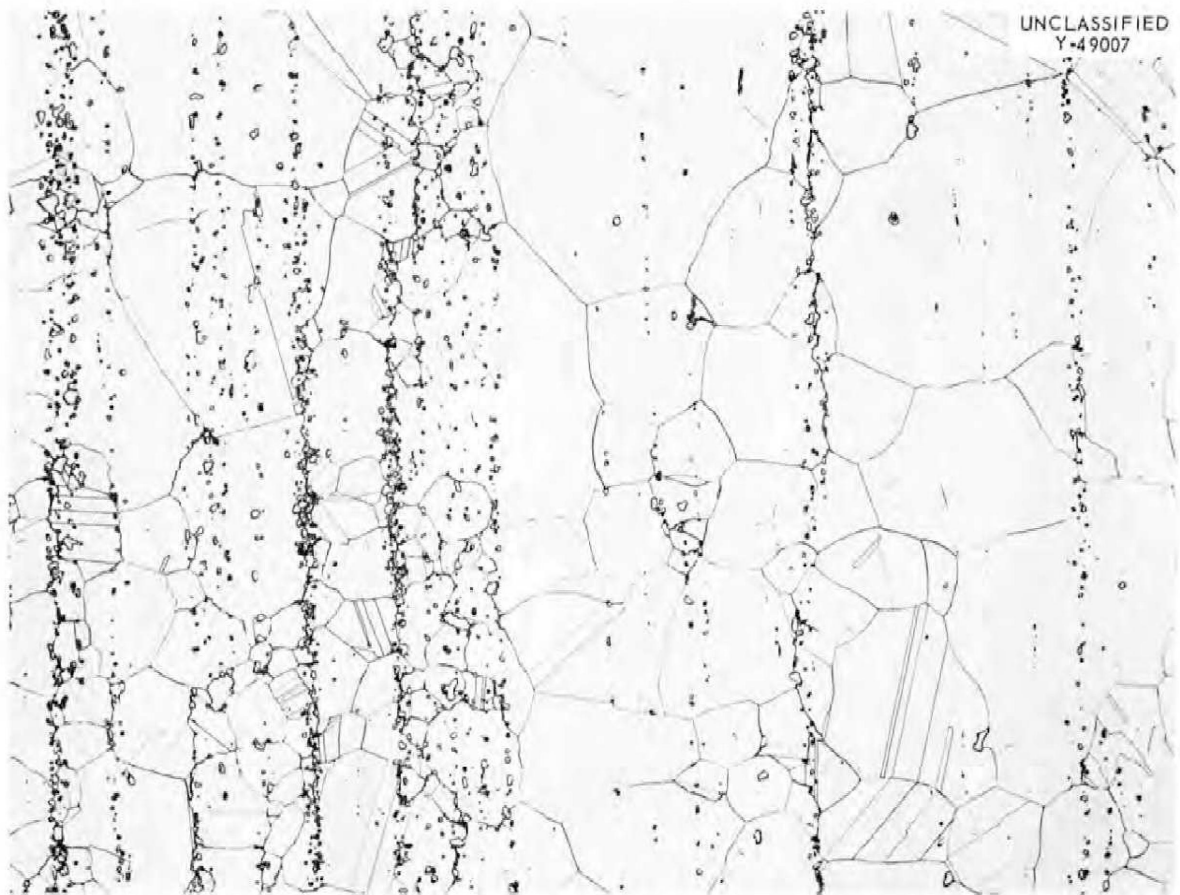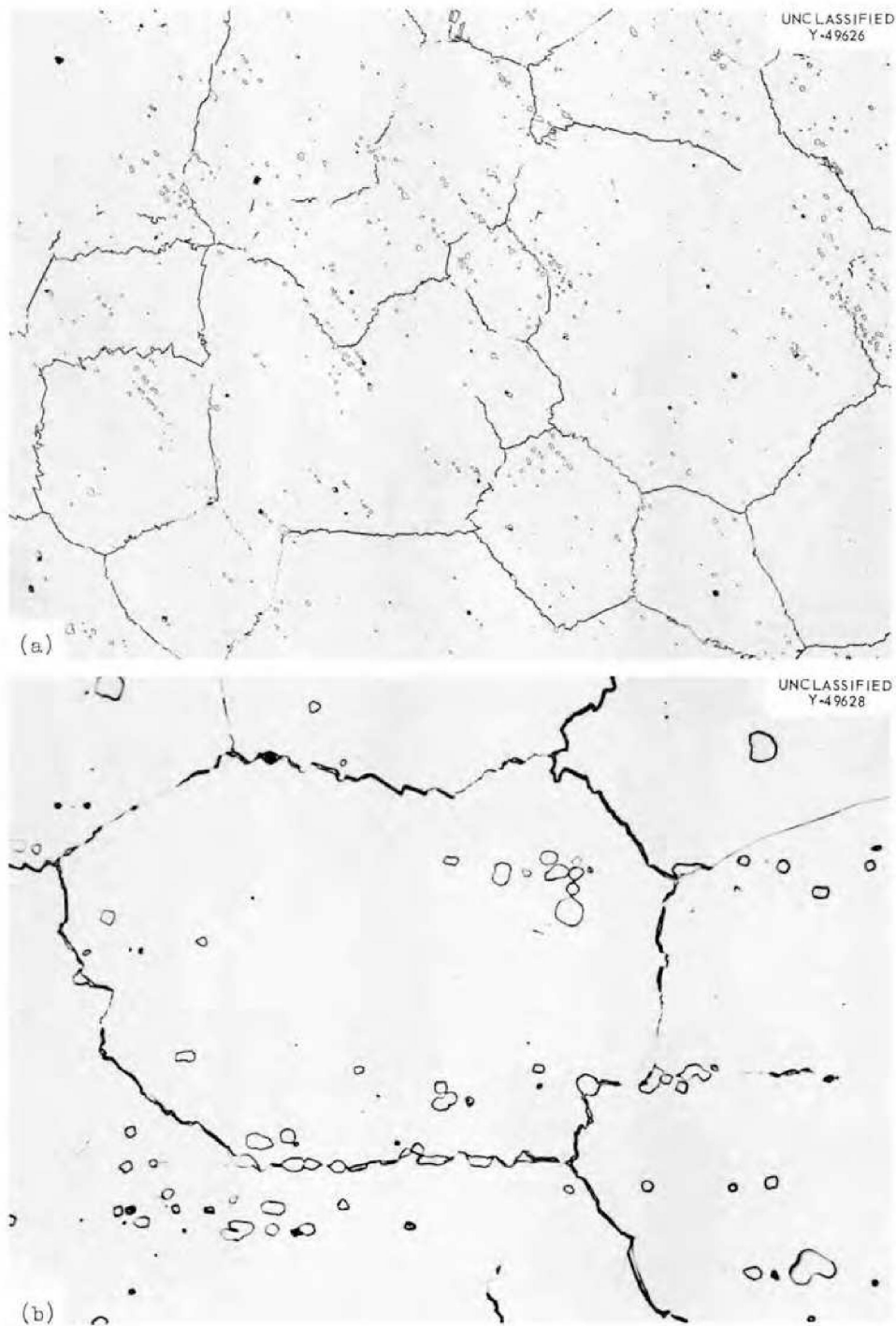
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Fig. 7. Photomicrograph of Hastelloy N Annealed 1 hr at 2250°F in Argon and Rapidly Cooled. Heat 5075. Hardness (DPH) average 179; range 166–196. Etched in glyceria regia. 100×

Fig. 8. Photomicrograph of Hastelloy N Annealed 1 hr at 2300°F in Argon and Rapidly Cooled. Heat 5075. Hardness (DPH) average 175; range 172–182. Etched in glyceria regia. 100×

Fig. 9. Photomicrographs of Hastelloy N Annealed 1 hr at 2400°F in Argon and Rapidly Cooled. Heat 5075. Hardness (DPH) average 168; range 153–175. Etched in glyceria regia. (a) 100X, (b) 500X. Reduced 19%.
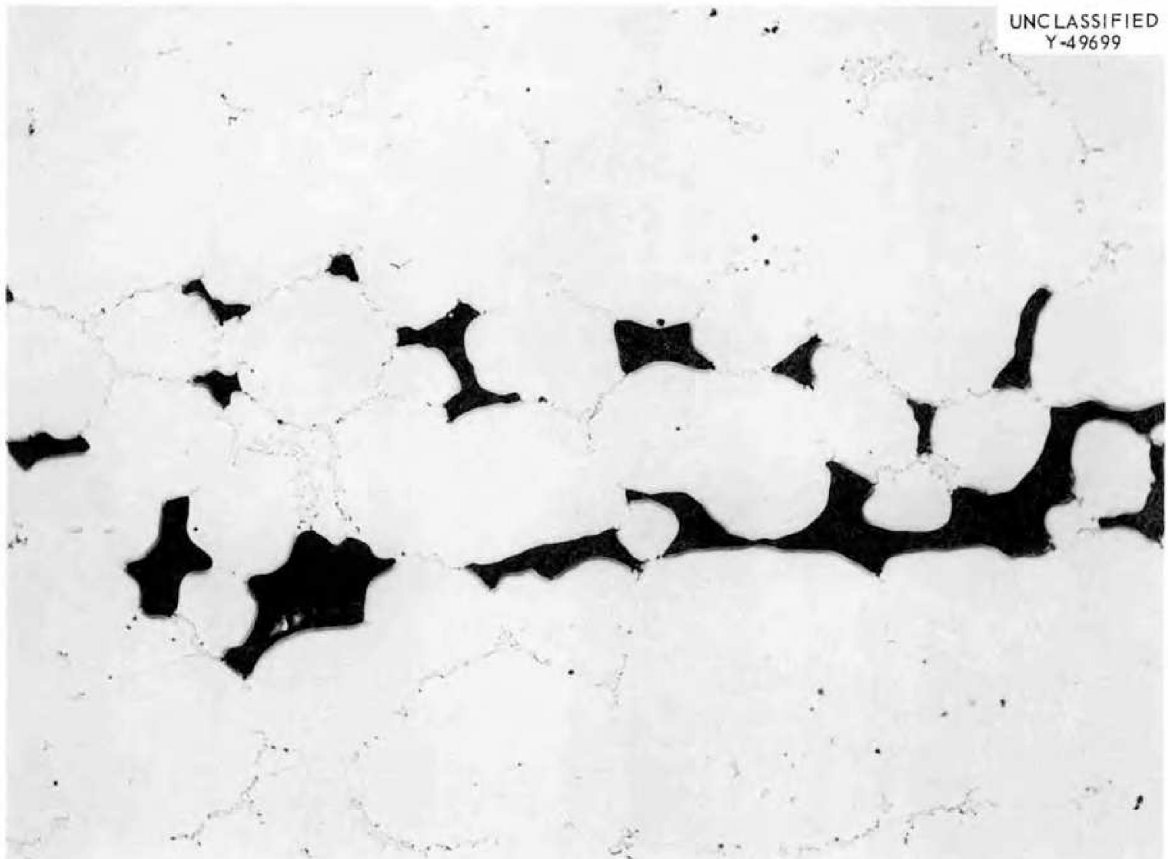
Fig. 10. Photomicrograph of Hastelloy N Annealed 1 hr at 2500°F in Argon and Rapidly Cooled. Heat 5075. Photograph taken with specimen in the as-polished condition. Hardness (DPH) average 149; range 140–159. 100×

Fig. 11. Photomicrograph of Hastelloy N Annealed 1 hr at 2150°F
in Argon and Rapidly Cooled. Heat 2477. Etched in glyceria regia. 100X

Fig. 12. Photomicrograph of Hastelloy N Annealed 1 hr at 2200°F in Argon and Rapidly Cooled. Heat 2477. Etched in glyceria regia. 100×
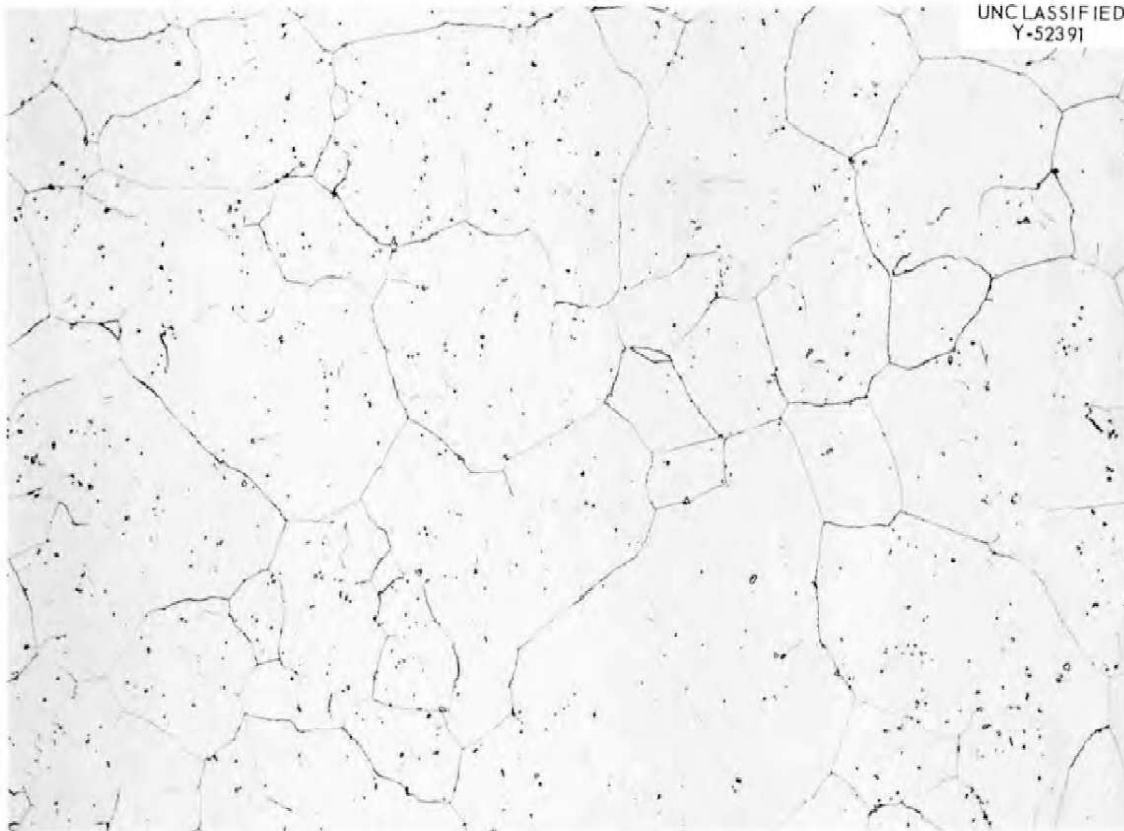
Fig. 13.   Photomicrograph of Hastelloy N Annealed 1 hr at 2300°F
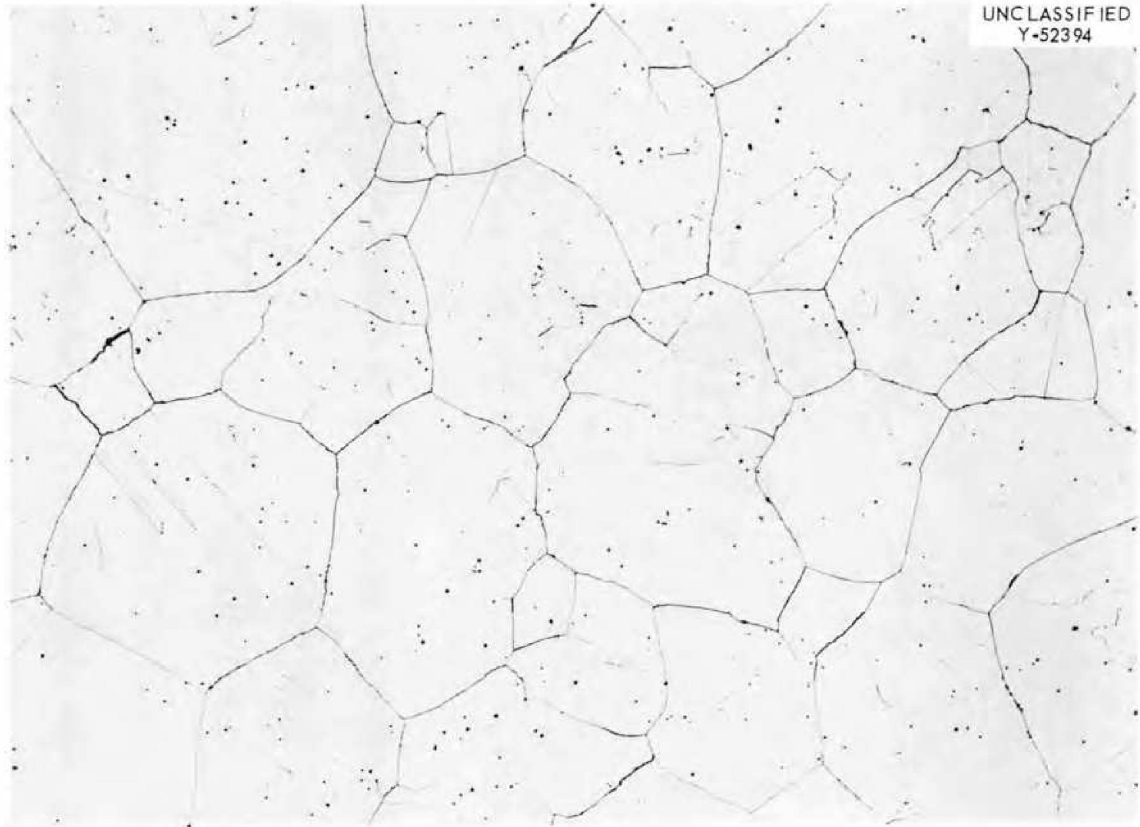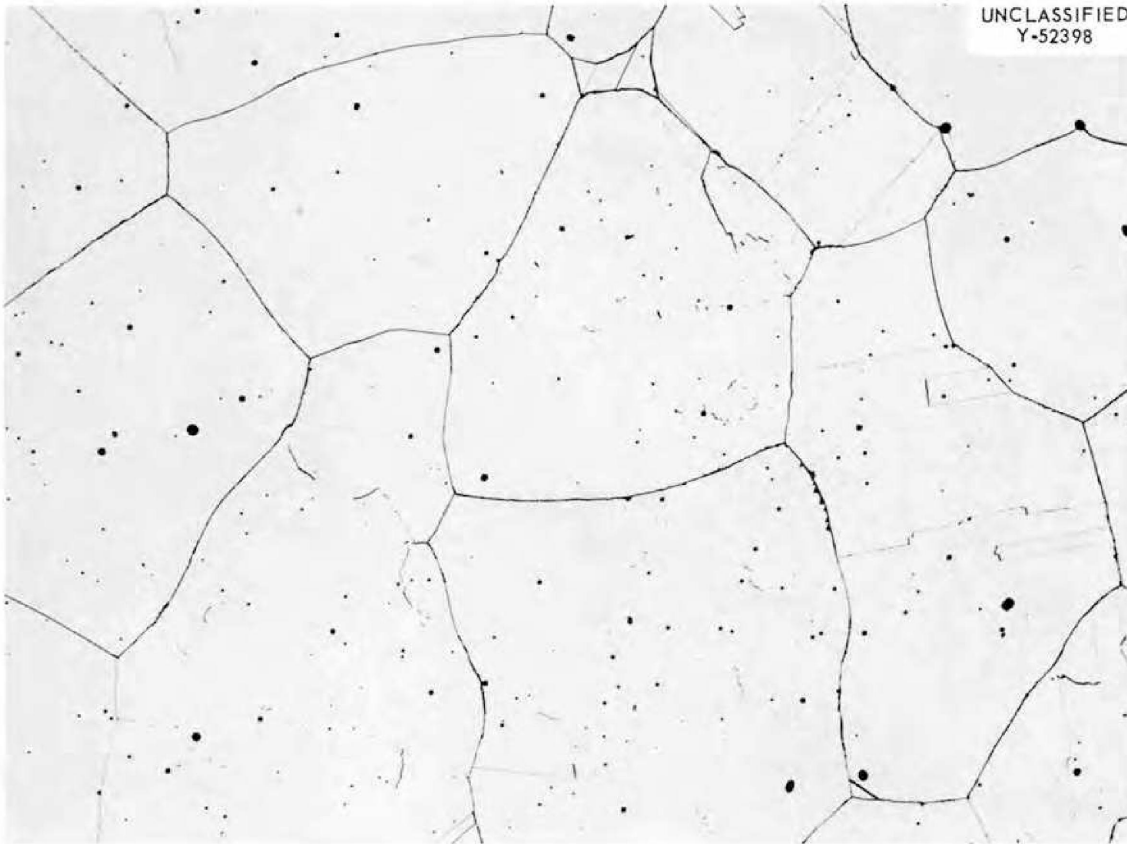in Argon and Rapidly Cooled.   Heat 2477.   Etched in glyceria regia.   100×

Fig. 14. Photomicrograph of Hastelloy N Annealed 1 hr at 2400°F in Argon and Rapidly Cooled. Heat 2477. Etched in glyceria regia. 100×

Fig. 15. Photomicrograph of Hastelloy N Annealed 1 hr at 2500°F
in Argon and Rapidly Cooled. Heat 2477. Etched in glyceria regia. 100×

and SP-25. Annealing at 2200°F causes some grain growth and solution of a significant number of the precipitates. A 2300°F anneal causes some additional grain growth and further solution of precipitate. A 2400°F anneal produces a structure which appears to be single phase with a few inclusions present. The grains are equiaxed and the grain boundaries regular, contrasted with the irregular boundaries shown in Fig. 9 for heat 5075. Annealing at 2500°F produces further grain growth but no intergranular voids indicative of melting are produced. Faint traces of a lamellar intergranular product are visible.

Several of the tested specimens were examined metallographically. Figure 16 shows the fracture of a specimen annealed at 2150°F and tested at 75°F. The fracture was transgranular and most of the small cracks away from the fracture appeared to have been initiated by cracks in the precipitates. The structure resulting from a test at 800°F is quite similar with the exception that more cracks are present. Testing at 1200°F resulted in failure by combined intergranular and transgranular modes. Numerous intergranular cracks are visible away from the fracture, and cracking of the precipitate continues to occur at this temperature. Testing at 1400°F produces an intergranular fracture and numerous intergranular cracks. The precipitate particles do not crack very frequently at this temperature but cracks are initiated by separation at the precipitate-matrix interface. The structure resulting from testing at 1600°F is shown in Fig. 17. It is quite striking that the intergranular cracks are predominantly normal to the applied stress and cover the entire grain width. The structure obtained by testing at 1800°F is shown in Fig. 18. The specimen is heavily cracked and recrystallization has occurred. This specimen exhibited high rupture ductility.

Specimens tested at 1600°F after annealing 1 hr at 2200, 2250, and 2300°F are shown in Figs. 19, 20, and 21, respectively. Although the ductility showed a large reduction as a result of increasing the annealing temperature, there are no striking differences in microstructure except the increase in grain size. However, since the intergranular cracks extend the entire grain width, the change in grain size cannot be ignored. A specimen annealed 100 hr at 2300°F and tested at 1600°F is shown in Fig. 22.
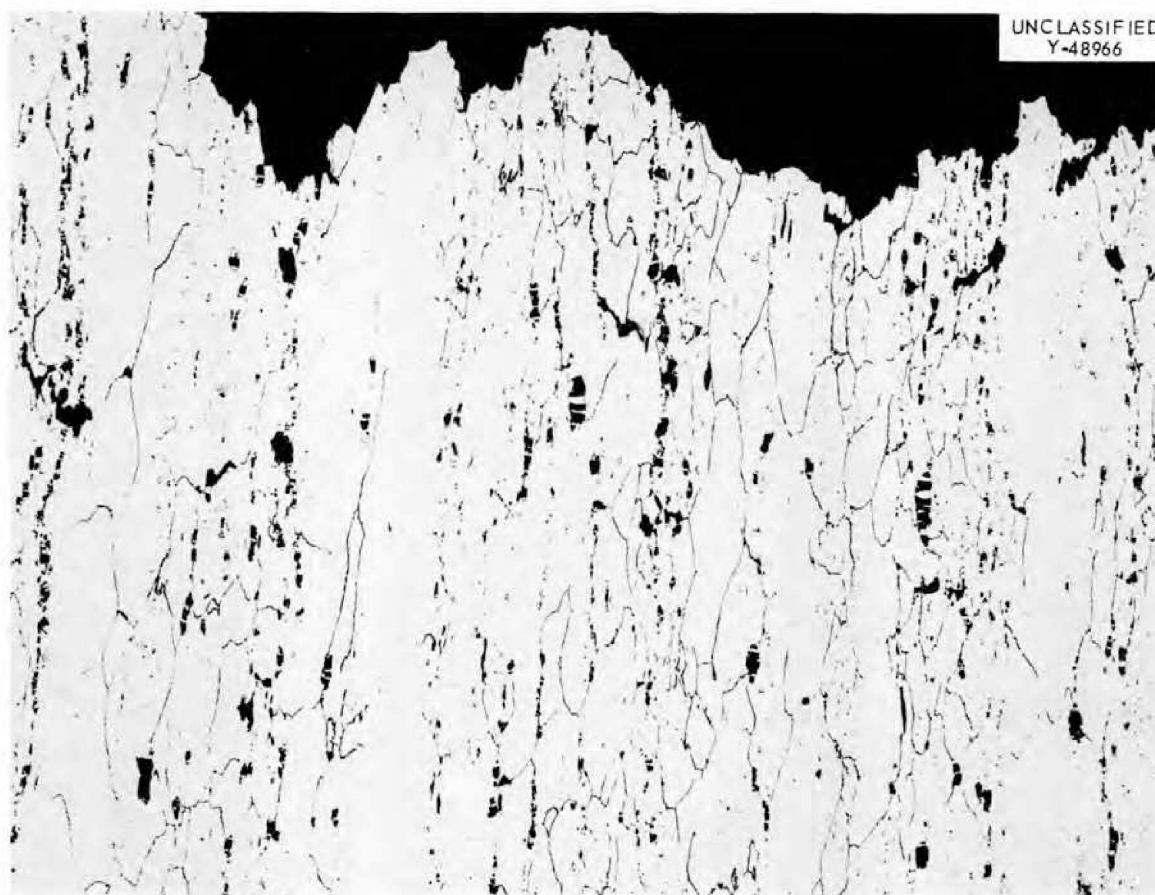
Fig. 16. Fracture of a Hastelloy N Specimen Tested at 75°F and Annealed 1 hr at 2150°F Prior to Testing. Heat 5075. Etched in glyceria regia. 100×
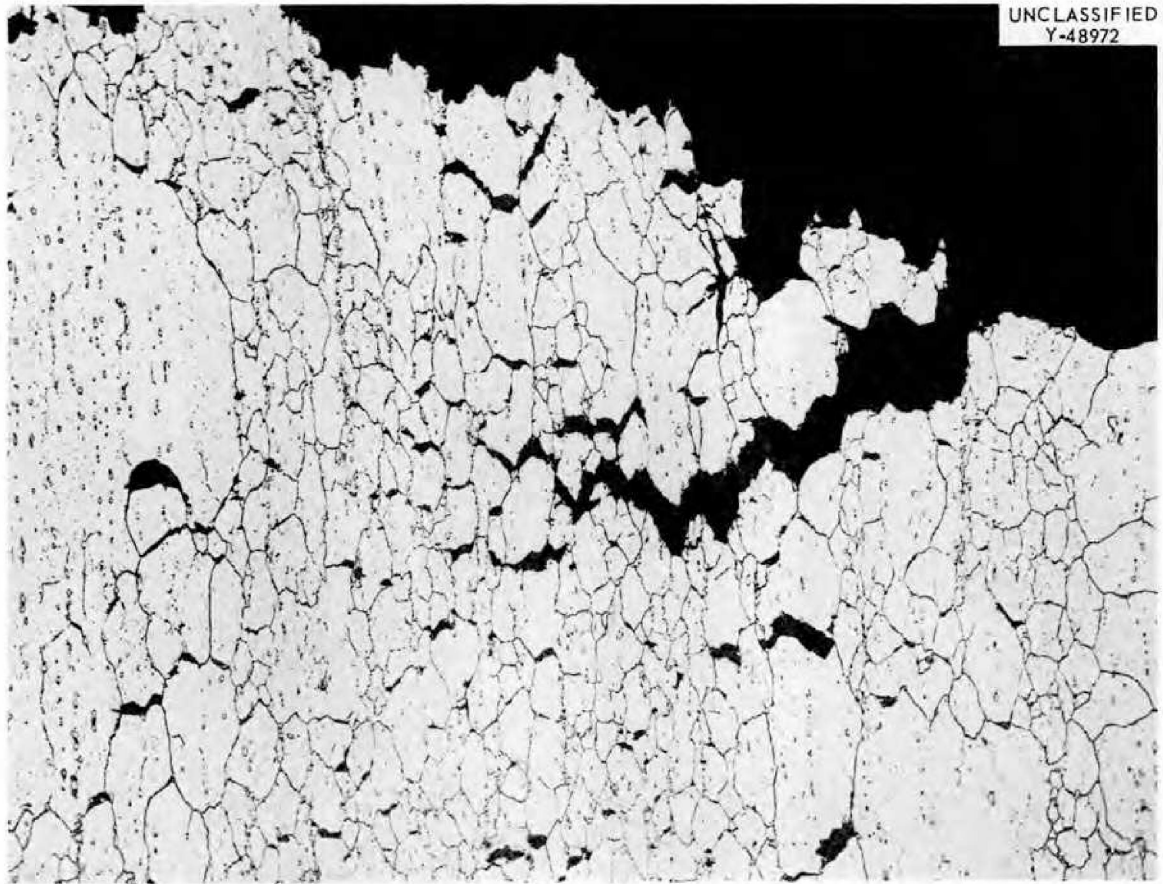
Fig. 17. Fracture of a Hastelloy N Specimen Tested at 1600°F and Annealed 1 hr at 2150°F Prior to Testing. Heat 5075. Etched in glyceria regia. 100×
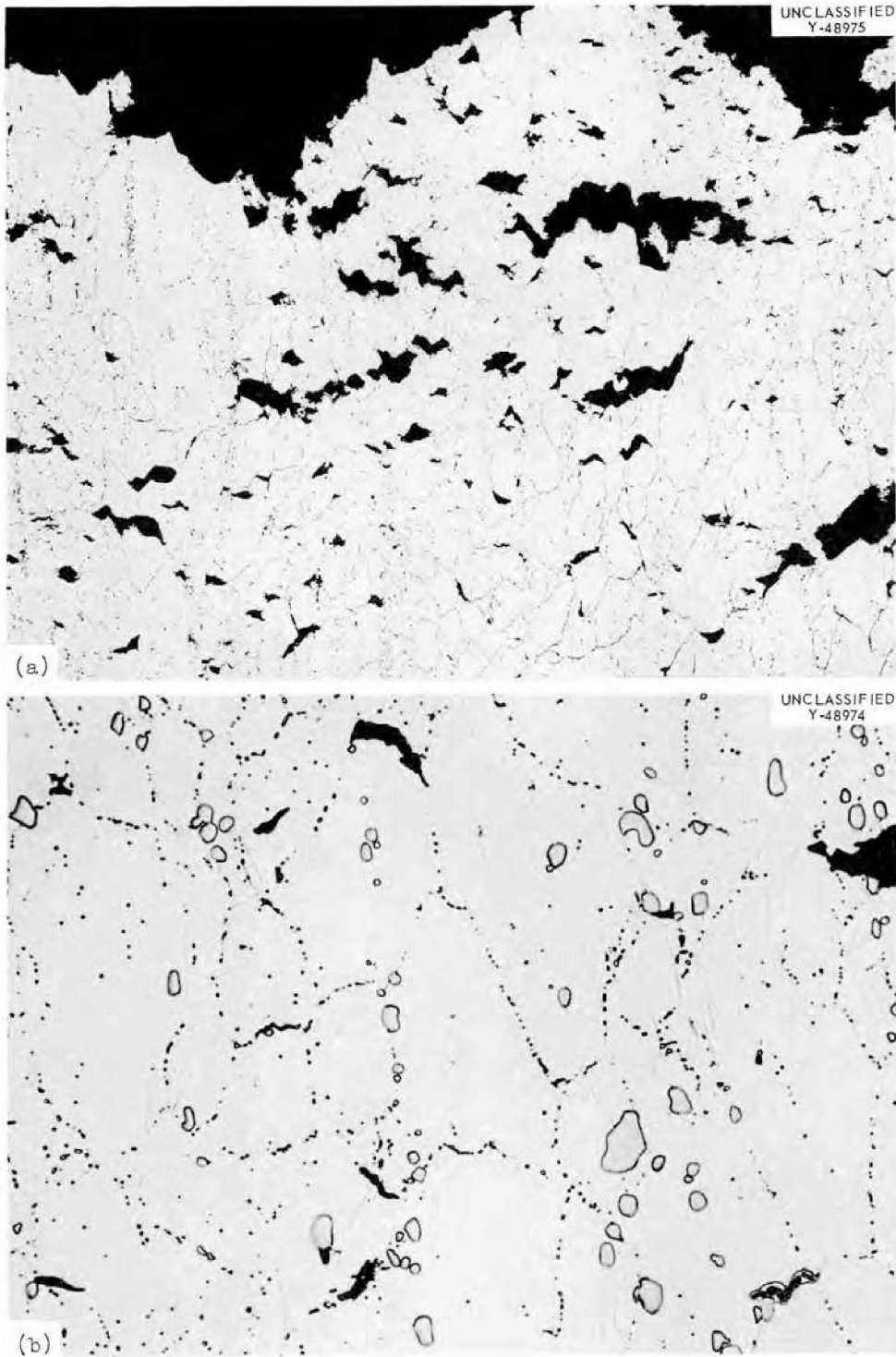
Fig. 18. Hastelloy N Specimen Tested at 1800°F and Annealed 1 hr at 2150°F Prior to Testing. Heat 5075. Etched in glyceria regia. (a) 100×, (b) 500×. Reduced 17%.
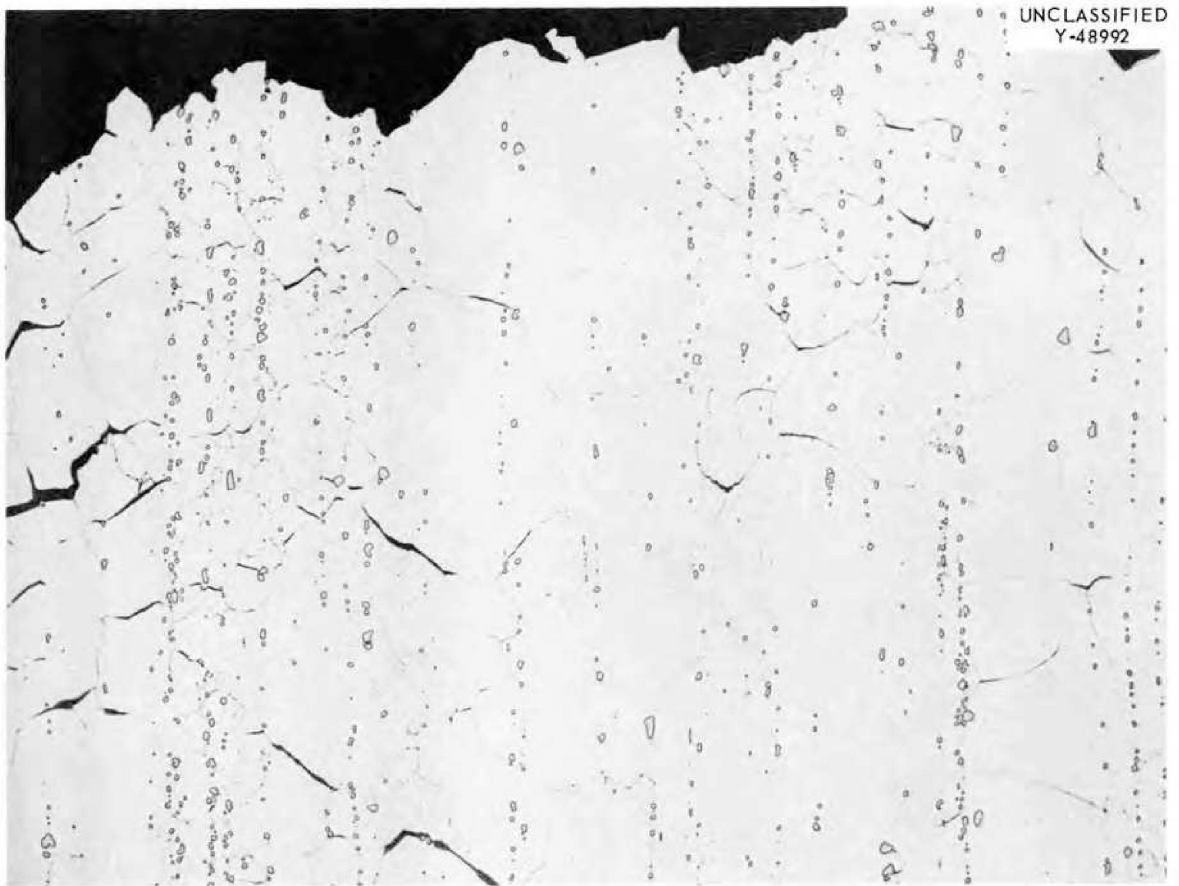
Fig. 19. Fracture of a Hastelloy N Specimen Tested at 1600°F and Annealed 1 hr at 2200°F Prior to Testing. Heat 5075. Etched in glyceria regia. 100×
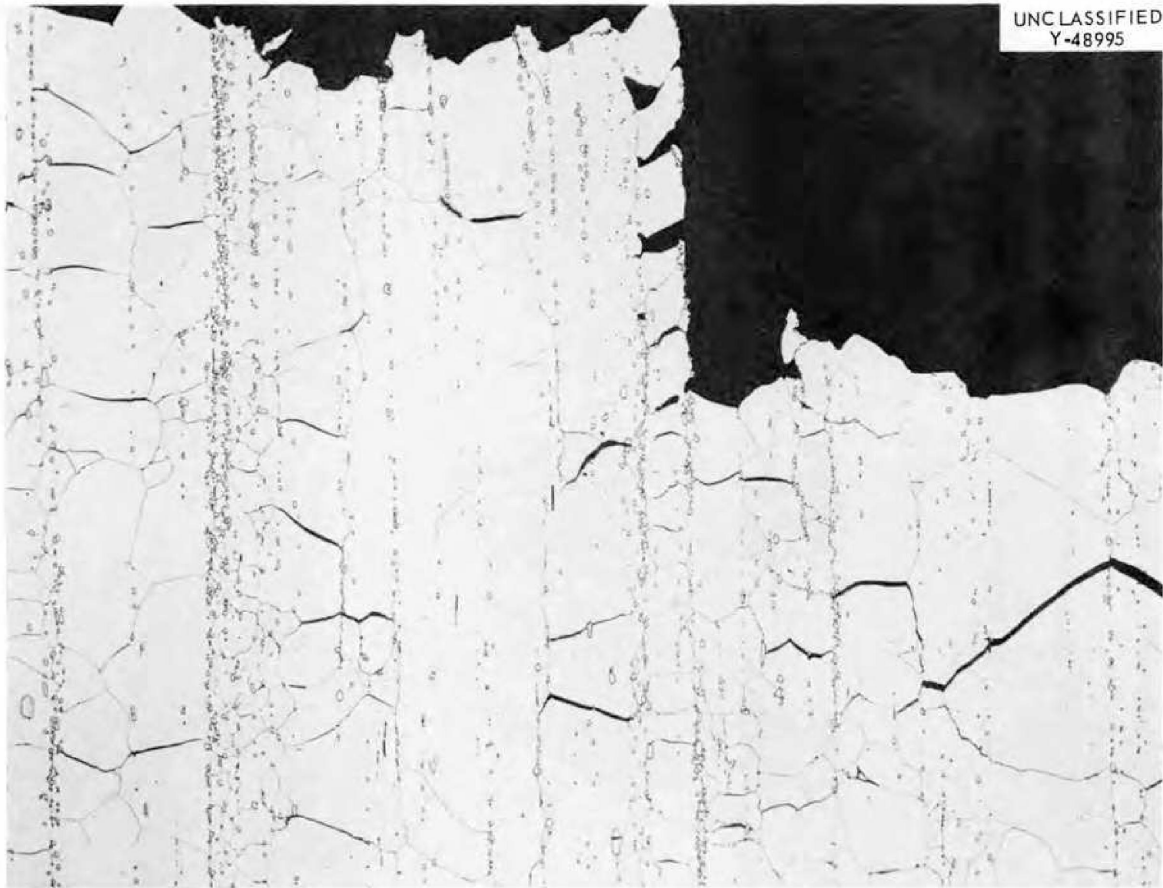
Fig. 20. Fracture of a Hastelloy N Specimen Tested at 1600°F and Annealed 1 hr at 2250°F Prior to Testing. Heat 5075. Etched in glyceria regia. 100×
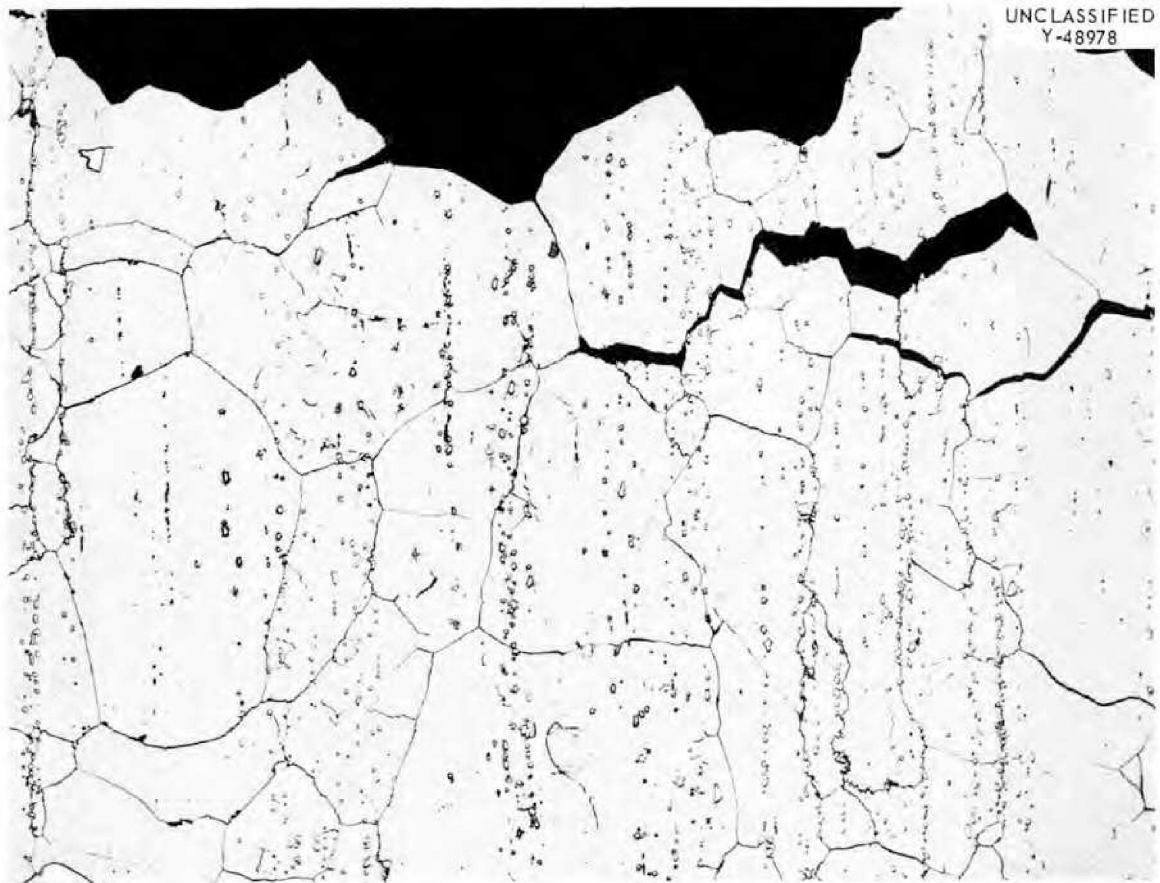
Fig. 21.   Fracture of a Hastelloy N Specimen Tested at 1600°F and Annealed 1 hr at 2300°F Prior to Testing.   Heat 5075.   Etched in glyceria regia.   100×
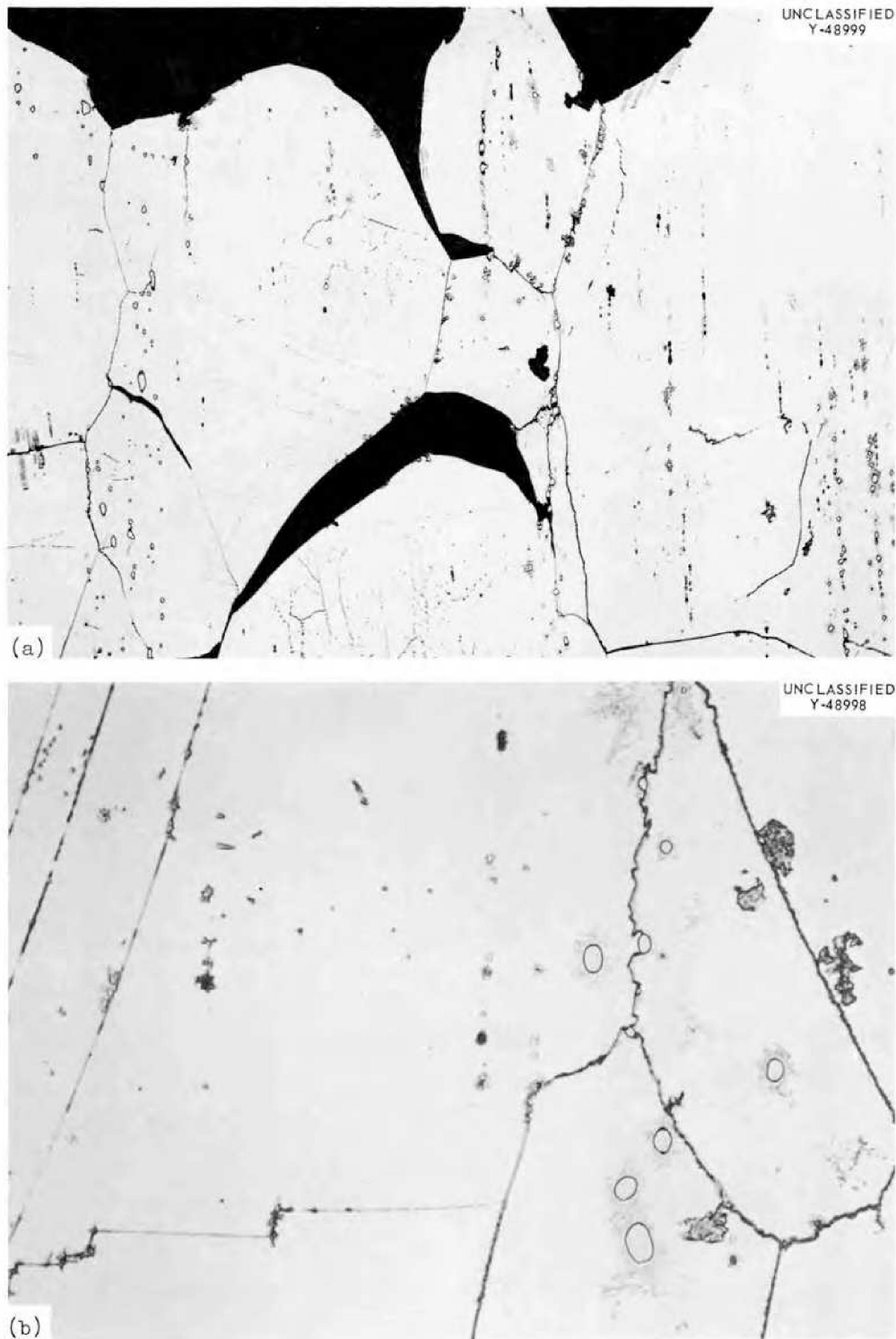
Fig. 22. Hastelloy N Tested at 1600°F and Annealed 100 hr at 2300°F Prior to Testing. Heat 5075. Etched in glyceria regia. (a) 100×, (b) 500× . Reduced 16%.

This anneal has resulted in some solution of the precipitates and the formation of patches of a lamellar grain boundary phase. A substructure is also evident, probably having been decorated by carbon precipitation during the 1600°F tensile test.

Although all of the microstructures used to illustrate the structure of tested specimens have been of heat 5075, the structures of heats 5073, 5074, and SP-25 were quite similar with only one noteworthy exception. The data in Tables 2, 3, 4, and 5 show that some heats under certain conditions went through a ductility minimum and then exhibited good ductility at 1600 to 1800°F. It was found by metallographic studies that this recovery in ductility was always associated with recrystallization. To illustrate, heat 5075 exhibited good ductility at 1800°F after having been annealed at 2150°F and the microstructure in Fig. 18 indicates that recrystallization occurred. Heat 5073T, after being annealed at 2300°F, continued to show decreasing ductility with temperature at 1800°F, and Fig. 23 indicates that recrystallization did not occur during test. Heat 5074 also exhibited poor ductility at 1800°F after a 2300°F anneal and was found not to be recrystallized. However, after a 2150°F anneal, heat 5074 was ductile at 1800°F and Fig. 24 shows that recrystallization did occur during the test.

Tested specimens of heat 2477 (vacuum melted) exhibited structures quite similar to those of the other heats at temperatures up to about 1400°F. Figure 25 shows the microstructure of a specimen annealed at 2300°F and tested at 1400°F. The reduction in area of this specimen was about 30%. Figure 26 shows the microstructure of a specimen annealed at 2300°F and tested at 1600°F. The reduction in area was 39%. Instead of recrystallization occurring, extensive polygonization has resulted. Hence, it appears that the recovery of fracture ductility at elevated temperatures occurs through recrystallization in the air-melted heats and through polygonization in the vacuum-melted heat.

Fig. 23. Microstructure of Hastelloy N Tested at 1800°F and Annealed 1 hr at 2300°F Prior to Testing. Heat 5073T. Etched in glyceria regia. 100X

Fig. 24. Fracture of Hastelloy N Tested at 1800°F and Annealed 1 hr at 2150°F Prior to Testing. Heat 5074. Etched in glyceria regia. 100×
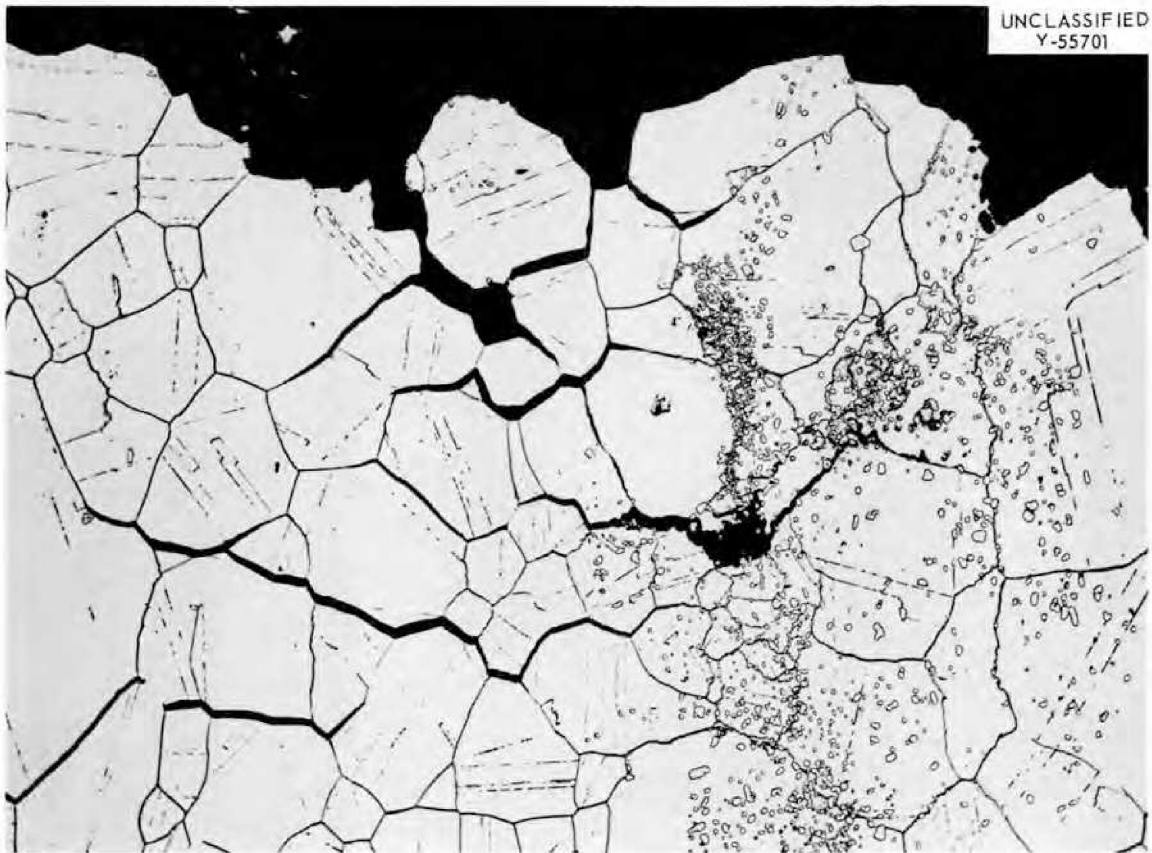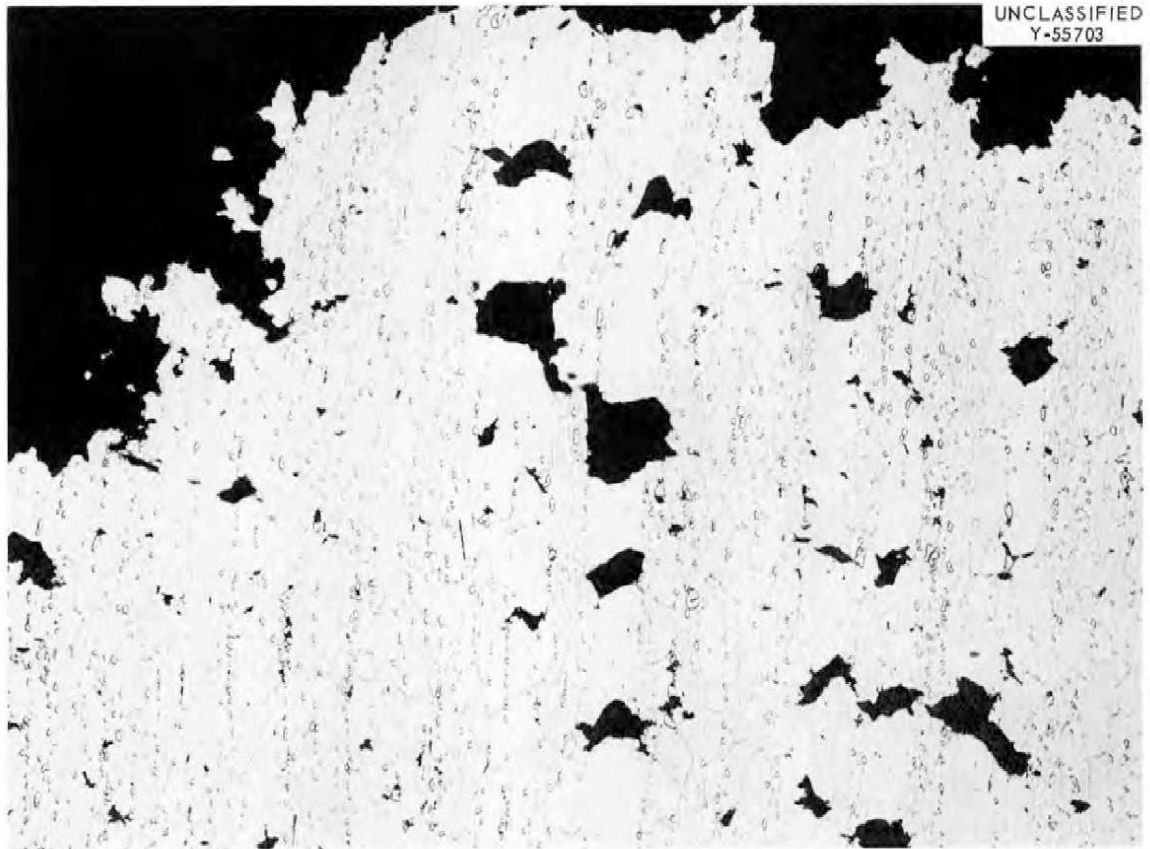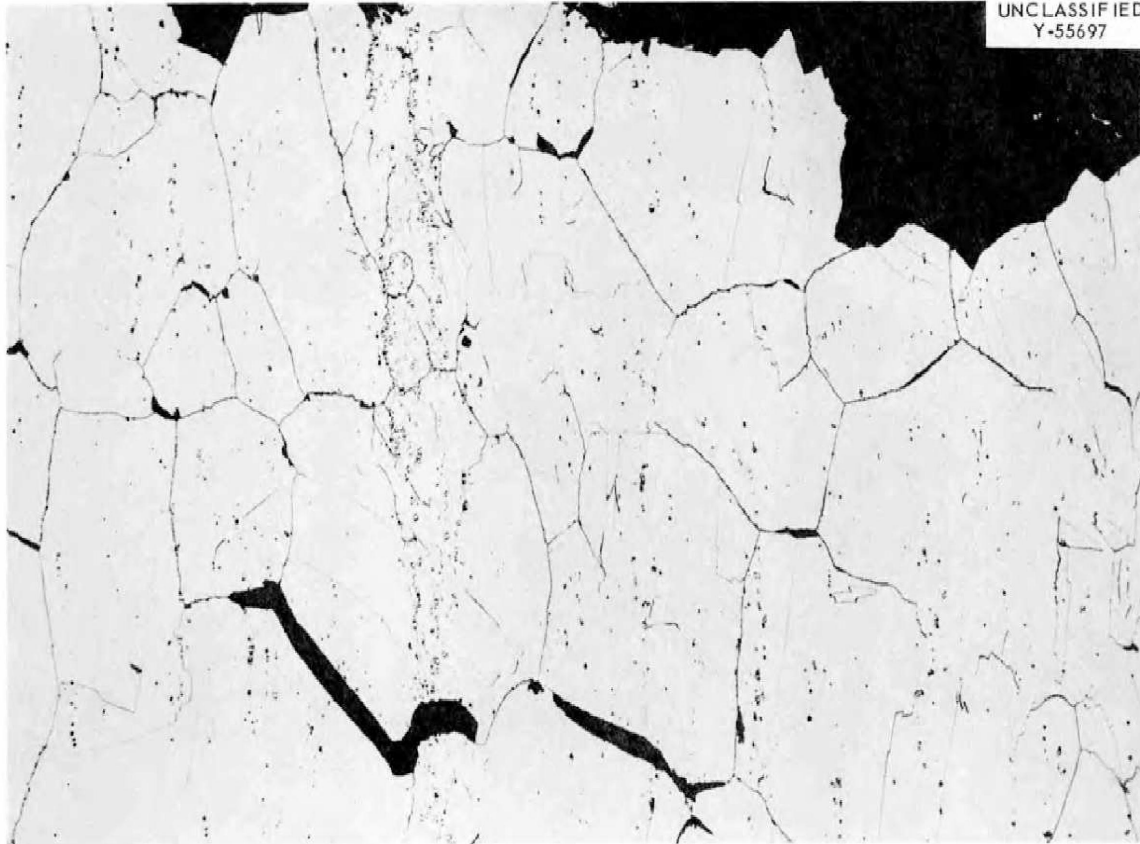
Fig. 25.   Fracture of Hastelloy N Tested at 1400°F and Annealed 1 hr at 2300°F Prior to Testing.   Heat 2477.   Etched in glyceria regia.   100×
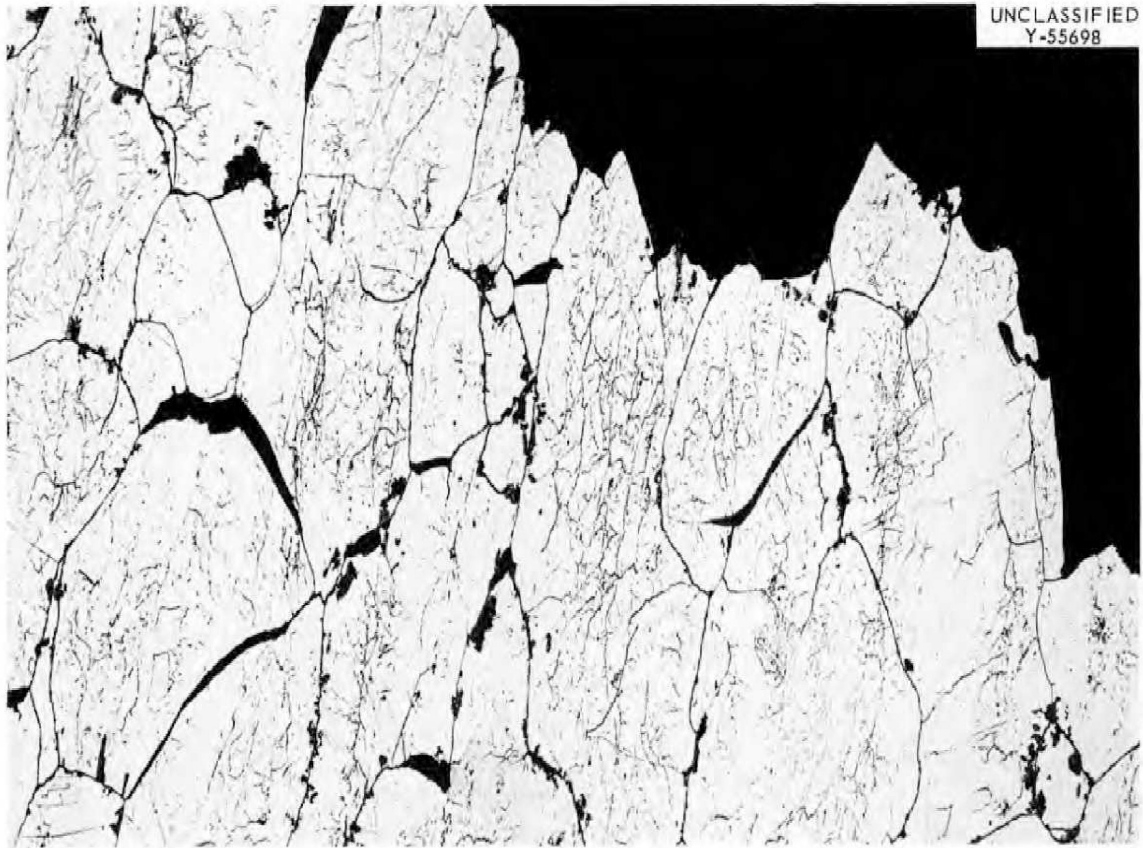
Fig. 26.   Fracture of Hastelloy N Annealed at 1600°F Prior to Testing.
Heat 2477.   Etched in glyceria regia.   100X.

Orientation

Because of the stringers that were present in the air-melted heats, it was thought probable that the properties might be quite different in directions normal and parallel to the stringers. The tests summarized in Table 4 for heat 5073 indicate that the tensile properties of this material are not influenced by specimen orientation.

Influence of Cooling Rate

The normal cooling rate used after the pretest anneal was about 500°F/min down to about 500°F. Several tests were run to determine what influence varying the cooling rate had on the tensile properties. The results shown in Table 9 indicate that the fracture ductility is improved by reducing the cooling rate from the pretest annealing temperature of 2300°F. The microstructure of the specimen cooled at a rate of 2°F/min is shown in Fig. 27. This structure should be compared with that shown in Fig. 21 where the cooling rate was about 500°F/min. The flow lines in Fig. 27 are quite significant and indicate that the material was able to absorb large amounts of plastic deformation.

Table 9. Effect of Cooling Rate on the Tensile Properties
of Hastelloy N

Heat 5075

(All specimens annealed 1 hr at 2300°F and cooled
at indicated rate; tested at 1600°F)

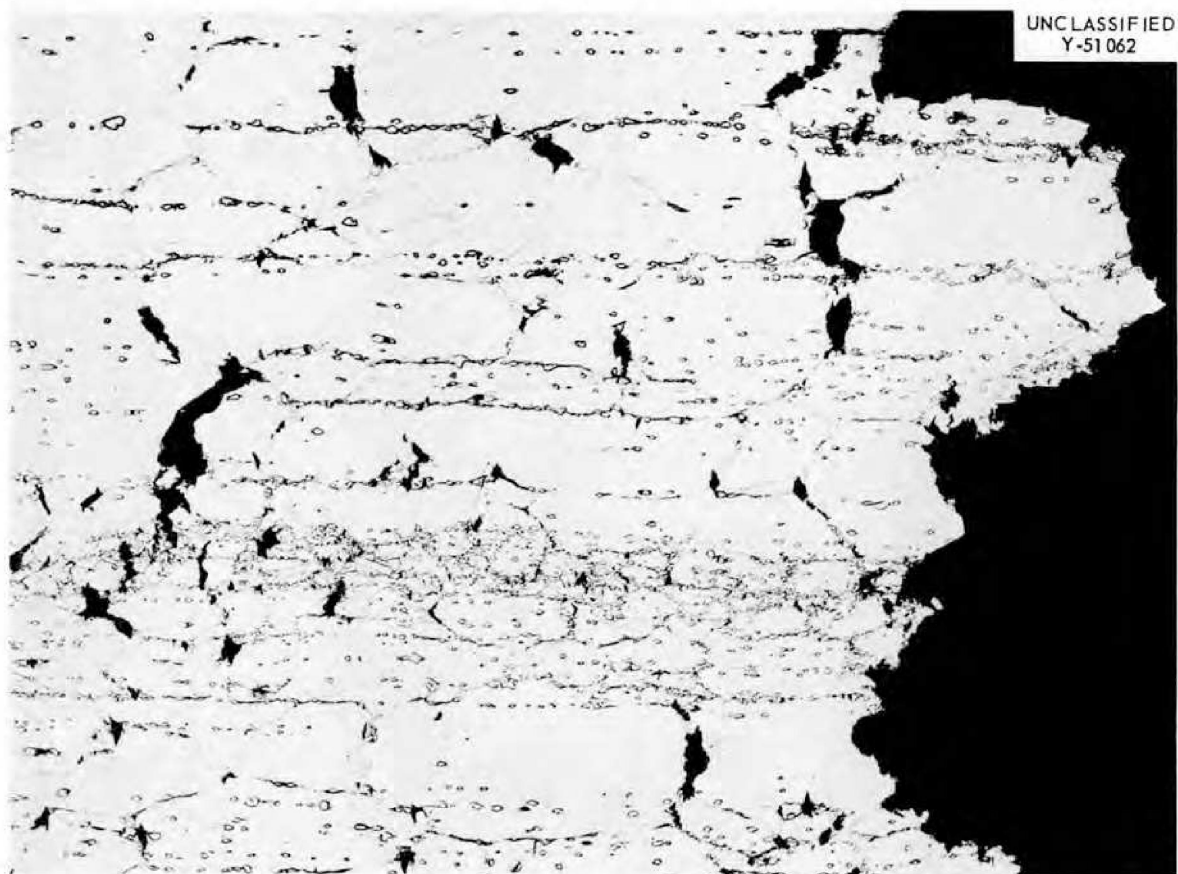| Cooling Rates (°F/min)	Yield Strength (psi)	Ultimate Tensile Strength (psi)	Elongation (%)	Reduction in Area (%)
200–500	27,300	38,100	7.5	3.97
8.1	22,800	37,700	32.0	26.32
4.2	22,400	36,700	39.0	34.08
2.0	22,800	37,700	32.0	26.32

Fig. 27. Fracture of a Hastelloy N Specimen Tested at 1600°F;
Annealed 1 hr at 2300°F and Cooled at a Rate of 2°F/min Prior to Testing.
Heat 5075. Etched in glyceria regia. 100X

Influence of Cold Working

Since high-temperature pretest anneals which produce low fracture
ductilities at 1200 to 1800°F do not lower the ductility at 75°F, experi-
ments were run to see what influence working at 75°F would have on the
ductility in the 1200 to 1800°F range. Typical results are shown in
Table 10. The improvement in fracture ductility brought about by pre-
straining at 75°F is quite significant. A photomicrograph of the specimen
strained 20% is shown in Fig. 28. The microstructure is quite similar to
that of a specimen which had not been prestrained (Fig. 21). The only
significant differences are the flow lines present in the prestrained
specimen.

Table 10. Influence of Cold Working on the Tensile Properties
of Hastelloy N

Heat 5075

(Annealed 1 hr at 2300°F in argon, rapidly cooled;
prestrained indicated amount at 75°F;
test temperature: 1600°F)

Prestrain (%)	Yield Strength (psi)	Ultimate Tensile Strength (psi)	Elongation (%)	Reduction in Area (%)
0	27,300	38,100	7.5	3.97
5	34,000	35,200	10.5	10.04
20	33,100	34,100	26.5	26.14

Influence of Carbon Content

Several specimens were decarburized in H_2-H_2O gas to determine the
influence of carbon content on the fracture ductility. Typical results
are shown in Table 11. A reduction in carbon content from 0.068 to
0.015 wt % resulted in very small increases in the fracture ductility at
both 75 and 1600°F; the strength was also reduced slightly. The micro-
structure was apparently not modified by the loss of carbon. Hence, it

Fig. 28. Fracture of Hastelloy N Specimen Tested at 1600°F, Annealed 1 hr at 2300°F, and Strained 20% at 75°F Prior to Testing. Heat 5075. Etched in glyceria regia. 100×

Table 11. Influence of Carbon Content on the Tensile Properties
of Hastelloy N

Heat 5075

(Annealed 1 hr at 2300°F in argon, rapidly cooled)

Carbon Content (wt %)	Test Temperature (°F)	Yield Strength (psi)	Ultimate Tensile Strength (psi)	Elongation (%)	Reduction in Area (%)
0.068	75	40,100	110,000	58.0	51.77
0.015	75	40,200	104,000	61.5	59.75
0.068	1600	27,300	38,100	7.5	3.97
0.015	1600	24,400	34,900	8.5	13.27

seems that the carbon concentration would have to be reduced to very low
levels in order to improve the ductility significantly.

Influence of Aging

Since annealing studies had shown that all of the precipitate could
be dissolved in heat 2477, the possibility of embrittlement as a result
of reprecipitation was considered.   Specimens were annealed at 2400°F and
aged for 100 hr at 1500, 1700, and 1900°F.   At 1500°F, the structure shown
in Fig. 29 resulted.   At 1700°F, some precipitation occurred along twin
boundaries, and at 1900°F no visible reprecipitation occurred.

Several tensile tests were run to determine whether this reprecipi-
tation altered the tensile properties.   The results of these tests are
summarized in Table 12.   Aging for 100 hr at 1200°F had very little, if
any, effect on the tensile properties.   However, cold working the speci-
mens prior to aging at 1200°F increased the strength by an amount which
increased with increasing cold working.   The resulting structure after
annealing at 2400°F, cold working 10%, and testing at 1400°F is shown in
Fig. 30.   The precipitate formed under these conditions is too fine to be
resolved at a magnification of 1000 diameters.   Annealing for 100 hr at
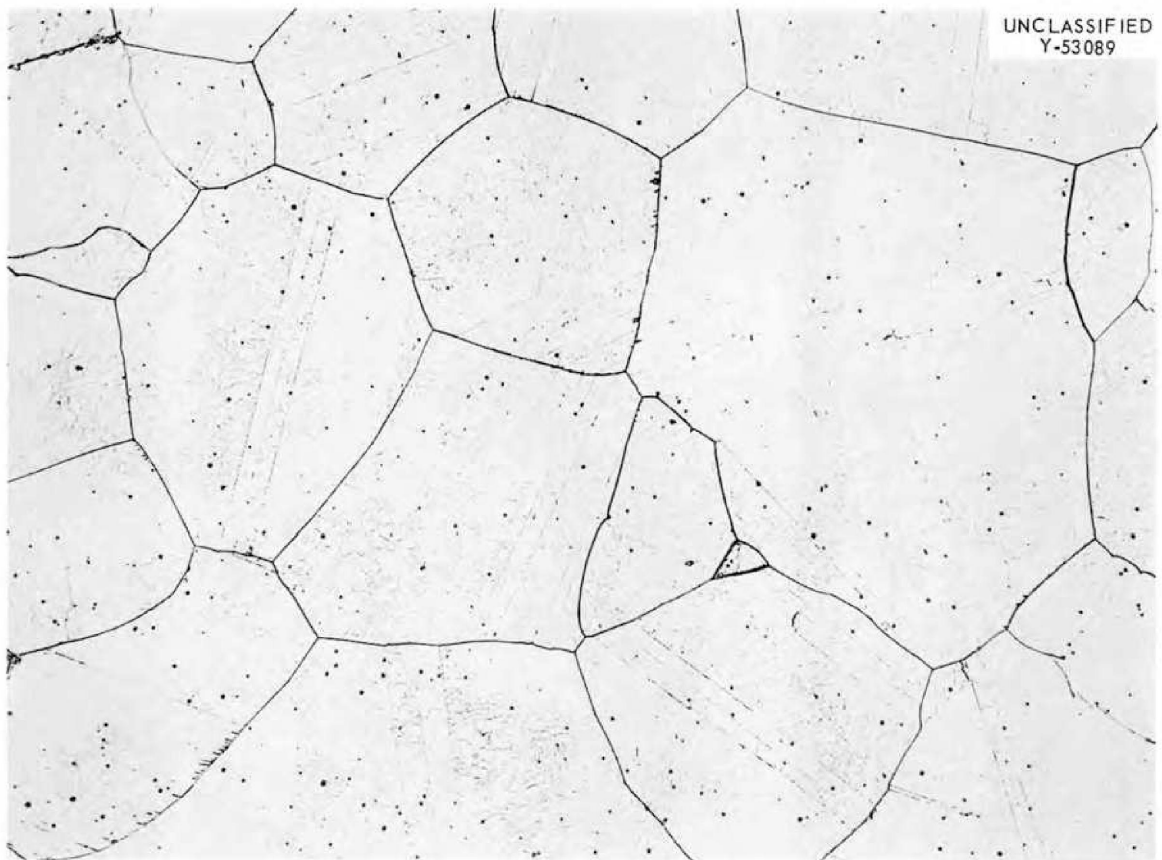
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Y-53089

Fig. 29. Photomicrograph of Hastelloy N Annealed 1 hr at 2400°F and Aged 100 hr at 1500°F. Heat 2477. Etched in glyceria regia. 100×
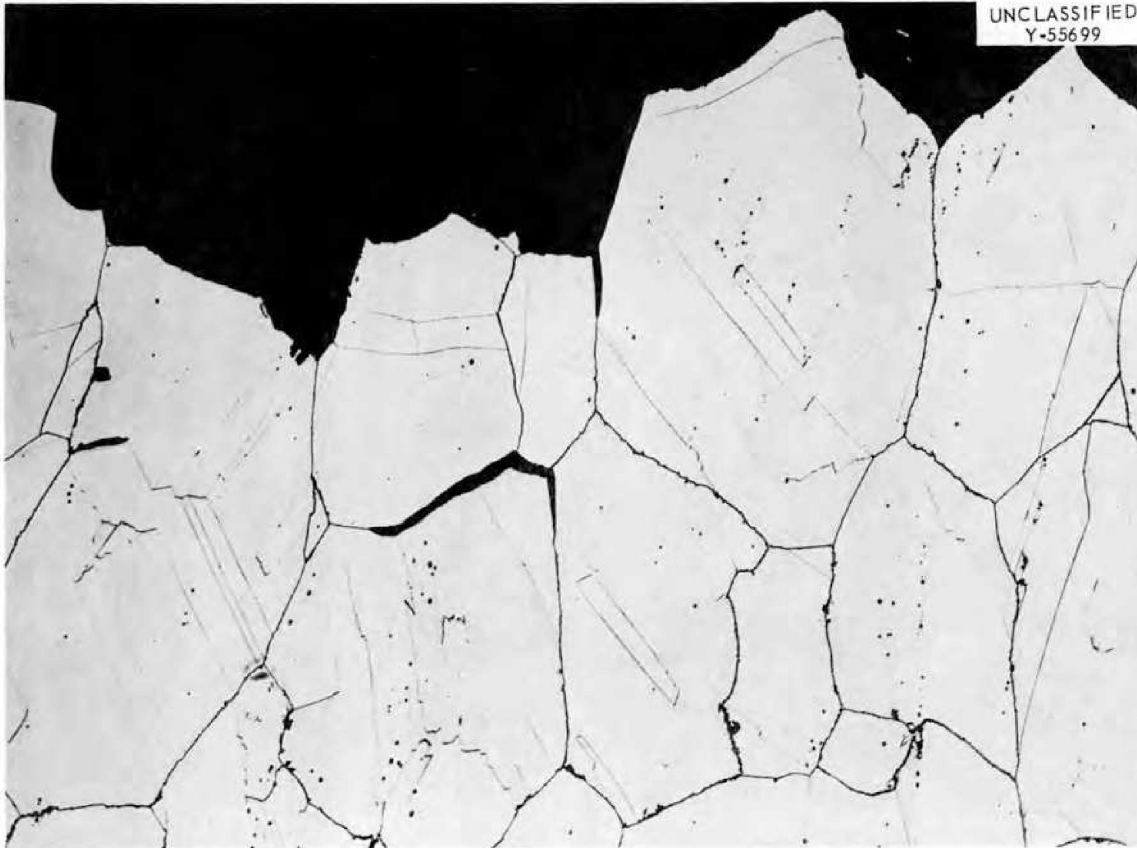
Fig. 30. Fracture of a Hastelloy N Specimen Tested at 1400°F, Annealed 1 hr at 2400°F, Cold Worked 10%, and Aged 100 hr at 1200°F Prior to Testing. Heat 2477. Etched in glyceria regia. 100×

Table 12. Tensile Properties of Hastelloy N After
Various Aging Treatments

Heat 2477

Treatment Prior to Testing	Test Temperature (°F)	Yield Strength (psi)	Ultimate Tensile Strength (psi)	Elongation (%)	Reduction in Area (%)
a	75	38,400	109,000	55.5	62.2
b	1400	42,300	68,600	41.5	39.9
c	75	38,700	104,000	75.0	62.0
c	1400	23,400	60,800	29.5	29.5
100 hr at 1200°F[d]	75	36,300	101,000	69.5	58.2
100 hr at 1200°F[d]	1400		60,600	33.5	38.7
Cold Worked 10% and 100 hr at 1200°F[d]	1400	44,300	70,500	28.0	22.7
Cold Worked 20% and 100 hr at 1200°F[d]	1400	60,500	77,500	33.0	20.6
100 hr at 1500°F[d]	75	50,800	112,000	45.0	45.2
100 hr at 1500°F[d]	1400	31,400	64,700	23.5	30.9

[a]Annealed 1 hr at 2150°F in argon, fast cooled.
[b]As-Received; mill annealed at 2150°F.
[c]Annealed 1 hr at 2300°F in argon, fast cooled.
[d]Annealed 1 hr at 2400°F in argon, fast cooled.

1500°F does not result in strengths as high as observed after aging at 1200°F. The microstructure of a specimen aged at 1500°F and tested at 1400°F is shown in Fig. 31. The most important fact is that the changes in strength produced by aging this alloy do not result in fracture ductilities low enough to be of concern.

Further aging studies were carried out on air-melted heats 5074 and 5075. The results of these studies are summarized in Table 13. All
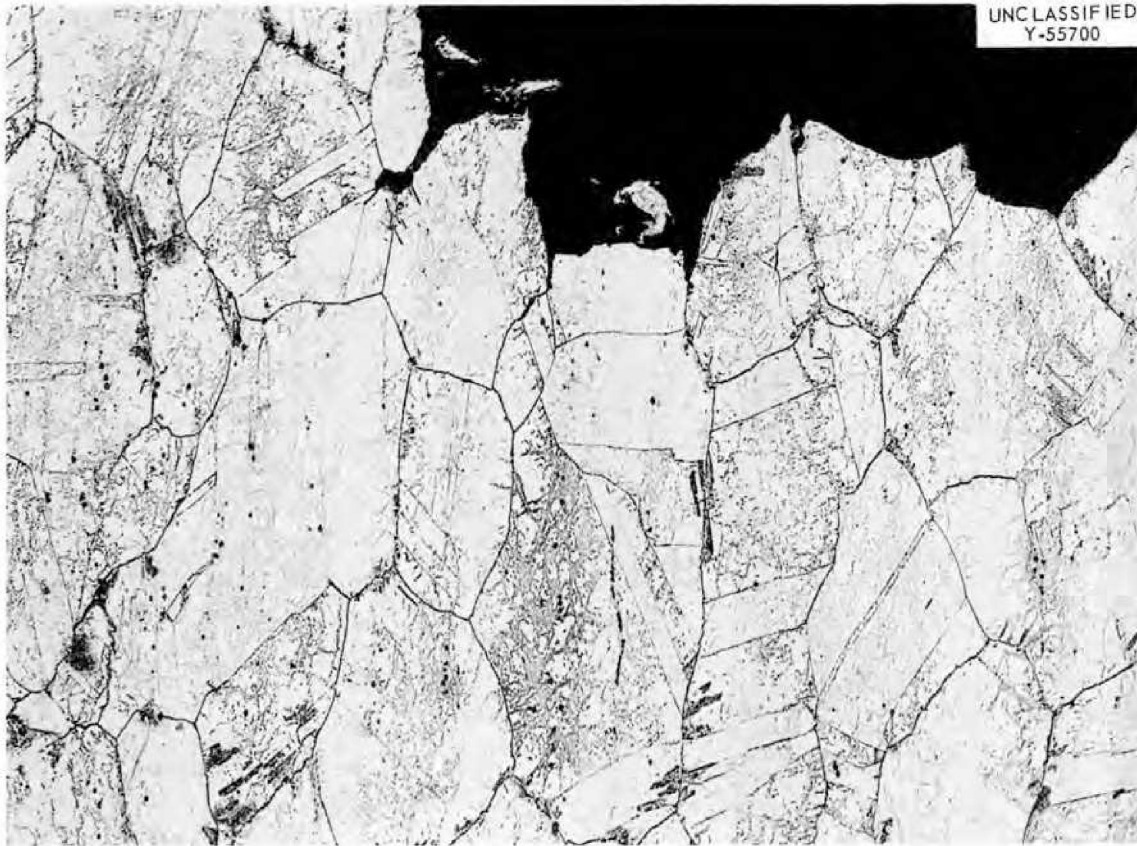
Fig. 31. Fracture of Hastelloy N Tested at 1400°F, Annealed 1 hr at 2400°F, and Aged 100 hr at 1500°F Prior to Testing. Heat 2477. Etched in glyceria regia. 100×

Table 13. Influence of Aging on the Tensile Properties of Hastelloy N
Test Temperature 1600°F

Heat Treatment	Yield Strength (psi)	Ultimate Tensile Strength (psi)	Elongation (%)	Reduction in Area (%)
Heat 5074				
a	25,900	36,000	26.0	20.5
10 hr at 1100°F[a]	26,700	36,600	17.0	19.8
100 hr at 1100°F[a]	26,300	35,700	12.5	13.2
1000 hr at 1100°F[a]	30,600	36,200	14.0	11.7
10 hr at 1200°F[a]	29,700	35,800	14.5	14.6
100 hr at 1200°F[a]	28,300	37,000	13.0	12.4
1000 hr at 1200°F[a]	29,700	35,800	39.5	39.8
b	24,700	36,100	8.5	4.8
1000 hr at 1100°F[b]	26,200	32,900	5.0	4.0
1000 hr at 1200°F[b]	23,800	36,400	47.5	47.0
Heat 5075				
a	29,700	37,300	30.0	32.22
100 hr at 1100°F[a]	27,500	34,900	16.5	13.18
1000 hr at 1100°F[a]	28,200	33,600	12.5	10.25
100 hr at 1400°F[a]	27,600	37,600	22.5	25.63
100 hr at 1600°F[a]	27,700	37,200	19.0	26.23
100 hr at 1800°F[a]	26,600	36,700	25.5	27.30
b	27,300	38,100	7.5	3.97
1 hr at 1600°F[b]	26,500	37,800	11.5	9.44
4 hr at 1600°F[b]	25,800	38,100	23.5	18.43
16 hr at 1600°F[b]	24,400	38,700	33.0	26.05
100 hr at 1600°F[b]	24,100	38,100	29.0	25.71

[a]Annealed 1 hr at 2150°F in argon, fast cooled.
[b]Annealed 1 hr at 2300°F in argon, fast cooled.

specimens were tested at 1600°F after aging. Aging at 1100°F after annealed at 2150°F reduces the ductility by almost a factor of 2 over a period of 1000 hr. The strength shows a slight increase over this same time period. Figure 32 shows the microstructure of a specimen annealed at 2150°F, aged 1000 hr at 1100°F, and tested at 1600°F. The structure looks quite similar to that shown in Fig. 17 for an unaged specimen. Aging at 1200°F produces a decrease in the ductility by about a factor of 2, but the required aging times are much less than at 1100°F. After 1000 hr at 1200°F, the material has apparently overaged and the fracture ductility is quite high. Figure 33 is a photomicrograph of a specimen annealed at 2150°F, aged 1000 hr at 1200°F, and tested at 1600°F. The specimen has recrystallized during the test. If the material is initially annealed at 2300°F and aged at 1100°F for 1000 hr, no apparent change in properties occurs. However, aging at 1200°F for 1000 hr results in a very high fracture ductility.

Heat 5075 gave results very similar to those of heat 5074. After annealing at 2150°F, aging at 1100°F reduced the fracture ductility. Aging for 100 hr at 1400, 1600, and 1800°F did not significantly alter the fracture ductility. After annealing at 2300°F, it was possible to improve the ductility by aging at 1600°F prior to testing at 1600°F. As can be seen from the data in Table 13, the ductility becomes progressively better as the aging time at 1600°F increases. Figure 34 shows the fracture of a specimen annealed at 2300°F, aged for 100 hr at 1600°F, and tested at 1600°F. The structure is quite similar to that of the unaged specimen (Fig. 21). The only significant difference is that the cracks are shorter and more numerous in the aged speckmen than in the unaged one.

DISCUSSION OF RESULTS

The results that have been presented indicate that the tensile properties of Hastelloy N can be altered significantly by various pretest anneals. Annealing at temperatures in excess of 2150°F lowers the fracture ductility at test temperatures in excess of 1200°F. The changes brought about at lower test temperatures do not appear to be of concern. Some of the heats of material exhibit a ductility minimum with temperature
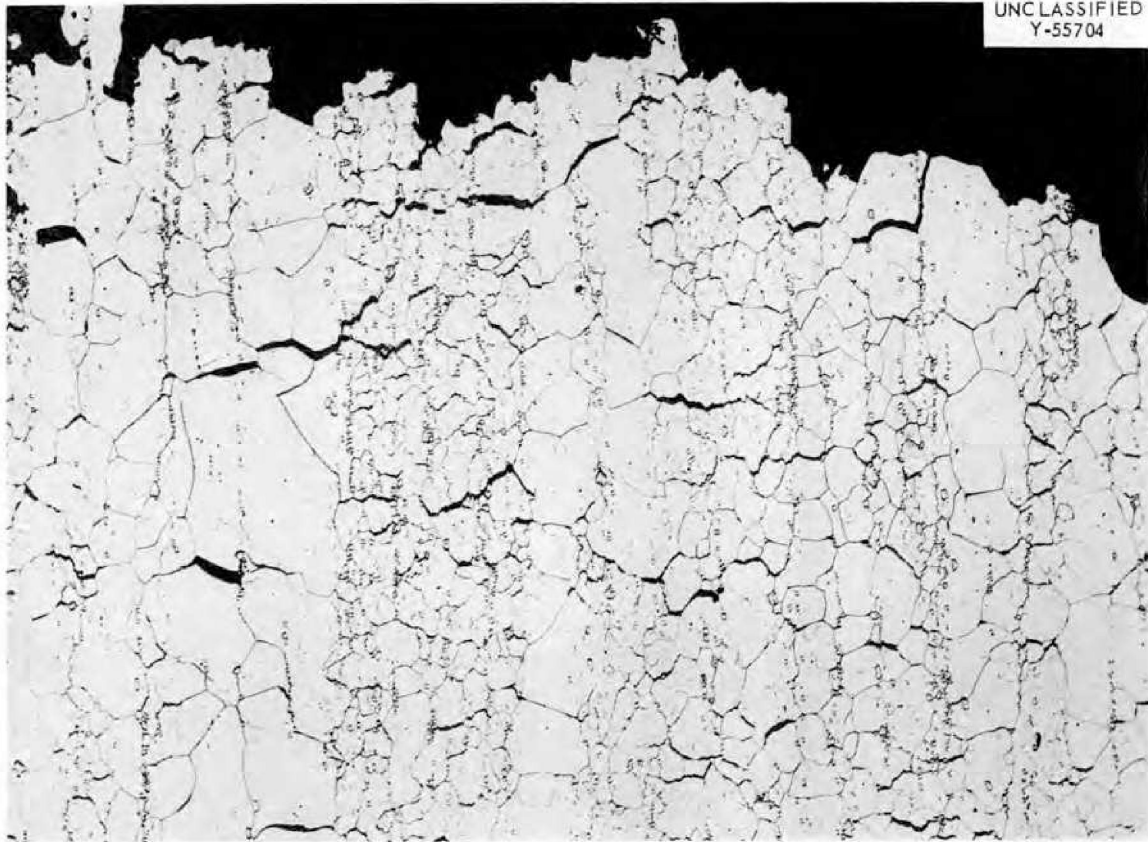
Fig. 32. Fracture of a Specimen Tested at 1600°F, Annealed 1 hr at 2150°F, and Aged 1000 hr at 1100°F Prior to Testing. Heat 5074. Etched in glyceria regia. 100X
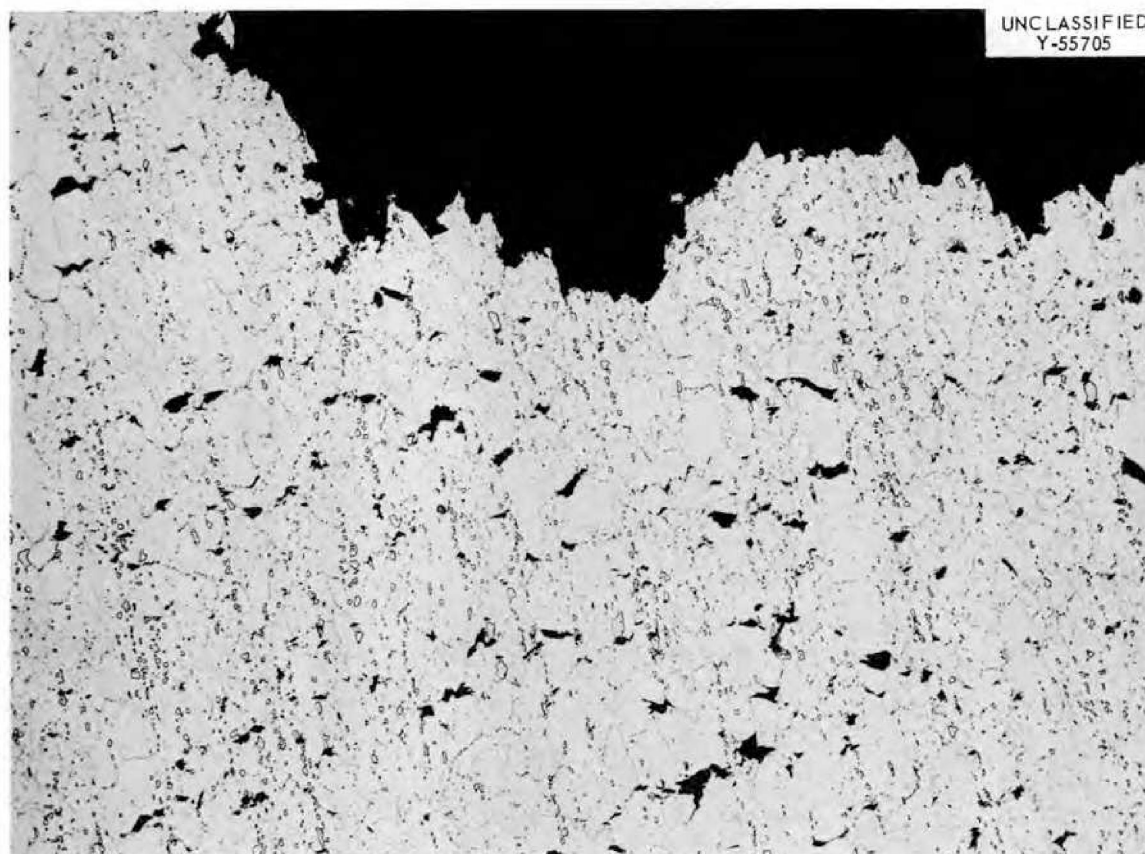
Fig. 33.   Photomicrograph of a Hastelloy N Specimen Tested at 1600°F, Annealed 1 hr at 2300°F, and Aged 1000 hr at 1200°F Prior to Testing. Heat 5074.   Etched in glyceria regia.   100×
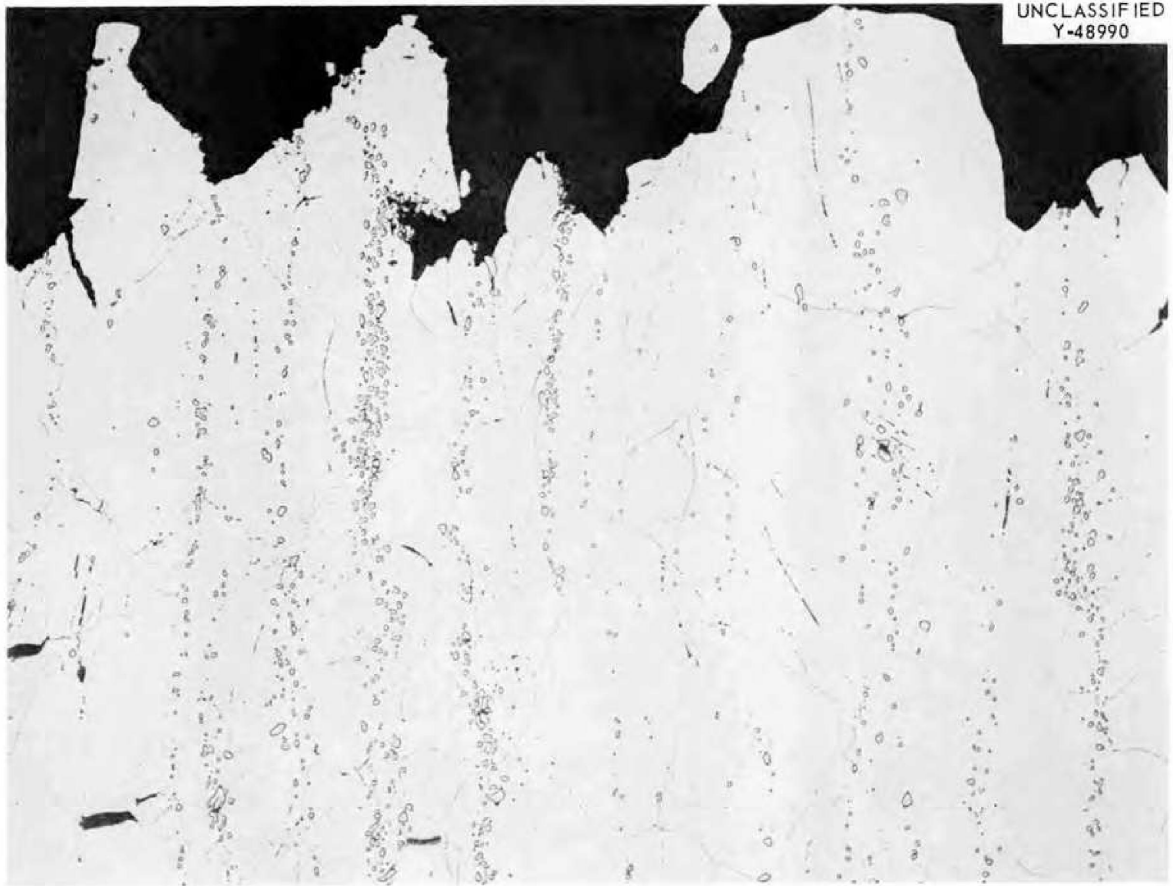
Fig. 34. Photomicrograph of Hastelloy N Specimen Tested at 1600°F, Annealed 1 hr at 2300°F, and Aged 100 hr at 1600°F Prior to Testing. Heat 5075. Etched in glyceria regia. 100×

and then good ductility at higher temperatures, whereas the ductility of other heats continues to decrease with increasing temperature through 1800°F, the maximum temperature investigated. The air-melted heats showed reductions in area at fractures of less than 2% under some conditions. The vacuum-melted material was more ductile with a minimum reduction in area of 26% being obtained.

Metallographic studies revealed that the microstructure of the air-melted material consisted of large precipitate particles dispersed in stringers in a solid solution matrix. These particles were identified as M_6C type carbides. As the pretest annealing temperature was increased above 2150°F, some grain growth occurred, the precipitate particles began to dissolved, and the grain boundaries became broader and etched more readily. At 2500°F the precipitate particles were completely dissolved and a lamellar grain boundary phase appeared. To determine whether the lamellar phase formed directly from the M_6C precipitates, the end of a wire was induction melted and cooled very rapidly. The photomicrograph in Fig. 35 shows the transformation taking place. The lamellar phase was not isolated and identified. Hence, it is not known whether it has a stoichiometric ratio different from M_6C or whether it simply represents another distribution of the same phase. Neither is it known whether the formation of this lamellar grain boundary phase is indicative of grain boundary melting or whether this is a solid-state transformation. The vacuum-melted material appeared quite similar to that of the air-melted material after annealing at 2150°F. However, the carbides appear to dissolve more rapidly in the vacuum-melted material and a single-phase microstructure is obtained by annealing at 2400°F. Metallographic studies have also shown that the recovery of fracture ductility at elevated temperatures is associated with recrystallization in the air-melted material and with polygonization in the vacuum-melted heat.

Further studies have shown that after Hastelloy N has been annealed at 2300°F, which should produce a very low minimum fracture ductility, it is possible to recover the ductility by several techniques. Cooling the material very slowly will assure good ductility upon reheating. Also, aging at a temperature in the 1600 to 1800°F range for a few hours seems

Fig. 35.   Hastelloy N Wire Melted on One End.   Heat 5075.   Etched in glyceria regia.   (a) 100X, (b) 500X.   Reduced 15%.

adequate. Working at a low temperature where the ductility is good before testing at elevated temperatures is also quite effective.

Although Hastelloy N is not an age hardenable alloy in the same sense as Inconel X and other materials which contain titanium and aluminum and form intermetallic compounds with nickel,[3] it does possess some aging tendencies. The ability to recover ductility after annealing at 2300°F by aging at 1600°F has already been pointed out. There is also a tendency to embrittle Hastelloy N after a 2150°F anneal by annealing in the 1100 to 1200°F range. The minimum ductility can be lowered by a factor of 2.

Although it is possible to cause wide variations in the fracture ductility of this material, the changes in microstructure are quite small. If a precipitate is formed during aging, it is too fine to be resolved at 1000X. It would also seem that such a fine precipitate would cause significant changes in the strength as well as the ductility. The most distinct microstructural features of the specimens tested at elevated temperatures are the large intergranular cracks. If the ductility is low, there are only a few very large intergranular cracks present. If the fracture ductility is high, the cracks are more numerous and shorter.

In attempting to explain the behavior of Hastelloy N, one cannot help but observe that numerous other metals and alloys have been known for some time to exhibit ductility minima and that the attempts to explain these results have been only partially successful.[4] Since experimenters have not succeeded in this area with metals such as pure copper, it is indeed a large undertaking to attempt to rationalize the behavior of Hastelloy N, a far from simple alloy. Before attempting this, a brief review of the general thoughts in the area of fracture ductility are in order.

Ductility minima have often been accounted for in terms of strain aging. Glen,[5] for example, has shown that various strain aging effects occur during tensile testing of steels, each associated with the presence of a particular alloying element. This minimum in ductility is accompanied by a maximum in yield strength, and also, serrated stress-strain curves are obtained. Hastelloy N exhibits serrated stress-strain curves over the temperature range of 800 to 1600°F and often small maxima in yield strength occur in the same temperature range. However, these serrations do not seem to be influenced by the variables which alter the rupture ductility. Hence

although strain aging due to different impurities occurs over certain temperature ranges, it is felt that strain aging is not responsible for the low ductility of Hastelloy N.

Rhines and Wray[4] have attributed brittleness in nickel to the occurrence of grain boundary shearing and associated intergranular rupture. According to this theory, cavities or voids, nucleating at four-grain intersections, grow intergranularly as long as they lie along shearing grain boundaries, thus decreasing the effective cross section of the metal and lowering its fracture ductility. The effectiveness of this process is opposed by the ability of the grain boundaries to move either by grain boundary migration or by recrystallization. Ductility minima occur under combinations of temperature and strain rate which result in a critical relation between the rates of void growth and the rate of grain boundary migration. Smith[6] also looked at the ductility minimum in nickel. Although he concurred with Rhines on several points, he felt that the intergranular voids were nucleated at grain boundary voids by a process proposed by Gifkins[7] which involved the formation of grain boundary jogs due to slip steps. Smith proposed that the opposing process was the ability of the grain boundaries to migrate and to straighten out these jogs before grain boundary shearing stresses sufficient to nucleate a void were developed. However, the voids noted by both Rhines and Smith were quite large and were either round or elliptical in section. Such features were not observed in the Hastelloy N specimens tested at elevated temperatures in the present study; instead, the cracks were wedge-shaped and extended the entire width of the grain boundary segment normal to the applied stress. However, recrystallization and polygonization were effective in recovering good ductility.

Grain boundary segregation of impurities has also been proposed to account for poor ductility. Bieber and Decker[8] have investigated the effects of a number of minor constituents on the ductility of nickel. Additions of several elements in concentrations up to 0.5 wt % were made and their influence on the rupture ductility evaluated. Noticeable loss of ductility between 1000 and 1500°F was reported for additions of as little as 0.0005 wt % of some of the impurities. The embrittlement was presumed to result from the fact that these elements, because of their incongruous

sizes and valences, tend to preferentially locate at grain boundaries, either in solution or as precipitates or films. However, the authors made no suggestion as to why the ductility was recovered at higher temperatures. Olsen et al.[9] observed a minimum in the ductility vs heat-treating temperature curve for cold-worked and recrystallized nickel containing 0.0009 wt % sulfur. The suggested explanation was that nickel sulfide formed early in the recrystallization process, and as growth proceeds, the sulfide is swept up and concentrated in the moving boundaries. At still higher temperatures, the sulfide would dissolve and diffuse into the matrix.

A slightly different impurity mechanism has been proposed to account for the ductility of Nimonic 80A.[10] During aging, a chromium-rich carbide is formed in the grain boundary. This depletes the adjacent area in chromium which increases the solubility for titanium and aluminum in these areas and results in $Ni_3(Al,Ti)$ not being precipitated in a band adjacent to the grain boundary. This "soft" area is able to absorb large amounts of strain, and very high ductilities are obtained. Although Hastelloy N does not contain significant amounts of titanium or aluminum, such a mechanism may be important with respect to carbide precipitation.

Although none of the mechanisms of embrittlement adequately describe the behavior of Hastelloy N, it is felt that the embrittlement of this alloy by impurity segregation is the general mechanism. The crack geometry (wedge-shaped rather than circular), the width of grain boundaries, the ease of etching of brittle specimens, and several other metallographic observations point toward the formation of a brittle grain boundary layer.

The influence of the several variables investigated can be rationalized as follows. Heating to progressively higher temperatures results in the migration of impurities to the grain boundaries. When the material is tested at elevated temperatures where the grain boundaries must sustain high shear stresses and crack propagation is quite rapid, the material fractures at very low strains. Apparently, temperatures above 2150°F result in the formation of this brittle grain boundary layer. This layer becomes more pronounced as the annealing temperature is increased. If the material is cooled very slowly or aged at a lower temperature, the grain boundary layer agglomerates and a propagating crack cannot advance as

quickly. Cold working the material prior to testing would produce grain boundary steps or jogs that would also interfere with the propagation of a crack. Aging studies on material annealed at 2150°F indicate that this grain boundary layer can also be developed by aging in the 1100 to 1200°F range.

Since the ductility of the vacuum-melted material is much superior to the arc-melted material and since there are variations among the arc-melted heats, it would appear that impurity elements are very important. The fact that reducing the carbon content improves the ductility and the metallographic observation that an intergranular carbide phase is formed at elevated temperatures suggest that carbon is an important impurity. The chemical analysis in Table 1 does not show any marked differences in carbon content between the heats. However, it may be that the presence of another impurity controls the behavior of the carbon. Significant differences in the analyses of the air- and vacuum-melted heats exist for sulfur, manganese, silicon, titantium, aluminum, and tungsten. The influence of these impurities on the fracture ductility should be investigated.

In considering the significance of results of this type, it is well to consider their practical importance. Several factors seem important:

1. When this material is being used in the mill-annealed (2150°F) state, prolonged use in the 1100 to 1200°F range will reduce the ability of the material to deform.

2. During welding, the material will be subjected to temperatures in the 2200 to 2800°F range. This will produce areas which are quite brittle. Good ductility can be recovered by slow cooling, reannealing at about 1600°F, or by cold working. The method used would be determined from practical considerations.

3. Creep tests are needed to define the ductility as a function of strain rate. However, in many structures, loading rates of the order of those used in this study are encountered.

SUMMARY AND CONCLUSIONS

It has been shown that the tensile properties of Hastelloy N can be altered significantly by heat treatment. Although small variations in strength are observed, it is felt that the influence on ductility is the most important. A vacuum-melted alloy was found to exhibit good ductility under all conditions studied. Four heats of air-melted material were found to exhibit quite low fracture strains in the 1400 to 1800°F range after annealing at temperatures in excess of 2200°F. Aging at 1600°F, very slow cooling rates, and cold working were effective in recovering the ductility. The recovery of good ductility at 1800°F in some heats was associated with recrystallization. After annealing at 2150°F, it was found that the high-temperature ductility was reduced by aging in the 1100 to 1200°F range.

It is felt that these effects can be explained in terms of the formation of a brittle grain boundary layer along which a crack can propagate easily at elevated temperatures. Breaking up the continuity of this layer by aging or cold working recovers good fracture ductility. The formation of this layer is associated with the presence of trace alloying elements.

ACKNOWLEDGMENTS

REFERENCES

1. T. K. Roche, <u>The Influence of Composition Upon the 1500°F Creep-Rupture Strength and Microstructure of Molybdenum-Chromium-Iron-Nickel-Base Alloys</u>, ORNL-2524 (June 24, 1958).

2. R. W. Swindeman, <u>The Mechanical Properties of INOR-8</u>, ORNL-2780 (Jan. 10, 1961).

3. Murray Kaufman, Trans. Met. Soc. AIME <u>227</u>, 405 (1963).

4. F. N. Rhines and P. J. Wray, Trans. Am. Soc. Metals <u>54</u>, 117 (1961).

5. J. Glen, J. Iron Steel Inst. (London) <u>190</u>, 30 (1958).

6. T. C. Smith, <u>Investigation of the Elevated Temperature Ductility Minima in Nickel</u>, Thesis, University of Tennessee (June 1963).

7. R. S. Gifkens, Acta Met. <u>4</u>, 98 (1956).

8. C. G. Bieber and R. F. Decker, Trans Met. Soc. AIME <u>221</u>, 629 (1961).

9. K. M. Olsen, C. F. Larkin, and P. H. Schmitt, Trans Am. Soc. Metals <u>53</u>, 349 (1961).

10. M. J. Fleetwood, J. Inst. Metals <u>90</u>, 429 (1961).

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