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AGING CHARACTERISTICS OF HASTELLOY B

R. E. Clausing
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W. D. Manly

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METALLURGY DIVISION

AGING CHARACTERISTICS OF HASTELLOY B

R. E. Clausing, P. Patriarca, and W. D. Manly

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AGING CHARACTERISTICS OF HASTELLOY B

R. E. Clausing P. Patriarca W. D. Manly

INTRODUCTION

A number of high-strength nickel-base alloys containing molybdenum have been used extensively by the petroleum, petrochemical, and chemical industries as materials for heat exchangers, towers, vats, vessels, tanks, etc. Although developed primarily for resistance to hydrochloric acid, they have been found to exhibit excellent corrosion resistance to other reducing chemicals and acid chlorides as well as to sulfuric, phosphoric, and acetic acids.[1-3]

Recently, the ASME Pressure Vessel Code recognized the alloys Hastelloy B (nominal composition, 28% molybdenum, 5% iron, balance nickel) and Hastelloy C (nominal composition, 17% molybdenum, 15% chromium, 5% iron, 5% tungsten, balance nickel) for service up to 650 and 1000°F, respectively.[4] The behavior in creep and the exceptional corrosion resistance to certain nonaqueous environments at elevated temperatures have made Hastelloy B attractive for application in nuclear reactors. Since Hastelloy B is age hardenable, extended service results in an increase in the yield strength and hardness, a low creep rate, and high stress rupture properties.[5] Figure 1 describes the stress rupture behavior of Hastelloy B along with that of Inconel and type 316 stainless steel at 1500°F.

Data obtained in the 1200 to 1400°F temperature range have shown Hastelloy B to have an unfavorably low ductility. The incidence of cracking of the base metal in the heat-affected zone when welded under conditions of severe restraint has been attributed to this characteristic.[1,3]

It was recognized that a further determination of the weldability of this alloy and of the properties of welded joints at the elevated temperatures of interest was required. However, it was deemed advisable to undertake a preliminary study to obtain more extensive data pertaining to the type and occurrence of aging and its effect on the mechanical properties of the wrought material for comparative purposes.

The aging studies were made by measuring the increase in hardness of Hastelloy B as a function of time and temperature and by observing metallographically any structural changes which occurred. As a measure of the mechanical properties, tensile tests were made at both room temperature and elevated temperatures on the material after various aging treatments.

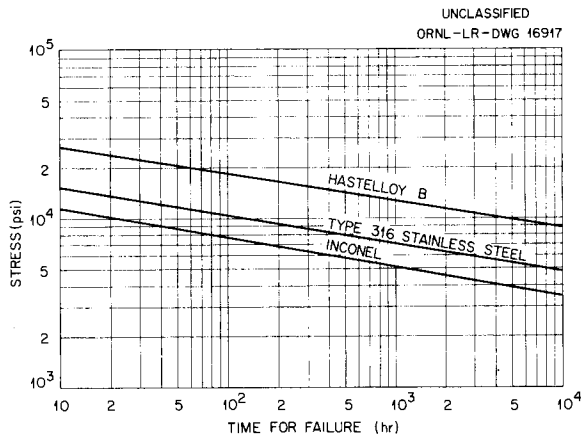

Fig. 1. Creep of Several Metals at 1500°F.

MATERIAL

Hastelloy B sheet, 0.065 in. thick, was used throughout the investigation. Its chemical composition, as provided by the manufacturer, was 27.0% molybdenum, 5.5% iron, 0.6% chromium, 0.6% silicon, and 0.035% carbon.

EQUIPMENT

Tensile properties of this material were determined by using a sheet metal specimen as described in Fig. 2. A hydraulically operated tensile machine equipped with a stress-strain recorder and strain pacer was used for both room-temperature and elevated-temperature tests. The strain rate was 0.05 in./min for all data reported.

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[1] C. G. Chisholm, "Welding and Other Fabrication Methods for Hastelloy Alloys," *Welding J.* 25, 1179–1183 (1946).

[2] Haynes Stellite Co., *Hastelloy High-Strength, Nickel-Base, Corrosion-Resistant Alloys*, p 6–13, Sept. 1, 1951.

[3] R. P. Culbertson, "Weldability of Wrought High-Alloy Materials," *Welding J.* 34, 220–230 (1955).

[4] R. M. Wilson, Jr., and W. F. Burchfield, *Nickel and High-Nickel Alloys for Pressure Vessels*, Welding Research Council Bulletin Series No. 24 (Jan. 1956).

[5] Haynes Stellite Co., *Haynes Alloys for High-Temperature Service*, p 51–53, 1950.

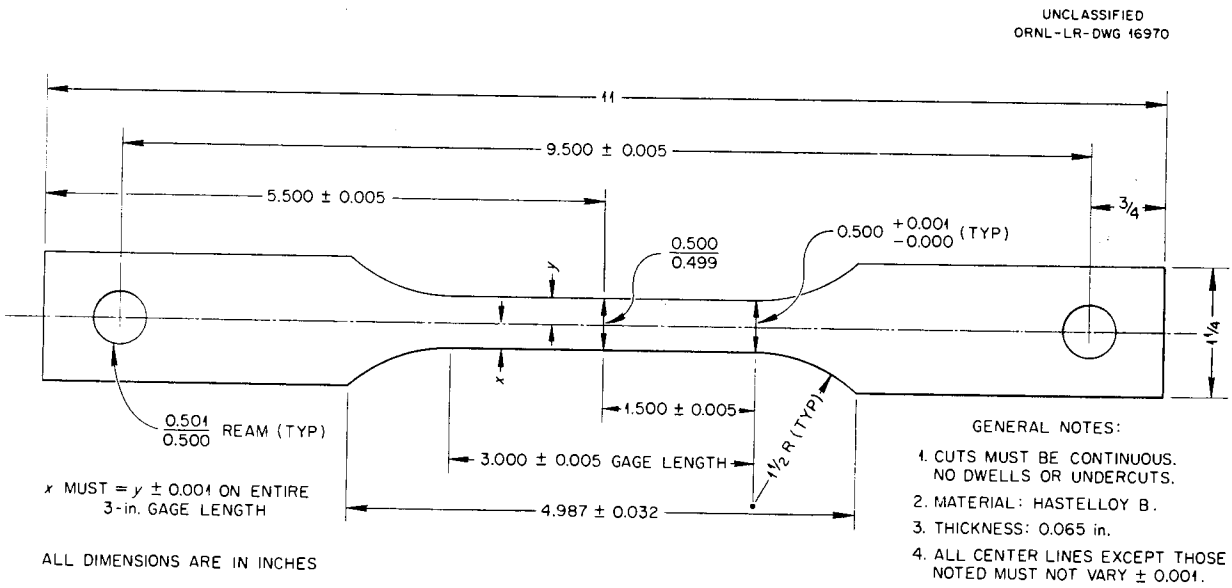


Fig. 2. Elevated-Temperature Tensile Specimen.

Aging of specimens at elevated temperatures required inert or reducing furnace atmospheres. All specimens were solution-annealed at 2100 to 2150°F in a tube furnace provided with a −80°F dew-point hydrogen atmosphere. Many of the long-time aging treatments were conducted in this type of furnace provided with helium or argon as a protective atmosphere, the gases having been dried with activated alumina. The majority of the hardness and metallographic specimens, however, were encapsulated in quartz containing a vacuum or purified inert atmosphere. These encapsulated specimens were subsequently aged in box-type wire-wound electric furnaces. Specimens prepared in this manner remained bright during heat treatment.

Metallographic specimens were prepared in the usual manner and etched with chrome regia (1 part 1% chromic acid solution, 10 parts water, and 3 parts hydrochloric acid) at room temperature for times varying from 3 to 5 sec. A research metallograph was used for observation and for photomicrography at magnifications up to 2000 dia. Hardness measurements were made with a Vickers diamond pyramid indenter and a 10-kg load.

EXPERIMENTAL PROCEDURE AND RESULTS

Since the behavior of Hastelloy B after prolonged exposure to elevated temperatures appeared to be the result of the precipitation of a phase or phases in the solid solution, the influence of several variables on the type, rate, and quantity of precipitate was investigated. The effect of aging temperature and time was determined, and the room-temperature and elevated-temperature mechanical properties were associated with the observed microstructures.

The binary nickel-molybdenum equilibrium diagram[6] shown in Fig. 3 was used as a guide in this study, with recognition given to the possibility

[6] F. H. Ellinger, "The Nickel-Molybdenum System," *Trans. Am. Soc. Metals* **30**, 607 (1942).

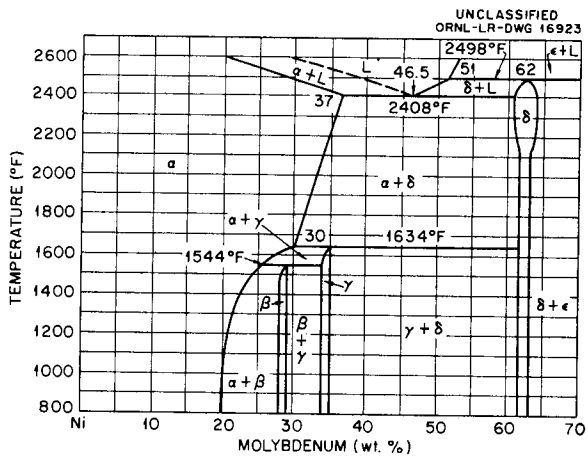


Fig. 3. Phase Diagram for Molybdenum-Nickel.

that the presence of iron and other elements could have a significant influence on the positions of the phase boundaries.

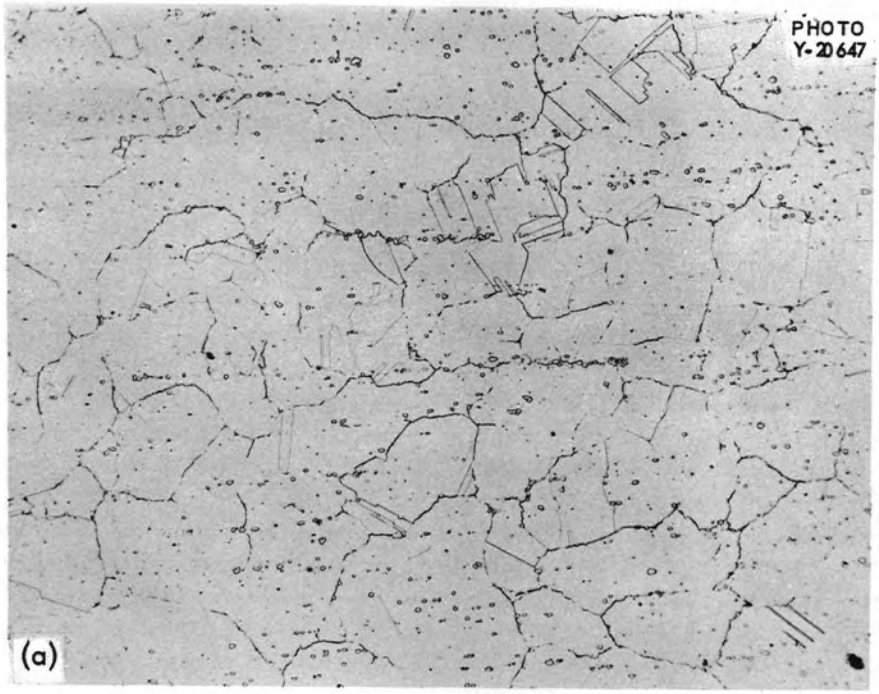
All specimens were solution-annealed for 2 hr at 2100°F prior to aging. A typical solution-annealed structure is shown in Fig. 4, photomicrographs of the alpha solid solution at 150 and 2000 magnifications. The presence of a second phase, possibly retained delta and/or complex carbides, is evident.

A number of tensile specimens which had been aged at 1100, 1300, 1500, and 1600°F for 100, 500, and 1000 hr were tested at room temperature to determine the change in properties resulting from elevated-temperature exposure. The results are summarized in Table 1. It can be seen that a relatively minor improvement in the room-temperature tensile strength resulted after aging but that an appreciable loss in ductility occurred, accompanied by a significant increase in hardness. The most prominent change in room-temperature tensile strength, ductility, and hardness is associated with prolonged aging at 1300°F. The tensile data of Table 1 are treated graphically in Fig. 5. The pronounced loss in room-temperature ductility after aging at 1300°F is evident.

Although the room-temperature behavior of this material is considered important, of greater interest in this study was the effect of aging on the elevated-temperature behavior. A number of tensile specimens were aged for varying periods at 1100, 1300, 1500, and 1600°F and tested at the temperature of aging. Examination of the results, summarized in Table 2, will reveal a number of trends, the most prominent again being the loss in ductility after aging for 1000 hr at 1300°F. These data are compared graphically with those for the solution-annealed material in Fig. 6. It can be seen that the tensile strength of the specimens that were not aged decreased in a linear manner with testing temperature. It is interesting to note that there is a minimum in the ductility curve occurring between 1300 and 1500°F. The lower ductility of the tensile specimens tested at 1300°F would indicate that the precipitation reaction is greatly accelerated by a tensile stress. Stress-induced precipitation is very likely if the mechanism of formation is suitable. Below 1300°F the diffusion rates are so low that even under a tensile stress the reaction is quite slow. The severe reduction of the elongation at 1100°F after 1000 hr, however, indicates that precipitation is occurring.

Table 1. Effect of Aging Time and Temperature on the Room-Temperature Tensile Strength and Ductility of Hastelloy B

Aging Temperature (°F)	Treatment	Tensile Strength (psi)	Elongation (% in 3 in.)	Hardness (VPN)
Room	Solution-annealed for 2 hr at 2100–2150°F and air-cooled	118,600	32.0	205–207
1100	Aged for 100 hr	117,700	25.0	243–249
	Aged for 500 hr	126,000	14.0	285–287
	Aged for 1000 hr	135,700	18.5	285–289
1300	Aged for 100 hr	116,300	25.0	254
	Aged for 500 hr	140,600	12.5	339–351
	Aged for 1000 hr	135,100	6.2	409–425
1500	Aged for 100 hr	121,700	28.7	238
	Aged for 500 hr	109,800	12.5	272–274
	Aged for 1000 hr	126,600	16.0	283–285
1600	Aged for 100 hr	124,600	23.7	245–247
	Aged for 500 hr	132,000	20.0	281–285
	Aged for 1000 hr	130,900	23.7	279–281



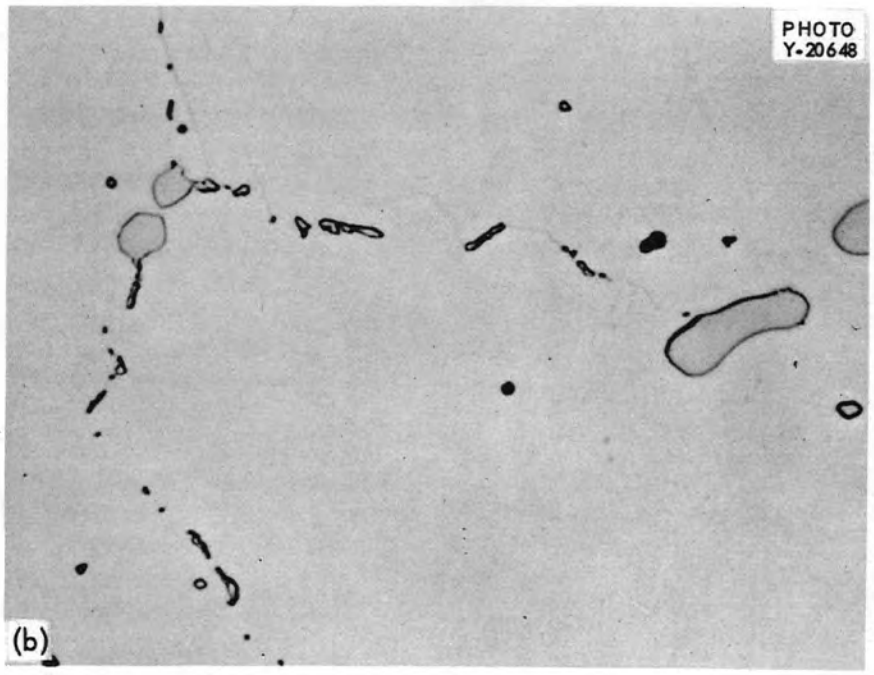


Fig. 4. Microstructure of Hastelloy B Solution-Annealed for 2 hr at 2100°F and Air-Cooled. Etched with chrome regia. (*a*) 150X; (*b*) 2000X.

The influence of the variables of aging temperture and time upon the microstructures of aged Hastelloy B specimens is shown collectively in Figs. 7, 8, and 9, photomicrographs at magnifications of 150 and 2000 of specimens aged at various elevated temperatures for 100, 500, and 1000 hr. It is interesting to note that the microstructure of Hastelloy B after aging at 1300°F is characterized by a Widmanstätten-type precipitate. As may be seen in Fig. 8, this precipitate is still in evidence at 1400°F but ceases to exist at 1500°F. Examination of the binary diagram of Fig. 3 would imply that this precipitate is the beta phase. The diagram also suggests that the needle-like precipitate coexisting in the alpha phase with the carbides and/or delta particles at 1500, 1600, and 1650°F is the gamma phase. It would appear that the alpha-plus-gamma to beta peritectic temperature lies between 1400 and 1500°F for the Hastelloy B used in this investigation.

Although there is an apparent lack of precipitate in the photomicrographs of Hastelloy B aged for

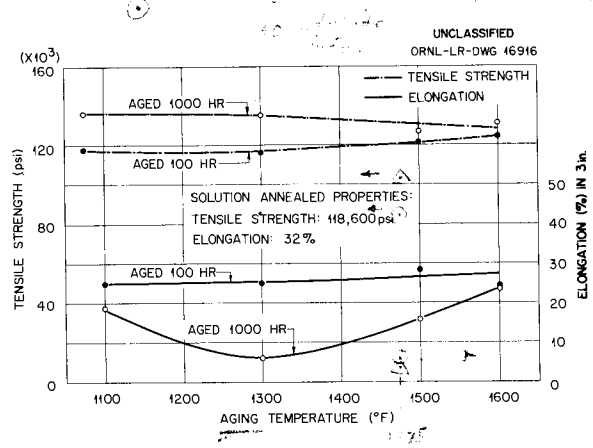


Fig. 5. Effect of Aging Time and Temperature on the Room-Temperature Tensile Strength and Ductility of Hastelloy B.

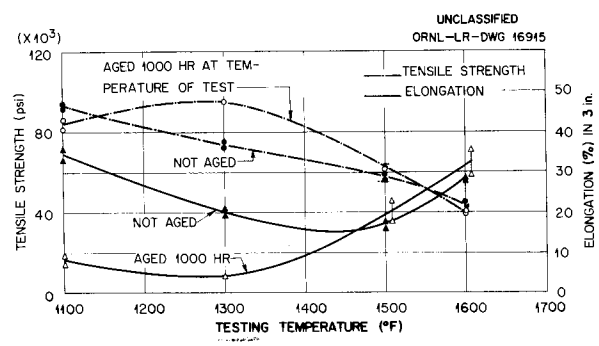


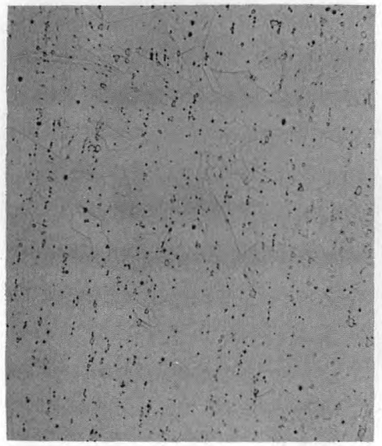
Fig. 6. Effect of Aging Time and Temperature on the Elevated-Temperature Tensile Strength and Ductility of Hastelloy B.

Table 2. Effect of Aging Time and Temperature on the Elevated-Temperature Tensile Strength and Ductility of Hastelloy B

Aging and Testing Temperature (°F)	Solution-Annealed*		Aged for 100 hr		Aged for 500 hr		Aged for 1000 hr	
	Tensile Strength (psi)	Elongation (% in 3 in.)	Tensile Strength (psi)	Elongation (% in 3 in.)	Tensile Strength (psi)	Elongation (% in 3 in.)	Tensile Strength (psi)	Elongation (% in 3 in.)
Room	118,600	32.0						
1100	93,700	36.3	83,100	22.0	76,100	11.3	85,700	9.0
	91,600	33.0	84,100	22.0	91,100	14.0	80,100	7.5
1300	74,000	20.0	60,700	6.0	92,600	6.0	95,900	4.0
	72,500	21.3	66,700	13.0	90,400	6.0		
1500	57,260	18.8	59,600	25.0	63,200	42.5	61,600	17.5
	59,300	16.3	59,700	18.8	63,100	37.5	62,400	26.7
1600	44,400	28.8	44,100	32.0	43,900	45.9	42,600	28.5
	43,800	28.8	45,100	42.5	45,300	49.0	42,600	36.0

*For 2 hr at 2100–2150°F and air-cooled.

1100°F

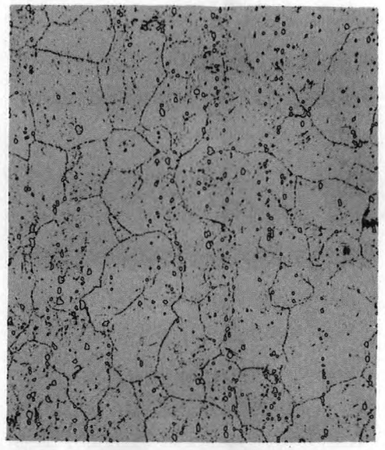


Y-16471 150X
VPN 243/249

1300°F

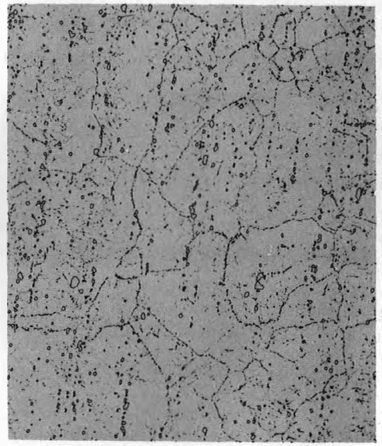
Y-16507 150X
VPN 254

1500°F



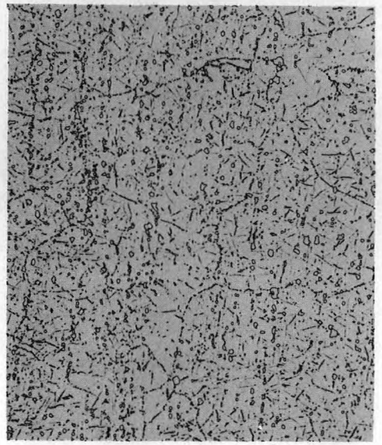
Y-16411 150X
VPN 238

1600°F

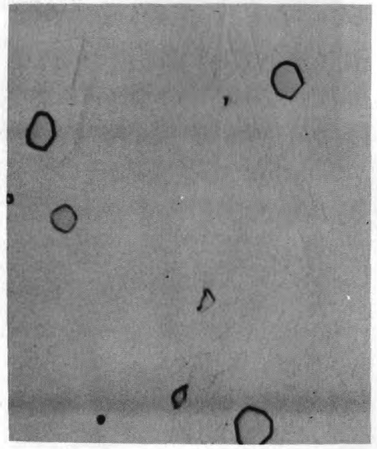


Y-16410 150X
VPN 245/247

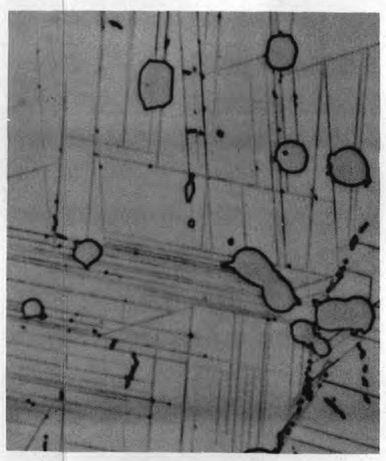
1650°F



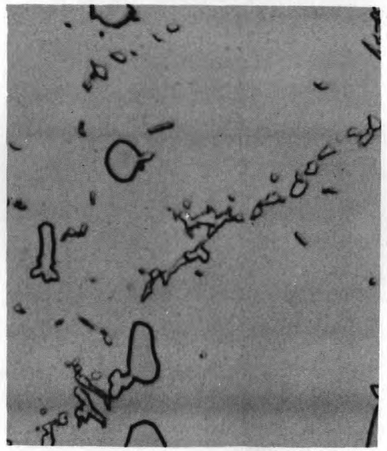
Y-16409 150X
VPN 268/272



Y-16515 2000X



Y-18764 2000X



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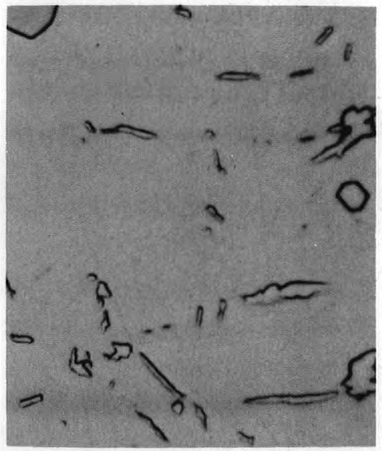

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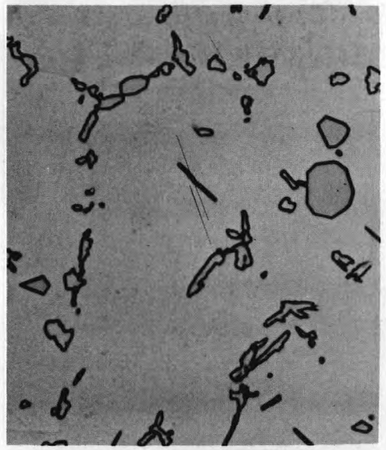
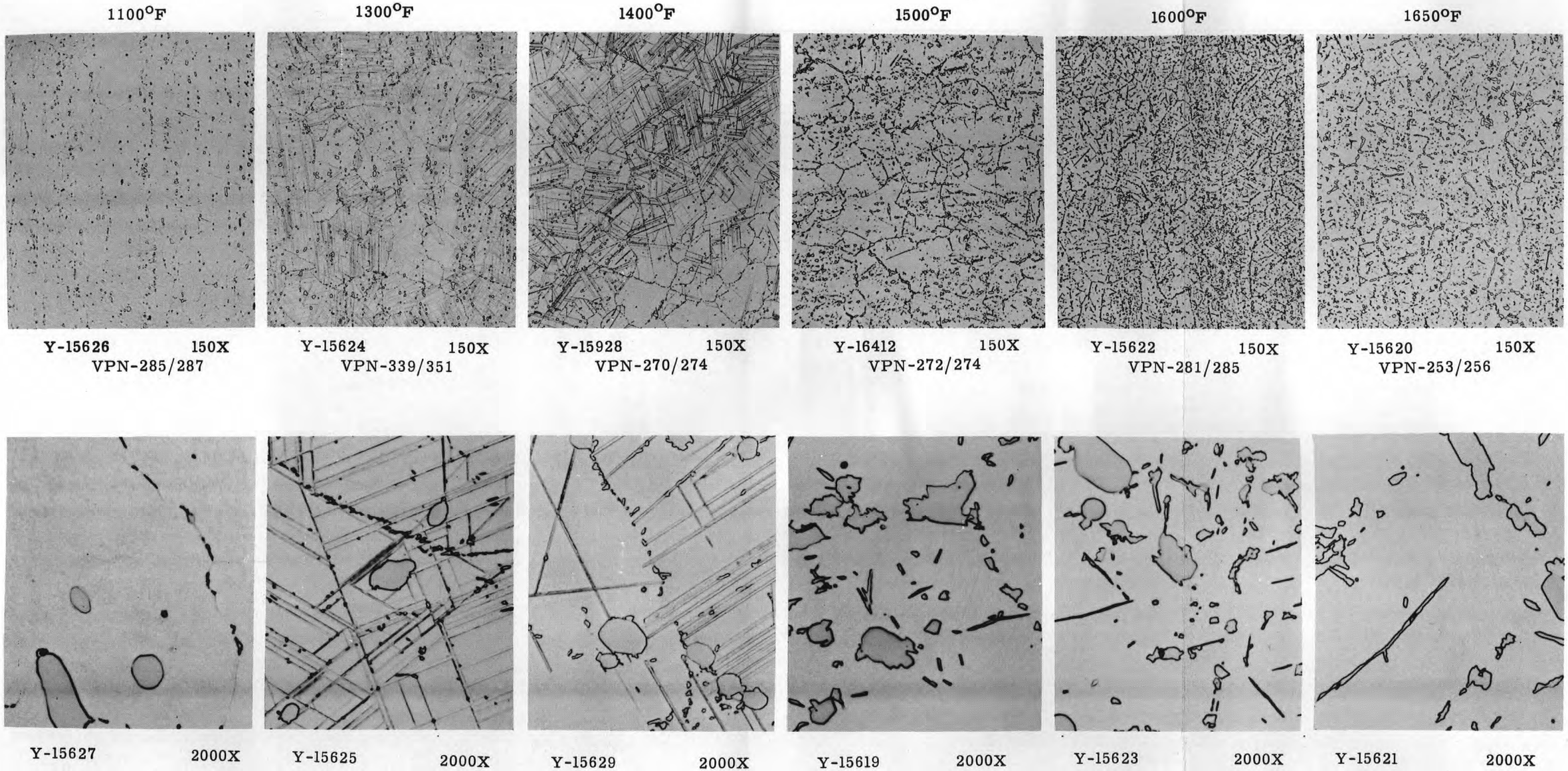


Y-16549 2000X

ETCHANT: CHROME REGIA

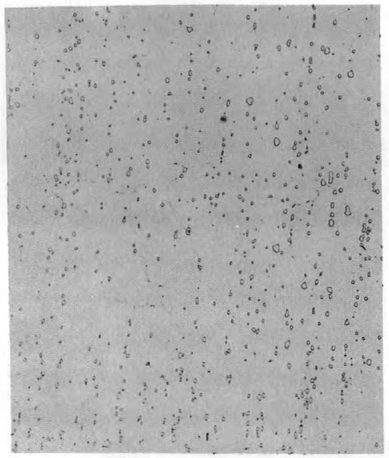
Fig. 7. Microstructure of Hastelloy B Aged from the Annealed Condition at Temperatures Shown for 100 hr.



ETCHANT: Chrome Regia

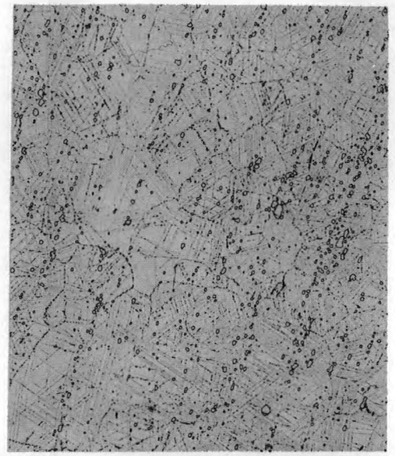
Fig. 8. Microstructure of Hastelloy B Aged from the Annealed Condition at Temperatures Shown for 500 hr.

1100°F



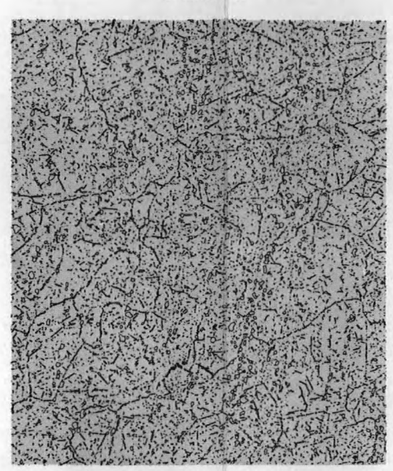
Y-16464 150X
VPN 285/289

1300°F



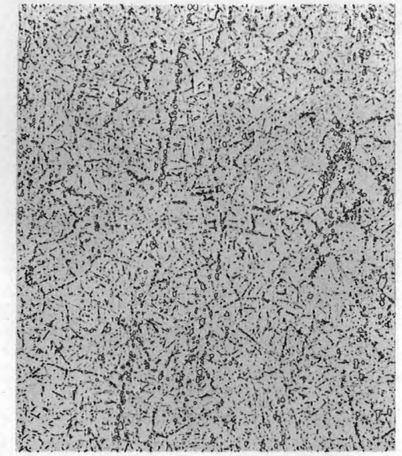
Y-16408 150X
VPN 409/425

1500°F



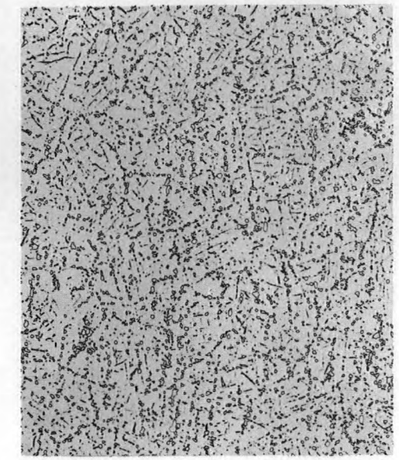
Y-16407 150X
VPN 283/285

1600°F



Y-16406 150X
VPN 279/281

1650°F



Y-16405 150X
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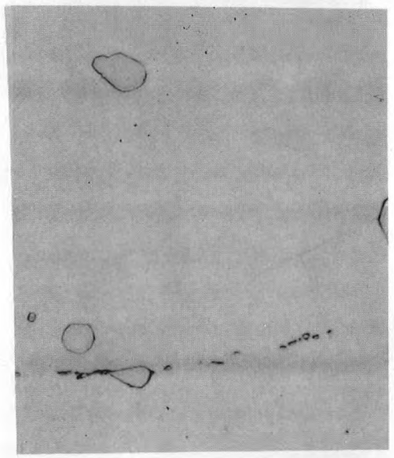

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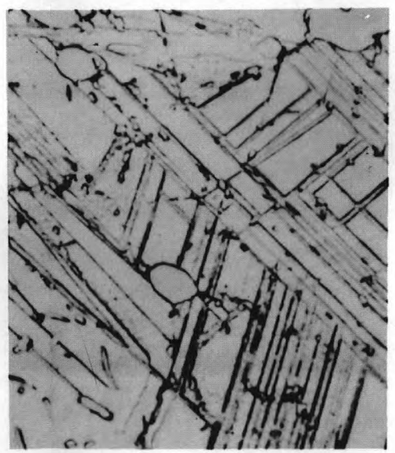
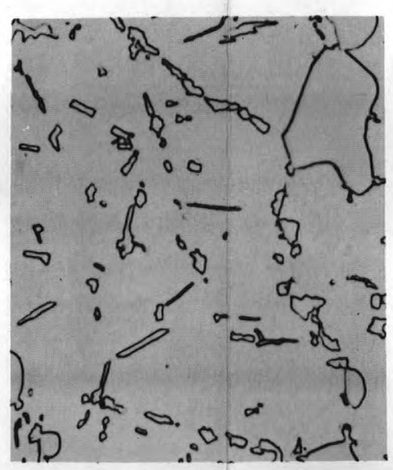
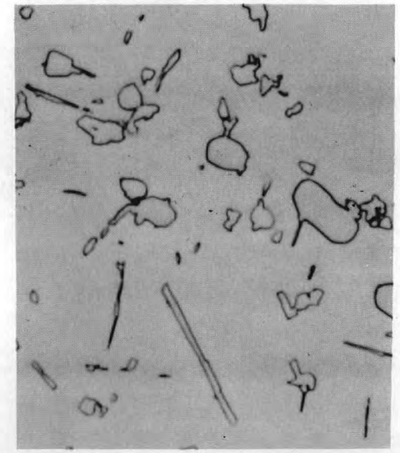


Y-18721 2000X



Y-18720 2000X



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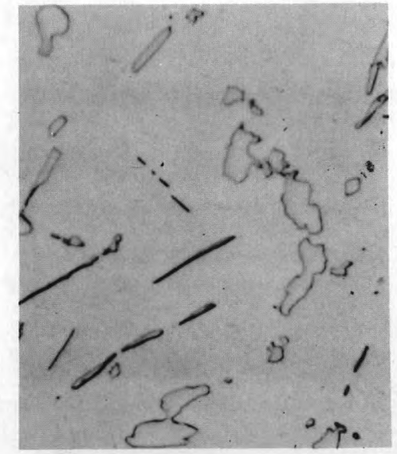

Y-18718 2000X

ETCHANT: Chrome Regia

Fig. 9. Microstructure of Hastelloy B Aged from the Annealed Condition at the Temperatures Shown for 1000 hr.

extended periods at 1100°F, the hardness changes observed suggest that a beta precipitation is occurring. The hardness data, shown graphically in Fig. 10, were compiled from tests of the specimens presented in Figs. 7, 8, and 9. The increase in hardness that occurred with an increase in aging time was significantly greater at 1300°F than it was at the other test temperatures. This hardness increase may be attributed to the appearance of the Widmanstätten-type precipitate, while that observed at 1500°F and above may be associated with the precipitation of the gamma phase. The hardness increase and loss in ductility after aging at 1100°F, however, could not be explained in terms of a readily resolvable precipitate. Optical microscopy at 2000X and electron microscopy at 12,000X failed to reveal a significant change in the microstructures. A limited and unsuccessful attempt was made to detect a precipitate by x-ray diffraction methods.

As indicated previously, the various constituents observed in the course of this investigation were tentatively identified by reference to the nickel-molybdenum binary diagram shown in Fig. 3. Although a study[7] of the special etching techniques associated with the metallography of Hastelloy B confirms the observations made, more positive identification is desirable. Electron diffraction studies may provide this identification.

[7] J. R. Riddle and R. R. Gray, *Metallography of Isothermally Heat Treated Hastelloy B*, to be issued as an ORNL report; presented at the 11th Conference of the AEC Metallographic Group, Nov. 7–8, 1956, Bridgeport, Conn.

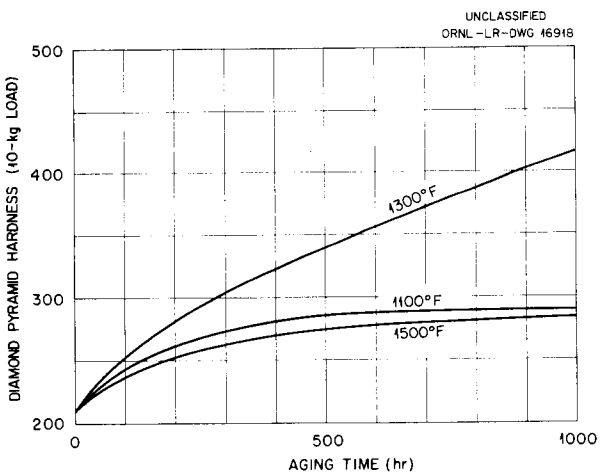

Fig. 10. Effect of Aging Time and Temperature on the Room-Temperature Hardness of Hastelloy B.

CONCLUSIONS

Within the limits of this investigation, in which microstructural and mechanical property studies of Hastelloy B were made, the following conclusions can be drawn.

Elevated-temperature precipitation occurs in this alloy, the nature of which is a function of the temperature and the time at temperature:

1. At 1300°F relatively rapid precipitation of a Widmanstätten-type structure occurs which has been tentatively identified as the beta phase of the nickel-molybdenum binary diagram.
2. After aging at 1100°F for periods to 1000 hr, the beta precipitate cannot be resolved by conventional or electron microscopy, although changes in hardness and other mechanical properties indicate its presence.
3. Between 1500 and 1650°F the precipitate is significantly different from that observed at lower temperatures, being needle-like rather than Widmanstätten. Based on the nickel-molybdenum binary diagram, the structure has been tentatively identified as the gamma phase.

Aging results in a relatively minor improvement in the room- and elevated-temperature tensile strength, accompanied by a marked reduction in room- and elevated-temperature ductility:

1. The most prominent loss in ductility is associated with aging at 1300°F. These properties are associated with the presence of the Widmanstätten precipitate.
2. A significant loss in ductility accompanied by an increase in room-temperature hardness occurs upon aging at 1100°F. The mechanism which produces this change appears to be a submicroscopic beta precipitate, with the phase diagram assumed to be correct.

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