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METALLOGRAPHY AND AGING CHARACTERISTICS
OF HASTELLOY B

J. R. Riddle
R. J. Gray



OAK RIDGE NATIONAL LABORATORY

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METALS AND CERAMICS DIVISION

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JUNE 1964

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METALLOGRAPHY AND AGING CHARACTERISTICS OF HASTELLOY B

J. R. Riddle¹ and R. J. Gray

ABSTRACT

Investigations of the high-temperature properties of Hastelloy B have shown that age hardening strongly influences the mechanical properties of the material. This report shows the microstructures and hardness data for isothermal heat treatments at five temperatures between 700 and 1000°C for periods to 1600 hr. To show the effects of aging, etching conditions are duplicated as closely as possible, and the details of the microstructures are shown at high optical magnifications and in electron micrographs. Polishing and etching techniques are also described. The largest increase in hardness occurred at the lowest temperature of the investigation, 705°C, due to precipitation in the form of Widmanstätten platelets. Evidence is presented that indicates the platelets to be Ni₃Mo. The spheroidal carbides in wrought Hastelloy B are shown to have an M₆C structure.

INTRODUCTION

The excellent corrosion resistance and high-temperature properties of Hastelloy B have prompted studies of the alloy at the Oak Ridge National Laboratory for possible reactor application. In the course of these investigations, it became apparent that the age-hardening characteristics of the alloy are extremely temperature dependent. Efforts to correlate microstructures and to determine the aging characteristics of the alloy were only moderately successful in evaluating random specimens. Therefore, the microstructural examination of a series of isothermal heat treatments of the same stock of material was undertaken.

The purpose of this investigation was to determine the microstructures and hardness values for heat treatments at temperatures of interest for nuclear applications and identify the products or phases by x-ray diffraction.

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AGING TREATMENTS

The material employed in this investigation was commercial 1/16-in. sheet and all specimens were from the same heat. Table 1 shows the chemical composition of this material. Prior to the isothermal heat treatments, the sheet was annealed 2 hr at 1150°C in a hydrogen atmosphere, then air cooled. The annealed sheet was sectioned into metallographic samples 1/4 × 3/4 in. and the individual samples were encapsulated in quartz tubing. During encapsulation, the tubing was flushed with argon and sealed under a negative gage pressure of argon.

Table 1. Composition of Hastelloy B

Element	Method of Analysis	Weight Percent	
		Min	Max
Nickel	Gravimetric	62.2	64.0
Molybdenum	Colorimetric	26.5	27.2
Iron	Volumetric	4.46	5.15
Silicon	Gravimetric	0.54	0.65
Carbon	Direct Combustion	0.03	0.05
Chromium	Colorimetric	0.26	0.34
Titanium	Colorimetric	0.063	0.077
Vanadium	Colorimetric	0.077	0.167
Sulfur	Polarigraphic	0.001	0.002
Phosphorus	Colorimetric	0.006	0.008
Manganese	Colorimetric	0.99	1.06
	Total	95%	99%

The isothermal heat treatments were conducted at temperatures of 705, 760, 815, 900, and 980°C for periods of 100, 200, 400, 800, 1200, and 1600 hr using resistance furnaces. Since considerable aging was evident in the microstructure and in the microhardness values, especially at the lower temperature of 705°C, the effect of prior heat treatment was studied by aging additional specimens for 1200 hr at 760, 815, or 900°C and then isothermally heat treating for 800 hr at 705°C.

Furnace temperatures throughout the heat treatments were maintained within $\pm 3^{\circ}\text{C}$ by means of Chromel-P/Alumel thermocouples and Brown recording controllers.

SPECIMEN PREPARATION

Grinding and Polishing

Hastelloy B metallographic specimens were ground and polished by methods similar to those described for nickel alloys in the Metals Handbook.² The specimens were mounted in Bakelite and the surface metal was removed by power grinding on a 240-grit silicon carbide belt. Initial grinding was followed by hand grinding on 320-, 400-, and 600-grit silicon carbide papers. Specimens were polished on rotary wheels using Linde A (0.3- μ alumina) on silk cloth, Linde A on Microcloth,³ and finally, Linde B (0.1- μ alumina) on Microcloth. Water was employed as the vehicle for all grinding and polishing operations.

The high degree of polish necessary for electron microscopy was accomplished by polishing the specimen 4 to 8 hr on a vibratory polisher using Microcloth and Linde B alumina. The final polish was obtained with magnesium oxide (Magomet³) and Microcloth on a motor-driven polishing wheel at 100 rpm.

The polishing behavior of Hastelloy B is strongly dependent on the heat treatment. The solution-annealed material was relatively soft and presented problems much like those for pure nickel. These materials usually required alternate polishing and etching to remove disturbed metal. A final polish with Magomet aided in the removal of fine scratches. The age-hardened materials presented no special polishing problems. In wrought Hastelloy B in the as-polished condition, carbides were outlined in relief.

²Taylor Lyman (ed.), Metals Handbook, p. 1044, American Society for Metals, Cleveland, Ohio, 1948.

³Buehler, Ltd., 2120 Greenwood Street, Evanston, Illinois.

Etching

Etching reagents employed at ORNL and recommended by the Haynes Stellite Company⁴ are listed in Table 2. The etching behavior of Hastelloy B depends on heat treatment. Dilute solutions (see reagents 1, 3, 4, and 9 of Table 2) are most suitable for etching the aged samples, but preferential etching and staining must be considered in evaluating the microstructure. Etching depends on grain orientation in wrought material. This effect is apparent in comparing the etching behavior at grain boundaries, at twin-matrix interfaces, and of precipitates within different grains. All of the etchants investigated exhibited various degrees of preferential etching.

Stains or water spots are often a problem after etching Hastelloy B. However, these stains can be held to a minimum by the method recommended by the Haynes Stellite Company (i.e., by soaking the etched specimen in boiling distilled water and then rinsing with alcohol and drying with forced air). Staining of a different nature occurs when fine Widmanstätten platelets are present; these stains are a corrosion product and are most evident in heavily etched samples. They often develop after lightly etched samples have been exposed to air for several minutes. The stains occur in localized regions and tend to obscure the amount and shape of the actual precipitate. Reagent No. 9 is probably the most suitable etchant for these microstructures, because initial staining is held to a minimum and the rate of attack is moderate.

To show the aging characteristics of Hastelloy B, photomicrographs are presented with closely duplicated etching conditions. The etching periods chosen were 20 sec for the solution-annealed material and 5 sec for specimens aged at temperatures between 705 and 900°C and 15 sec for specimens aged at 980°C. All etching was by immersion in reagent No. 1 of Table 2. Like any nickel-base alloy containing a large quantity of precipitate, the specimens aged at 705 and 760°C for the longer periods were rapidly attacked by this reagent; in fact, the etching period of

⁴Haynes Alloys for High-Temperature Service, p. 83, Haynes Stellite Company, Kokomo, Indiana, 1950.

Table 2. Etching Reagents Used for Nickel-Base Alloys

Reagent	Composition	Etching Time	Microstructure Revealed	Remarks
1 ^a	1 part HCl (conc) 1 part 10% chromic ^b 1 part water	2 sec to 1 min	any structure	Strength of solution is not critical. Etching rate can be controlled by HCl concentration. The etching end point can be readily controlled if etching time is 10 sec or more.
2 ^a	1 part HCl (conc) 1 part 10% chromic ^b	5 sec to 1 min	all structures except Widmanstätten platelets	
3	3 parts HCl (conc) 1 part HNO ₃ (conc) 4 parts glycerin	10 sec to 1 min	any structure	Use freshly prepared solution.
4	1 part H ₃ PO ₄ (conc) 4 parts H ₂ O ₂ (3%)	10 sec to 1 min	Widmanstätten platelets	Use freshly prepared solution.
5	1 part HCl (conc) 1 part HNO ₃ (conc) 1 part water	5 sec to 1 min	solution-annealed structure	Recommended only when reagent No. 2 reacts too slowly.
6	1 part HCl (conc) 9 parts H ₂ O ₂ (3%)	1 sec to 5 sec	all structures except Widmanstätten platelets	Electrolytic etch. Use 2 dry cells in series with carbon cathode.
7 ^a	1 part HCl (conc) 10 to 20 parts water	1 sec to 5 sec	all structures	Electrolytic etch. Use 2 dry cells in series with carbon cathode.
8 ^a	1 part HNO ₃ (conc) 1 part water	1 sec to 5 sec	all structures	
9	1 part HCl (conc) 10 to 20 parts alcohol	10 sec to 1 min	Widmanstätten platelets	Electrolytic etch. Use 1 dry cell (1 1/2 v) with carbon cathode.

^aReagents or variations of reagents recommended by Haynes Stellite Company.

^bChromic acid solution contains 10 g CrO₃ to each 100 ml water.

5 sec is too long. However, since a comparison of the relative amounts of precipitate formed is the real purpose of this investigation, the etching time variations were held to a minimum.

The chemical etching used for optical microscopy was also satisfactory for electron microscopy, except for the specimens annealed at 1150°C and those aged at 980°C. Apparently the relief pattern after etching for these two treatments was so high that the Formvar replicas would tear quite readily. Consequently, vacuum cathodic etching was used with better results.

MICROSTRUCTURAL EXAMINATION

Nickel-Molybdenum-(X) Compounds in Hastelloy B

The precipitates that form in Hastelloy B have never been satisfactorily identified. One reason is the incompleteness of the knowledge of the phases and phase boundaries in the nickel-molybdenum system as shown by the dashed lines in the phase diagram of Fig. 1, taken from Hansen and Anderko.⁵ The known intermediate phases in this binary system are MoNi₄ (beta), which has either a face-centered tetragonal system lattice⁵ or a body-centered tetragonal superlattice;⁶ MoNi₃(gamma), which has a close-packed hexagonal structure;⁵ and MoNi (delta). The crystal structure of delta has not been fully established, but Shoemaker⁷ has proposed a tentative structure with tetragonal symmetry.

Badger and Sweeney⁸ reported in 1946 that aging in Hastelloy B was due to the precipitation of molybdenum-nickel-iron intermetallic compounds. In a study of the aging characteristics of Hastelloy B,

⁵M. Hansen and K. Anderko, Constitution of Binary Alloys, 2nd ed., p. 968, McGraw Hill, New York, 1958.

⁶P. V. Guthrie and E. E. Stansbury, X-Ray and Metallographic Study of the Nickel-Rich Alloys of the Nickel-Molybdenum System II, ORNL-3078 (July 3, 1961).

⁷D. P. Shoemaker, Clara Brink, and Alvin Fox, Preliminary Structural Results on P Phase (Mo-Ni-Cr) and Delta Phase (Mo-Ni) and Structural Relationships of These and Other Phases to the Sigma Phase, Technical Report No. 1 to Office of Ordinance Research, Project TB2-0001 (April 25, 1955).

⁸F. S. Badger and W. O. Sweeney, Jr., "Metallurgy of High Temperature Alloys Used in Current Gas Turbine Designs," Symposium on Materials for Gas Turbines, Proceedings, pp. 99-112, American Society for Testing and Materials.

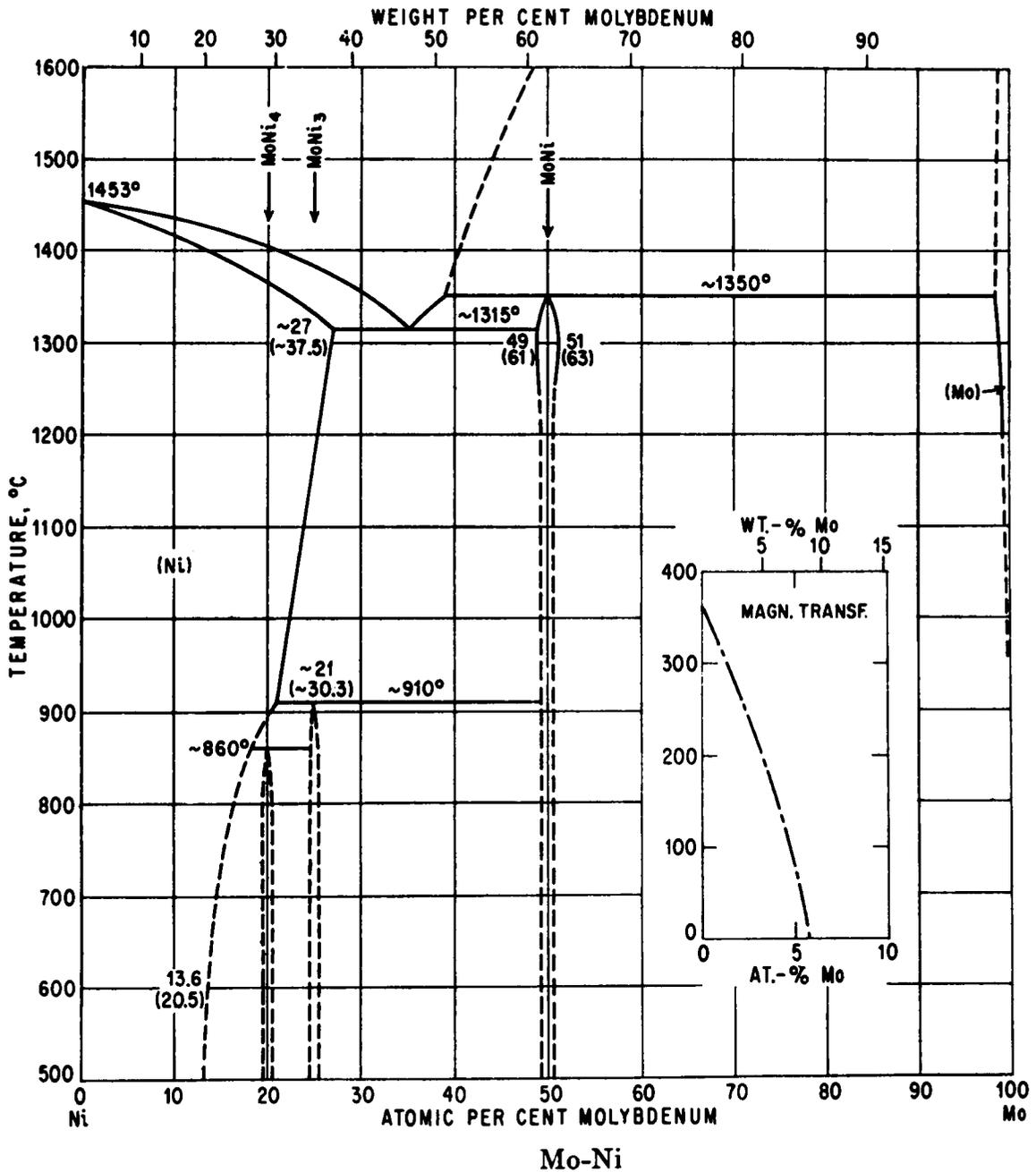


Fig. 1. Nickel-Molybdenum Phase Diagram by Hansen and Anderko.

Clausing *et al.*⁹ tentatively identified beta as the precipitate causing hardening at 590 and 705°C and gamma as the precipitate formed at 815°C. In the present investigation, powder x-ray diffraction patterns were obtained from specimens aged 1600 hr at 705, 760, 815, 900, and 980°C. All patterns showed lines for alpha-nickel and additional lines that did not correspond to any nickel-molybdenum phases. Line broadening prevented an analysis for the gamma phase.

Effect of Aging Treatment on Microstructure

The microstructure of the alloy prior to the isothermal heat treatments consisted of spheroidal carbides in an alpha solid-solution matrix (Fig. 2). The carbides were very stable - not changing in appearance throughout the isothermal heat treatments.

The isothermal heat treatment at 705°C produced fine Widmanstätten platelets (probably beta) as shown in Fig. 3. Since the etching conditions (i.e., composition and temperature of etchant and the etching period) were the same for each of the six specimens, undoubtedly equilibrium conditions were not attained after 1600 hr and more platelets would have precipitated during a prolonged heating period.

Aging at 760°C produced a microstructure similar to aging at 705°C, but the platelets precipitated more slowly, were not as fine, and as in the 705°C treatments, did not appear to have reached equilibrium conditions (i.e., more would precipitate with a longer heat treatment); see Fig. 4.

At the higher temperature of 815°C, the precipitate had a fine spheroidal shape (Fig. 5) and apparently reached an equilibrium in 400 to 800 hr.

At 900°C there was more globular precipitate after 100 hr, and the microstructure appeared stable after a 200- to 400-hr isothermal heat treatment (Fig. 6).

⁹R. E. Clausing, P. Patriarca, and W. D. Manly, *Trans. Am. Soc. Metals* 51, 123 (1959).

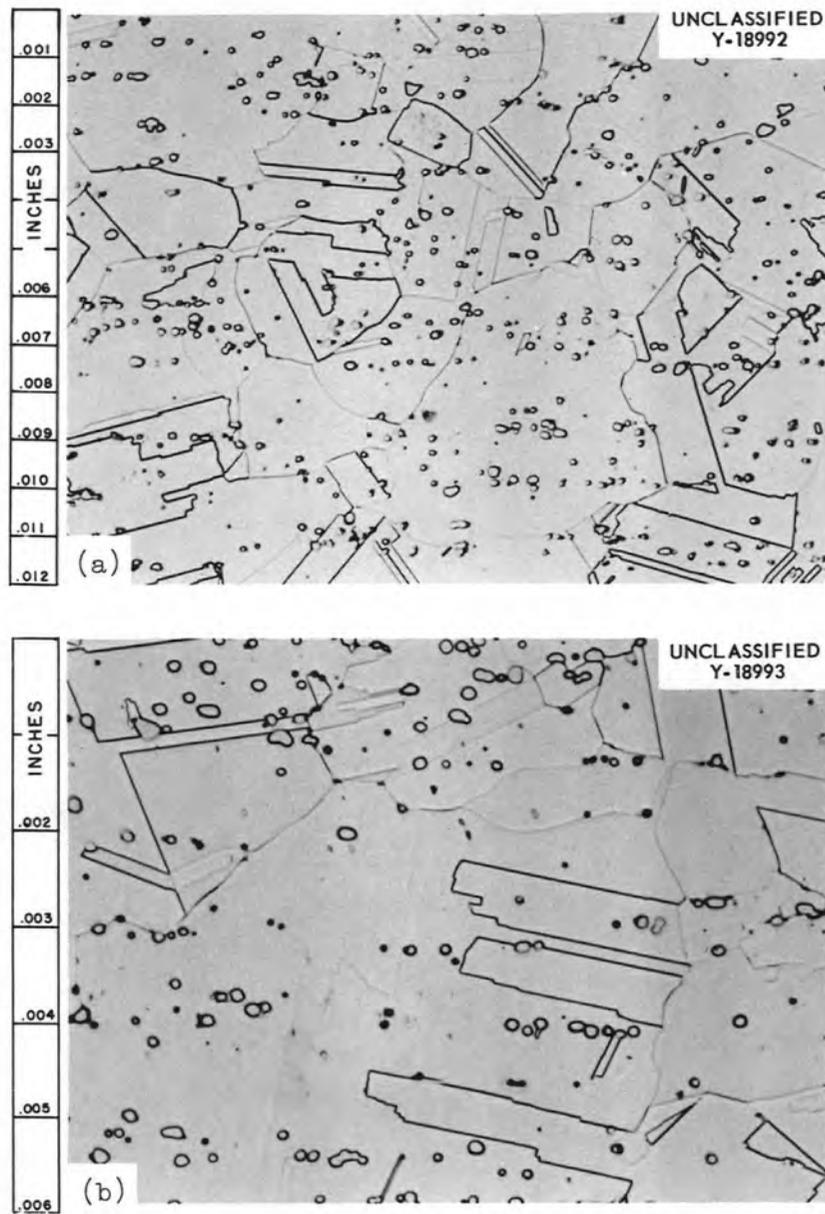


Fig. 2. Hastelloy B Solution Annealed 2 hr at 1150°C. The microstructure of the material prior to the isothermal heat treatments shows carbides in an alpha-nickel matrix. Many of the voids (dark spots) result from loss of carbide particles during grinding and polishing and from preferential attack of the etchant at carbide-matrix interfaces. (a) 250X. (b) 500X.

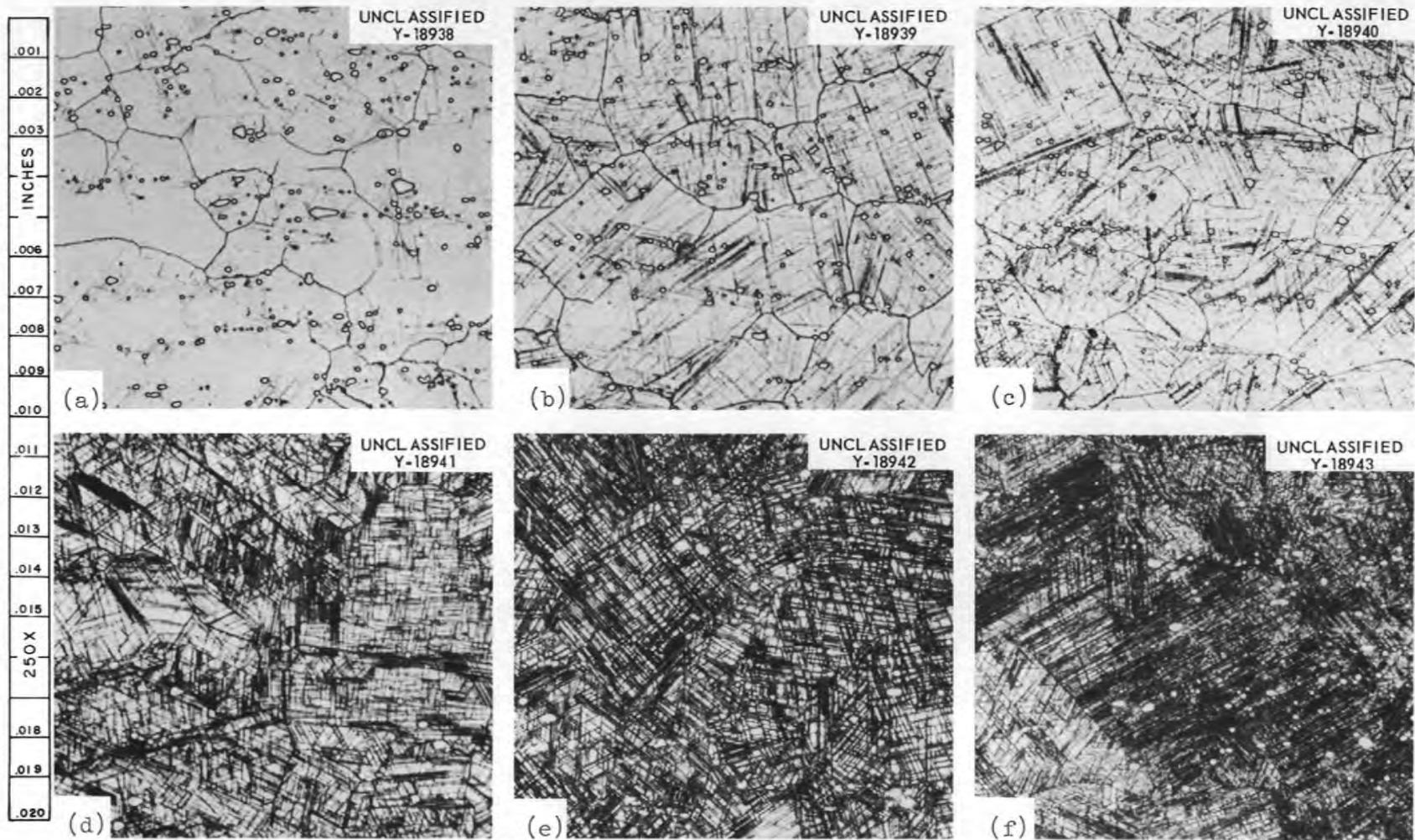


Fig. 3. Hastelloy B Aged for Various Periods at 705°C. All samples were etched with reagent No. 1 for 5 sec. 250X. (a) 100 hr. (b) 200 hr. (c) 400 hr. (d) 800 hr. (e) 1200 hr. (f) 1600 hr.

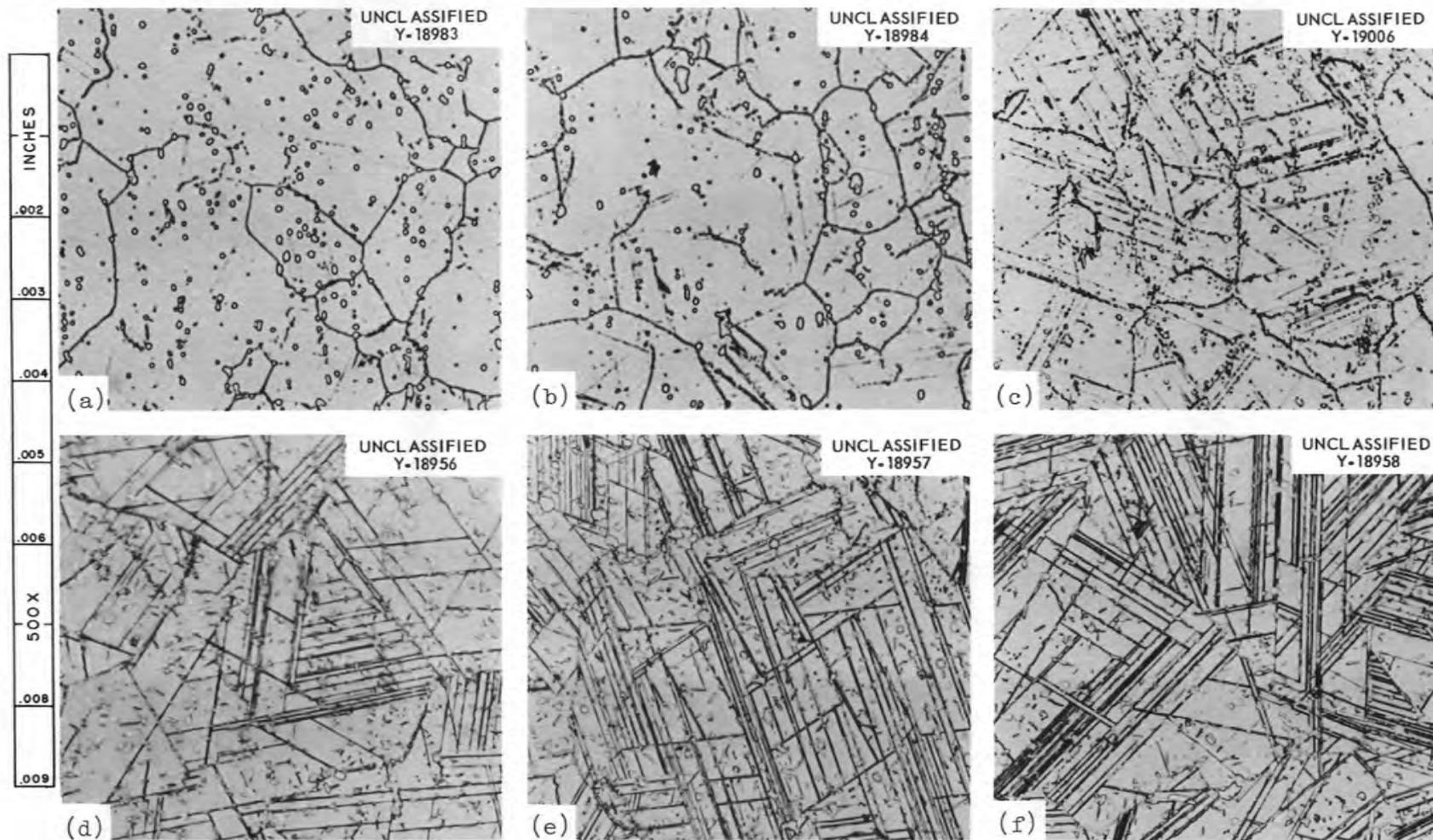


Fig. 4. Hastelloy B Aged for Various Periods at 760°C. All samples were etched with reagent No. 1 for 5 sec. 250x. (a) 100 hr. (b) 200 hr. (c) 400 hr. (d) 800 hr. (e) 1200 hr. (f) 1600 hr.

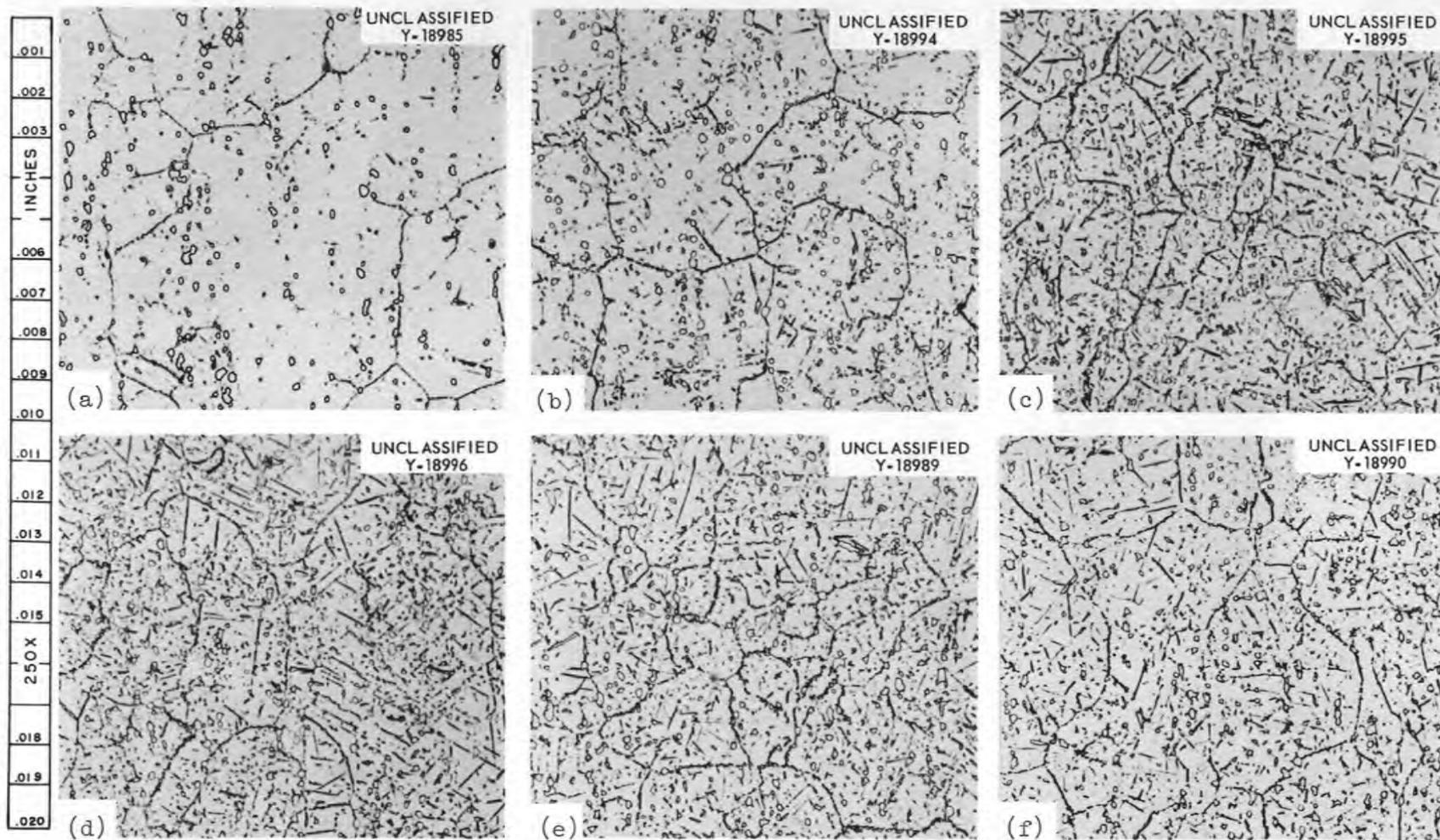


Fig. 5. Hastelloy B Aged for Various Periods at 815°C. All samples were etched with reagent No. 1 for 5 sec. 250X. (a) 100 hr. (b) 200 hr. (c) 400 hr. (d) 800 hr. (e) 1200 hr. (f) 1600 hr.

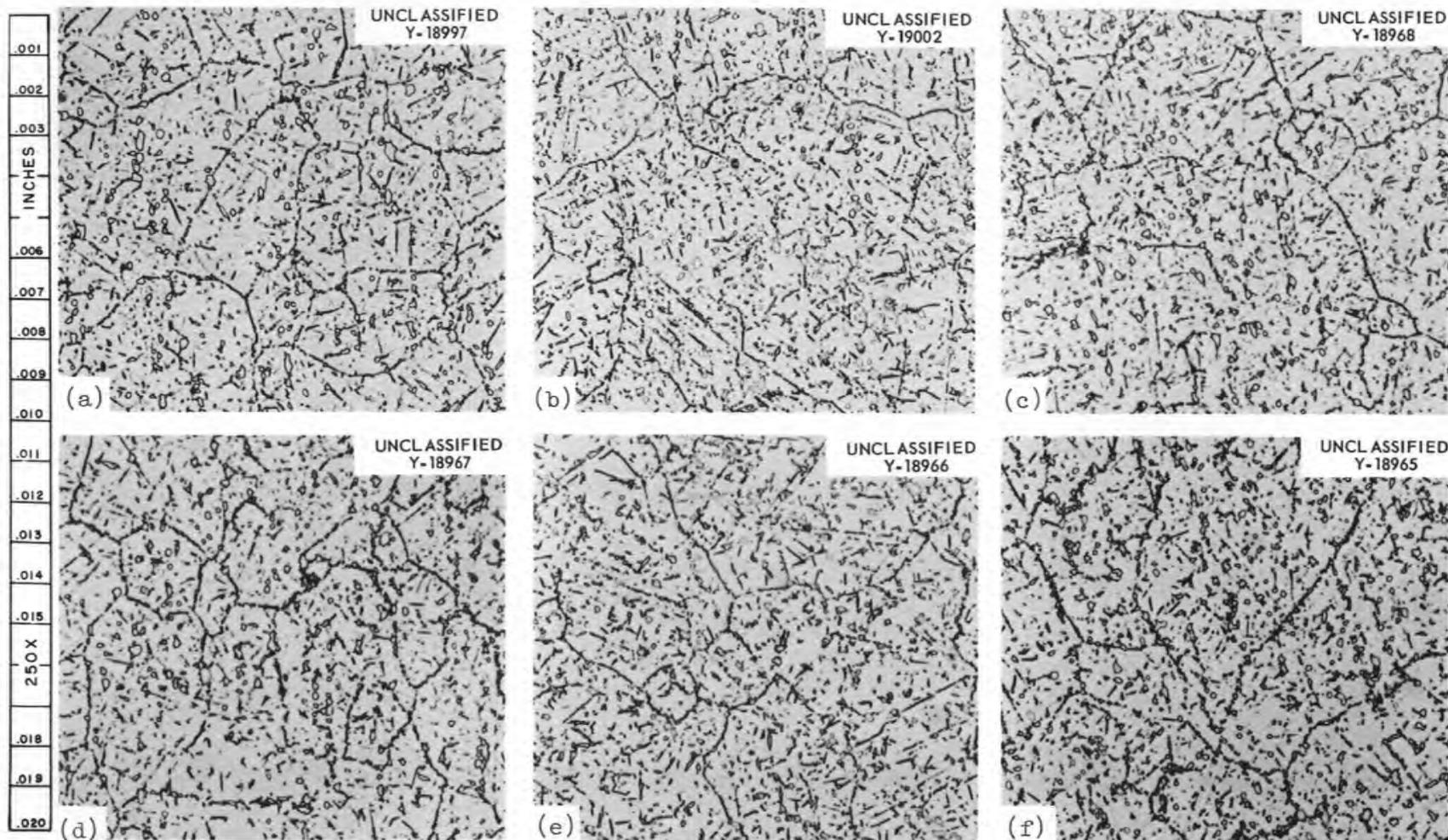


Fig. 6. Hastelloy B Aged for Various Periods at 900°C. All samples were etched with reagent No. 1 for 5 sec. 250X. (a) 100 hr. (b) 200 hr. (c) 400 hr. (d) 800 hr. (e) 1200 hr. (f) 1600 hr.

At 980°C, equilibrium was attained in 100 to 200 hr and the precipitate was coarser (Fig. 7). The etching period had to be increased to 15 sec for this group of specimens.

All these observations pertaining to equilibrium conditions were substantiated by hardness data, which will be presented later.

In evaluating the amount of precipitation present in specimens of this type, the use of metallographic techniques must be applied with caution. Examination of specimens that have been randomly etched could be very misleading, since the etching period is extremely influential in the apparent amount of precipitation present. To show this effect, the specimens aged 400 and 1600 hr at 705°C (see Figs. 4c and f) were repolished and etched 15 and 2 sec, respectively, with reagent No. 1, and the results are shown in Figs. 8 and 9. The etching period obviously affects the apparent amount of precipitate.

The shapes and amounts of precipitate after heat treatment at the five temperatures (705, 760, 815, 900, and 980°C) for 1600 hours are compared at 2000x in Fig. 10.

If the service temperature of Hastelloy B should be near 705°C, the formation of the fine network of fine platelets undoubtedly would adversely affect tensile and impact properties. The presence of the spheroidal precipitate at the higher temperatures suggests the use of a prior heat treatment above 705°C to prevent or retard the formation of the fine platelets in service. To investigate this, separate specimens were aged 1200 hr at 760, 815, and 900°C then heat treated 800 hr at 705°C. The resulting microstructures (Fig. 12) differed noticeably from that of the specimen that received only the 800-hr treatment at 705°C (Fig. 3d), especially the two specimens that received the prior heat treatments at 815 and 900°C. The hardness values will be compared later.

The main objection to the above suggestion for reducing the amount of fine platelets would be the impracticality of heat treatment at the high temperature.

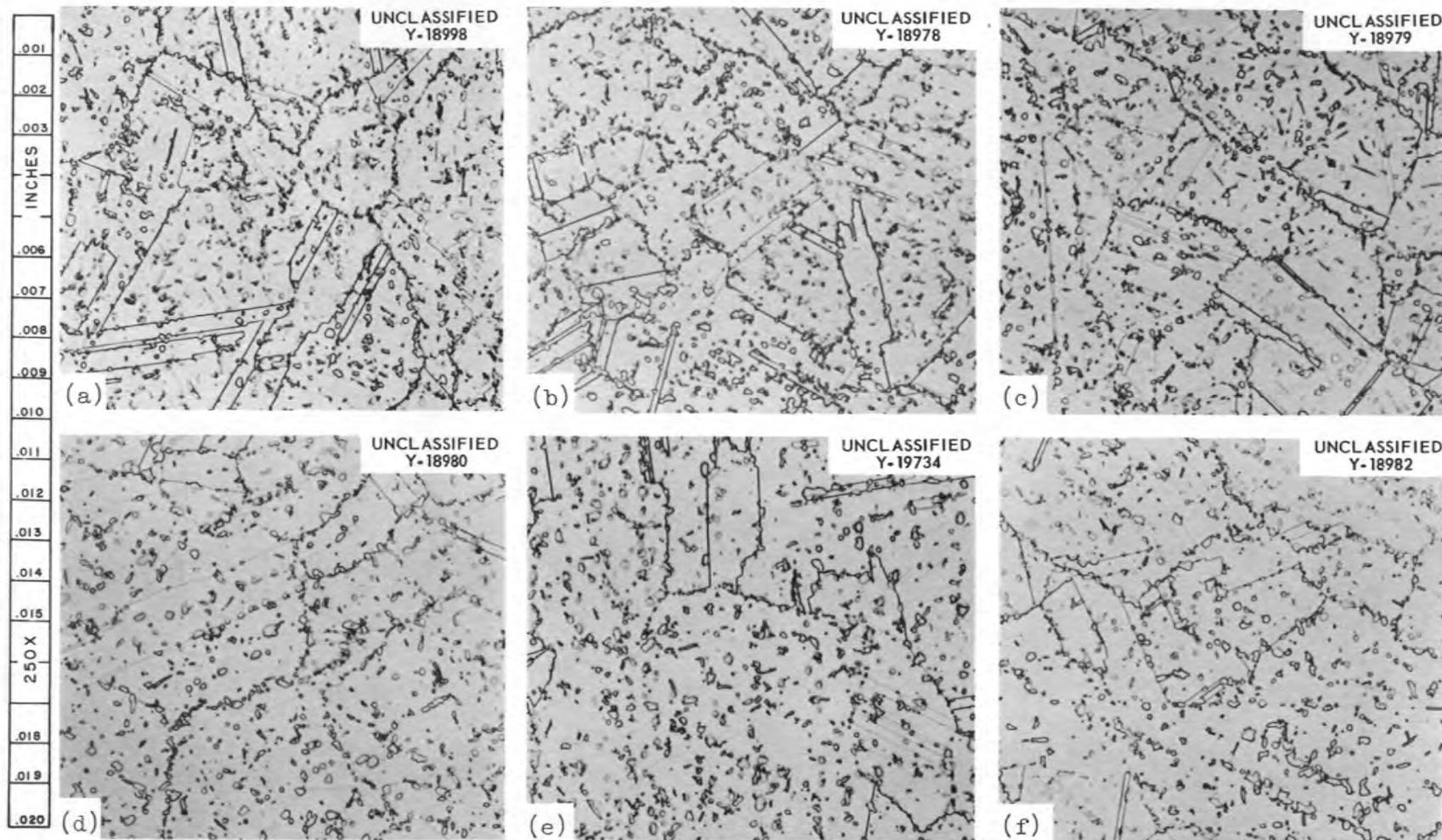


Fig. 7. Hastelloy B Aged for Various Periods at 980°C. All samples were etched with reagent No. 1 for 15 sec. 250X. (a) 100 hr. (b) 200 hr. (c) 400 hr. (d) 800 hr. (e) 1200 hr. (f) 1600 hr.

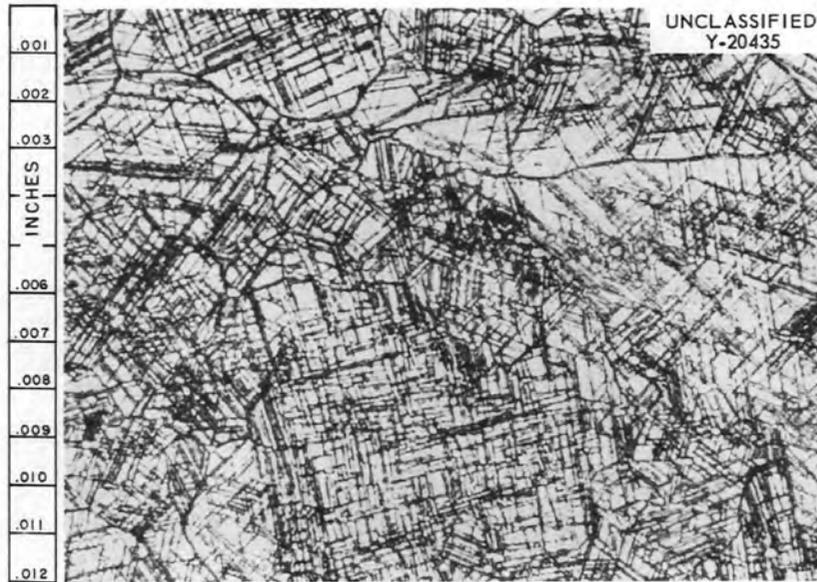


Fig. 8. Microstructure of Hastelloy B Aged 400 hr at 705°C and Etched 15 sec with Reagent No. 1. 250X.

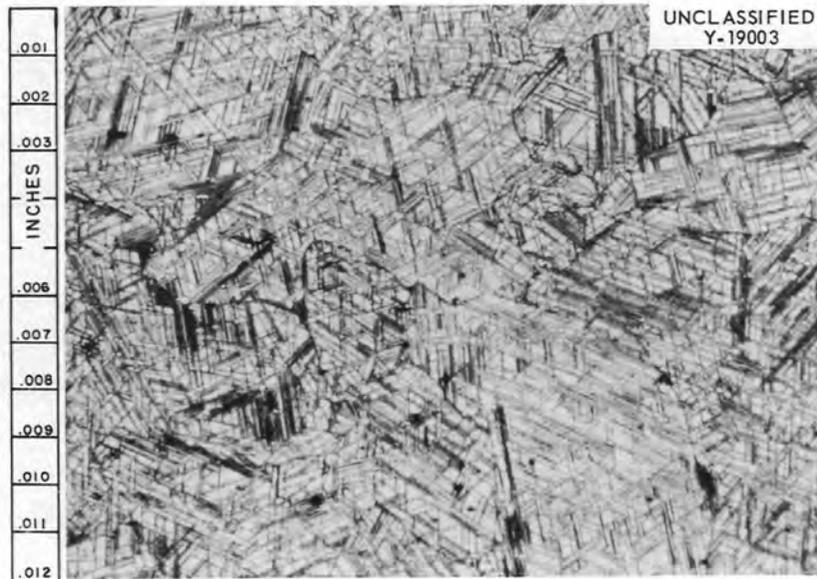


Fig. 9. Microstructure of Hastelloy B Aged 1600 hr at 705°C and Etched 2 sec with Reagent No. 1. 250X.

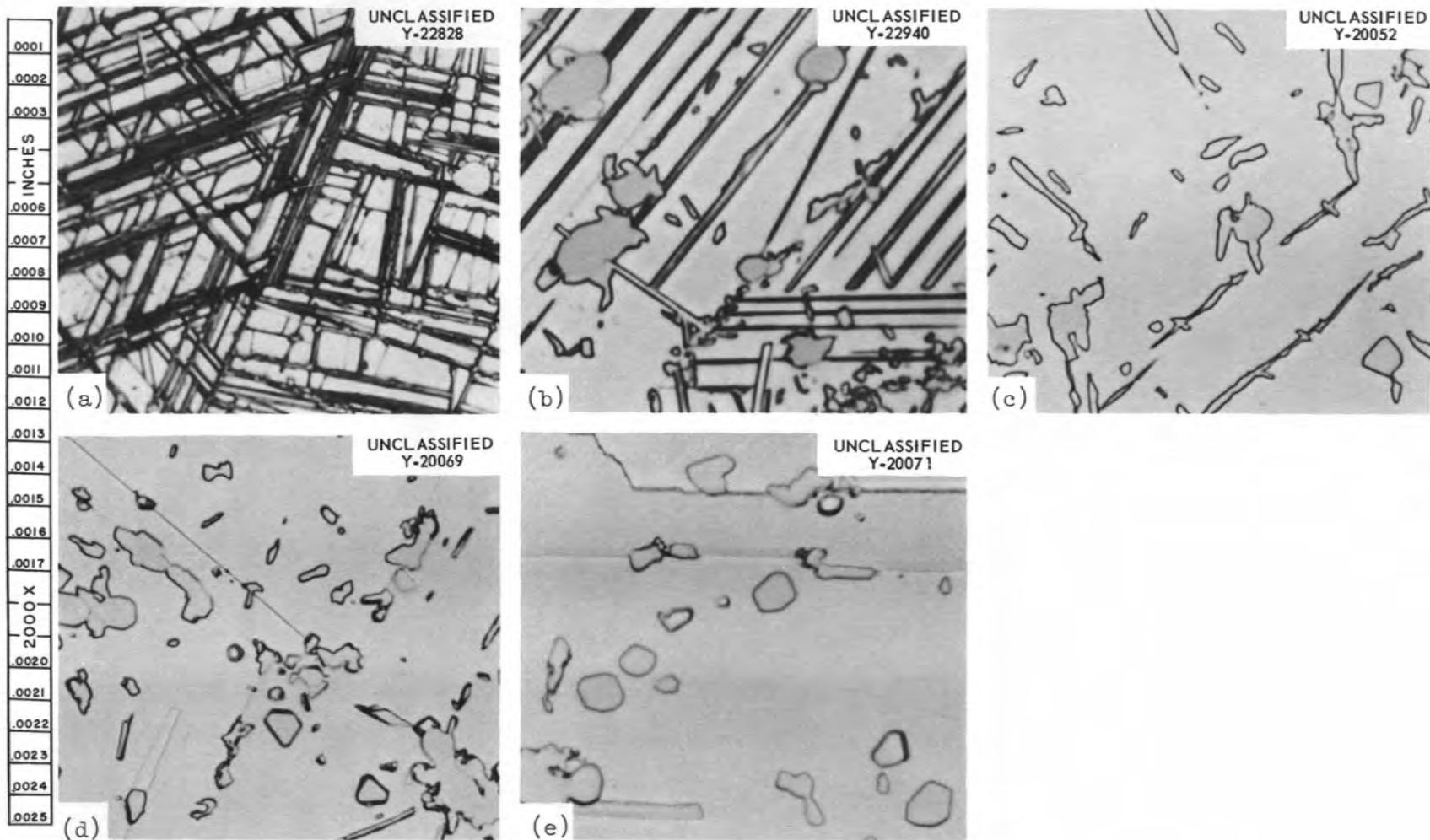


Fig. 10. Microstructures After 1600-hr Heat Treatments. Etchant: reagent No. 1. 2000x. (a) 705°C. (b) 760°C. (c) 815°C. (d) 900°C. (e) 980°C.

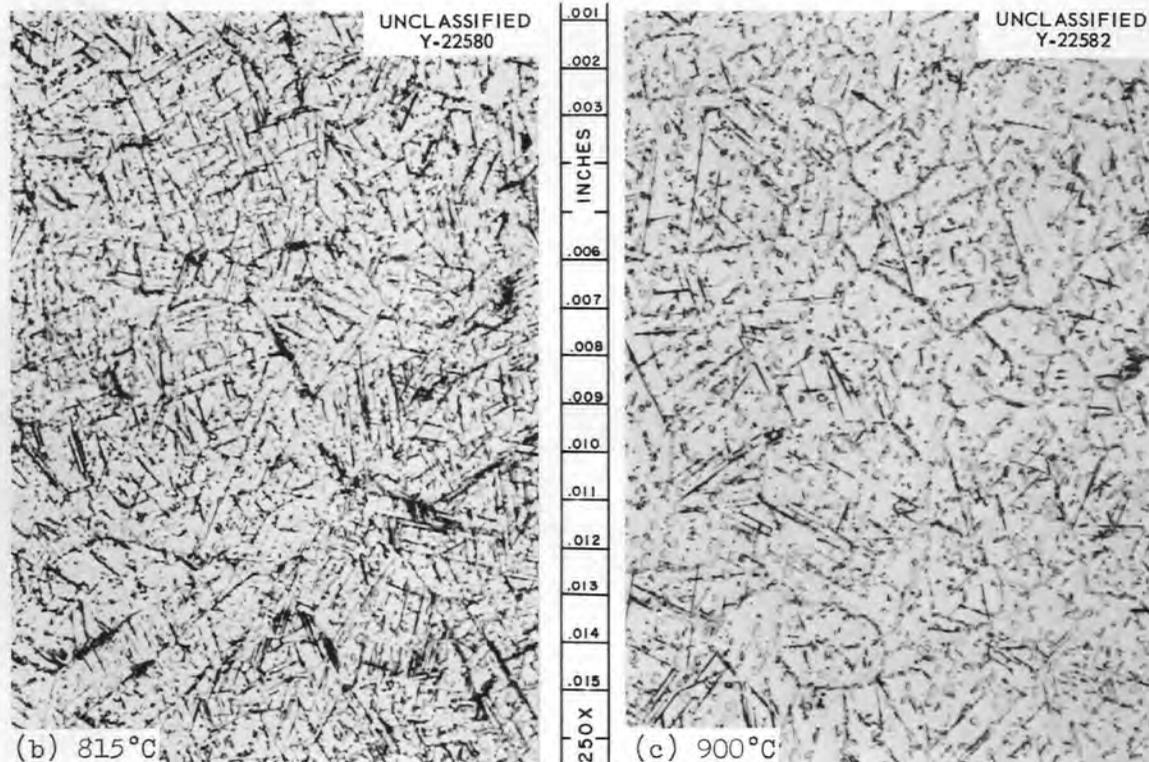
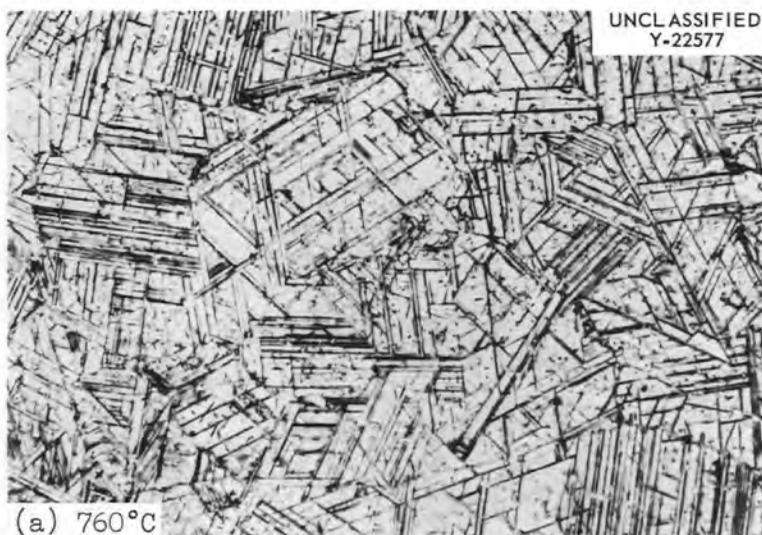


Fig. 11. Investigation of the Effect of Over-Aging Heat Treatments. The specimens were heat treated for 1200 hr at the indicated temperatures followed by a secondary aging heat treatment for 800 hr at 705°C. The effect of the secondary heat treatment can be determined by comparing the above microstructures with Figs. 4e, 5e, and 6e. The effect of the primary heat treatments can be determined by comparing the above microstructures with Fig. 3d. The etching conditions were the same as those used for the isothermal heat treatments. 250x.

Carbides

As mentioned previously, the spheroidal carbides appear in wrought and annealed specimens (Fig. 12) and remain essentially unchanged throughout the isothermal-heat treatments of this investigation. These results concur with those of Badger and Sweeney.¹⁰ The carbides stand in relief in the "as-polished" condition and appear to be inert to the etchants of Table 2. The carbides can be stained as shown in Fig. 12. However, the stains vary in color and can be confused with massive nickel-molybdenum precipitates. The carbides in Hastelloy B have been separated from the matrix by Hays¹¹ through electrolytic dissolution (see Appendix A) and appear to have an M_6C structure, according to the work of Cavin¹² (see Appendix B). Roche,¹³ in a study of Hastelloy B melts of varying carbon content, has shown that carbide particles form by a eutectic reaction and are coalesced into spheroidal particles during hot working. This mechanism would account for the tendency of the carbide particles to form in groups and to align in the direction of working.

Electron Microscopy

Electron micrographs were prepared to show the details of the microstructure after solution annealing 2 hr at 1150°C and also after the 1600-hr heat treatments at 705, 760, 815, 900, and 980°C. The electron micrographs were prepared from Formvar replicas shadowed with gold-manganin.

The relative elevations of the matrix and precipitates can be determined from the carbide particles in the microstructures. These stand in relief and are shadowed with light and dark sides, depending on their positions relative to the shadowing vapors. All the precipitates in these materials were elevated relative to the matrix.

The solution-annealed material, as shown in Fig. 13, was generally free of precipitates other than carbides. After 1600 hr at 705 or 760°C

¹⁰F. S. Badger and W. O. Sweeney, Jr., op. cit.

¹¹C. Hays, private communication.

¹²O. B. Cavin, private communication.

¹³T. K. Roche, private communication.

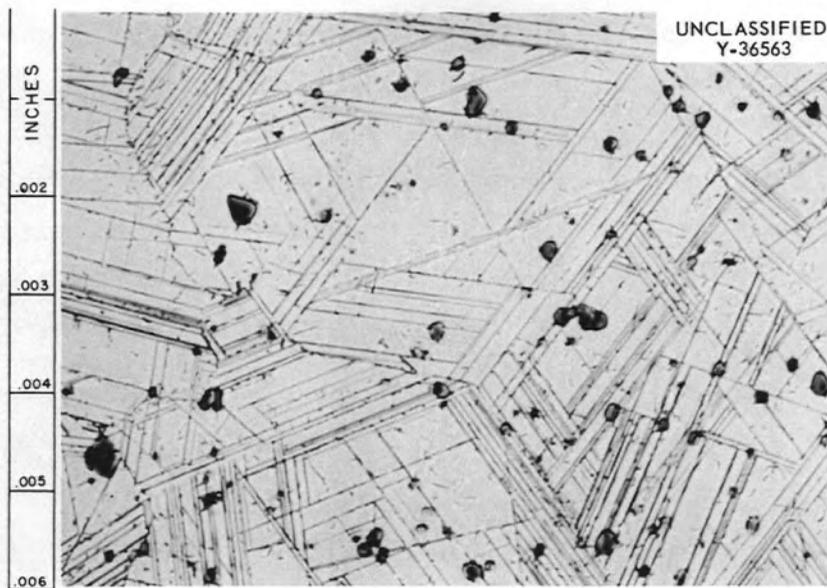


Fig. 12. Hastelloy B Aged 1600 hr at 760°C. Reagent No. 9 was used to show Widmanstätten platelets, and a KMnO_4 -NaOH etchant was used to stain the carbides. 500X.

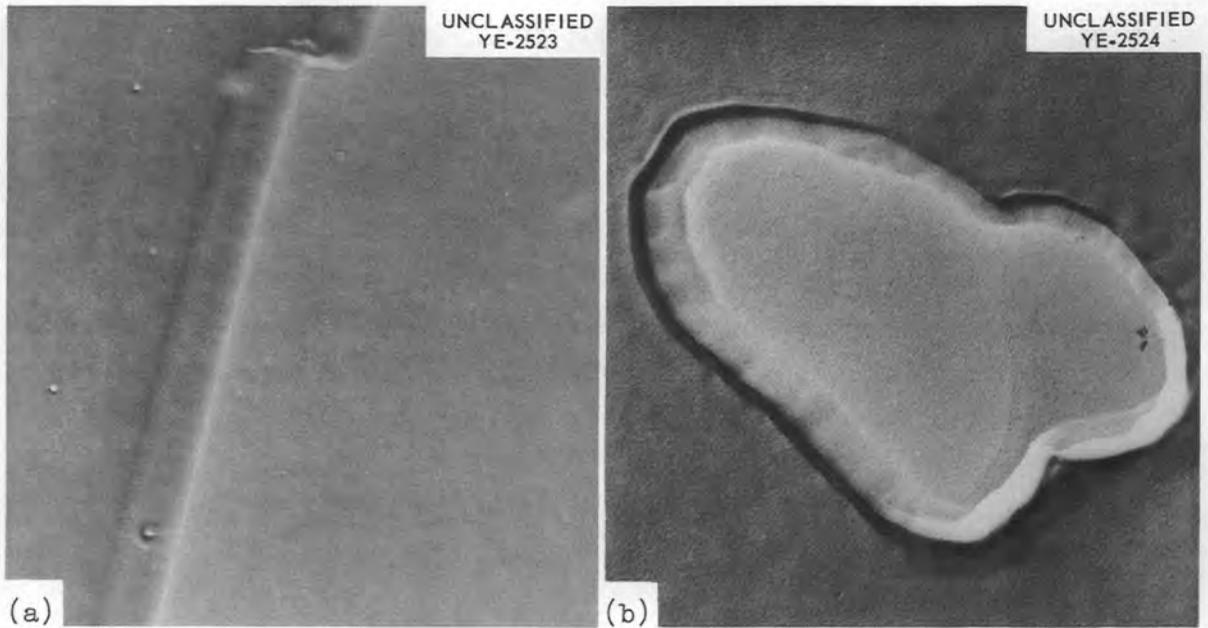


Fig. 13. Hastelloy B After Solution-Annealing Heat Treatment of 2 hr at 1150°C. Note the absence of any fine matrix precipitate. (a) Typical appearance of grain boundary. 8,200X. (b) Typical appearance of carbide undissolved by this treatment. Vacuum cathodic etch. 16,300X.

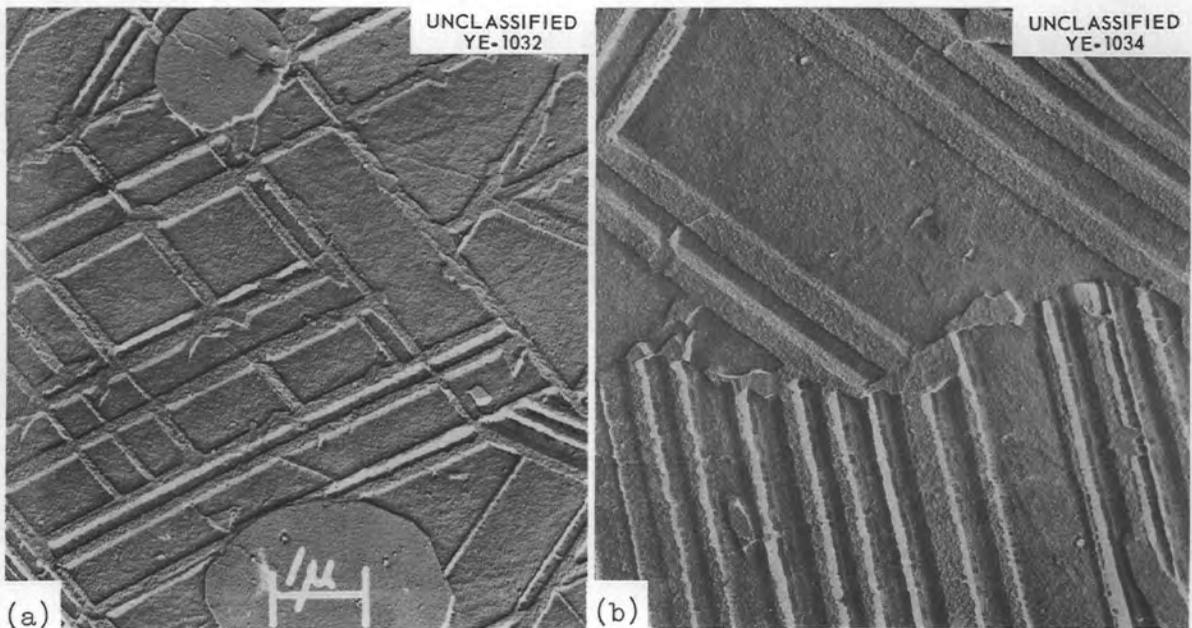


Fig. 14. Hastelloy B Heated 1600 hr at 705°C, Showing the Globular Carbides and the Widmanstätten Precipitate of Gamma Phase. Etched with reagent No. 1. (a) 12,000X. (b) 16,700X.

(Figs. 14 and 15), precipitation had occurred along the (111) planes of the matrix, at grain boundaries, and along carbide-matrix interfaces. The surface texture of the grain boundary precipitates and the Widmanstätten platelets in Fig. 16 show that the precipitates are the same phase. The more massive precipitates that formed at 815, 900, and 980°C (Figs. 16, 17, and 18) showed a greater tendency to form at grain boundaries and carbide-matrix interfaces. These precipitates have not been satisfactorily identified. For comparison, Fig. 19 shows the microstructure of a binary nickel-molybdenum alloy aged in the alpha-gamma region of the phase diagram of Fig. 1. A close similarity is evident between the Widmanstätten platelets formed in the binary alloy and those formed in Hastelloy B at 705°C (Fig. 14).

HARDNESS

Figure 20 shows the diamond-pyramid hardness of Hastelloy B as a function of time and temperature of aging. These curves are based on four impressions, each using a 10-kg load, on specimens aged 100, 200, 400, 800, 1200, and 1600 hr at each temperature. The normal hardness spread on each specimen was less than 10 points. The data of Fig. 20 were obtained on specimens that had been air cooled from the aging temperature. However, water-quenched specimens showed no notable difference either in hardness or in microstructure.

The amount of age hardening decreased with increasing aging temperature over the range investigated. The sluggishness of the precipitation reaction in Hastelloy B is shown by an increase in the hardness over a period of 1600 hr at 760°C and also by the time required to over-age at temperatures of 900 and 980°C. A comparison of the microstructures of Figs. 3 through 7 with the hardness curves of Fig. 20 shows that the age hardening that occurs at 705 and 760°C can be correlated with the formation of Widmanstätten platelets. Clausing *et al.*¹⁴ noted a sharp loss in ductility for Hastelloy B aged 1000 hr at 705°C and reported the hardness in this condition to be

¹⁴R. E. Clausing, P. Patriarca, and W. D. Manly, *op. cit.*

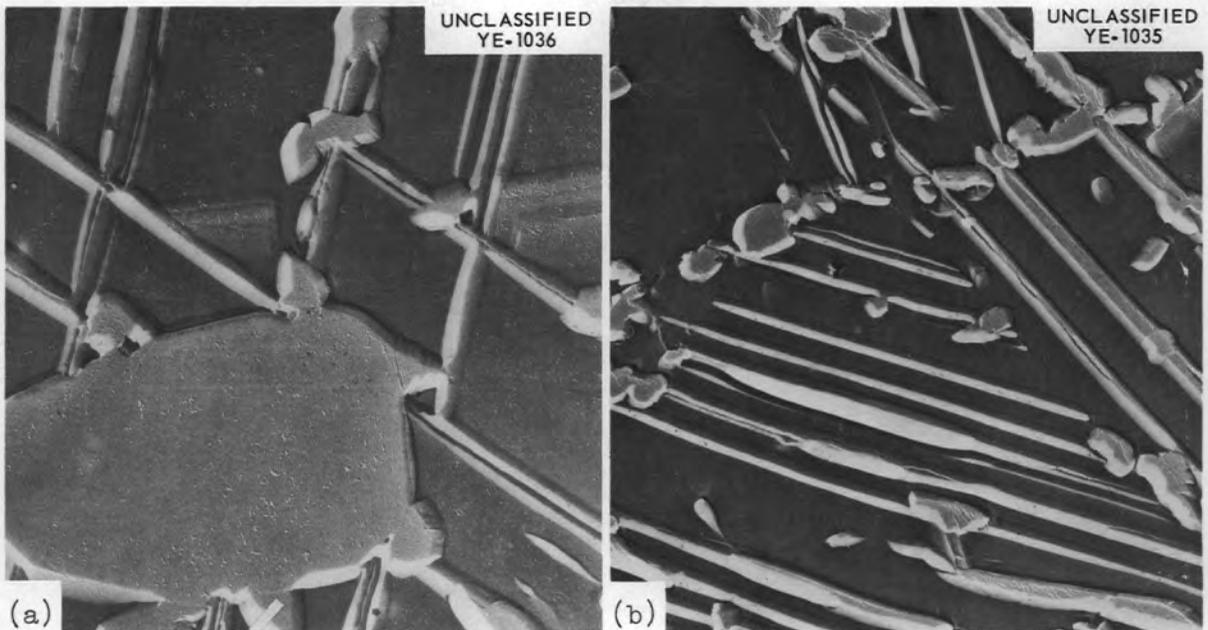


Fig. 15. Hastelloy B Heated 1600 hr at 760°C, Showing Widmanstätten Platelets and Grain Boundary Precipitate. Etched with reagent No. 1.
 (a) 9,000x. (b) 3,800x.

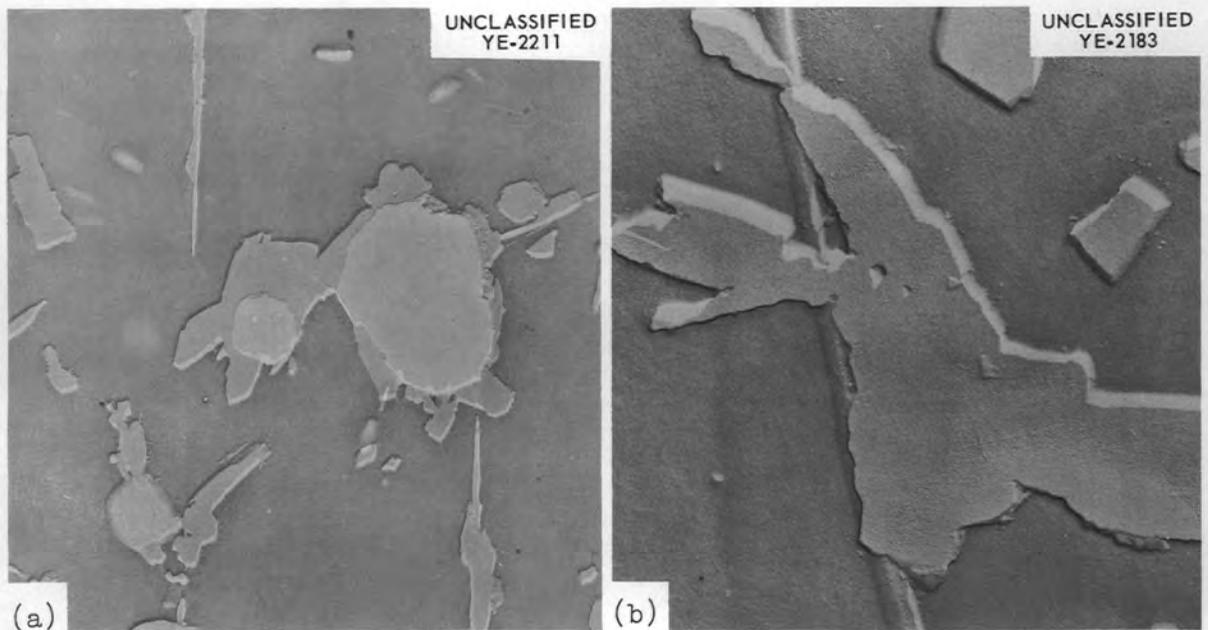


Fig. 16. Hastelloy B Heated 1600 hr at 815°C. The difference in the appearance of the carbide and the precipitate is evident in (a). Note the decrease in the amount of Widmanstätten precipitate.
 (a) 6,000x. (b) 16,400x.

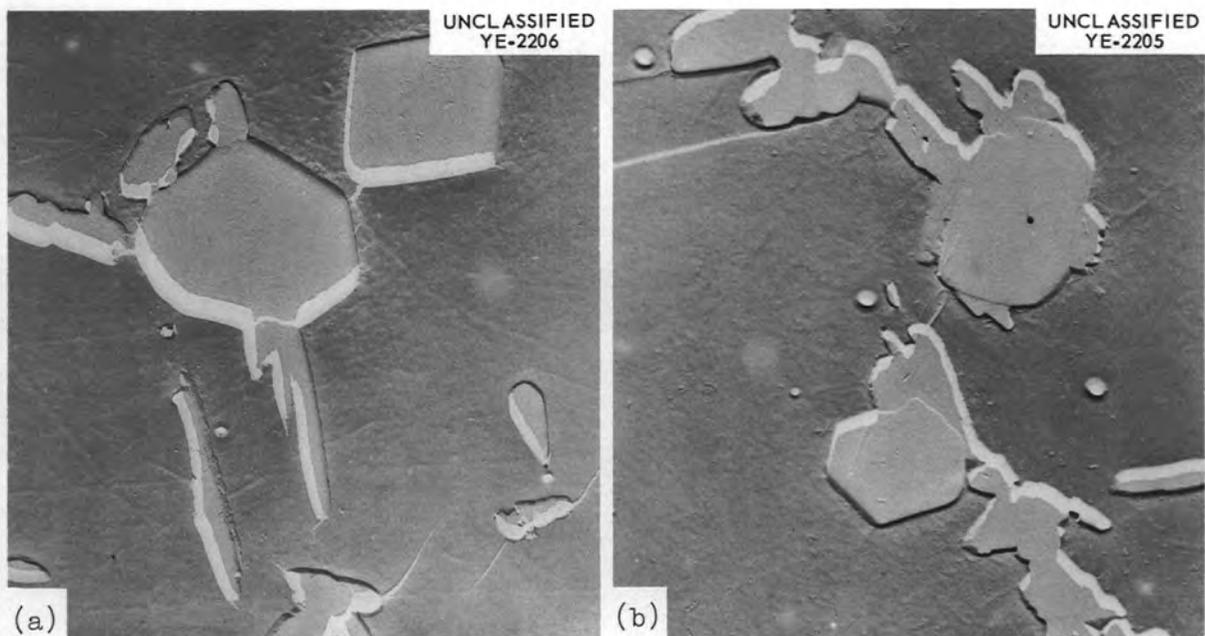


Fig. 17. Hastelloy B Heated 1600 hr at 900°C. Some "massive" carbides are evident. The precipitate may be gamma or delta. Etched with reagent No. 1. 6,000X.

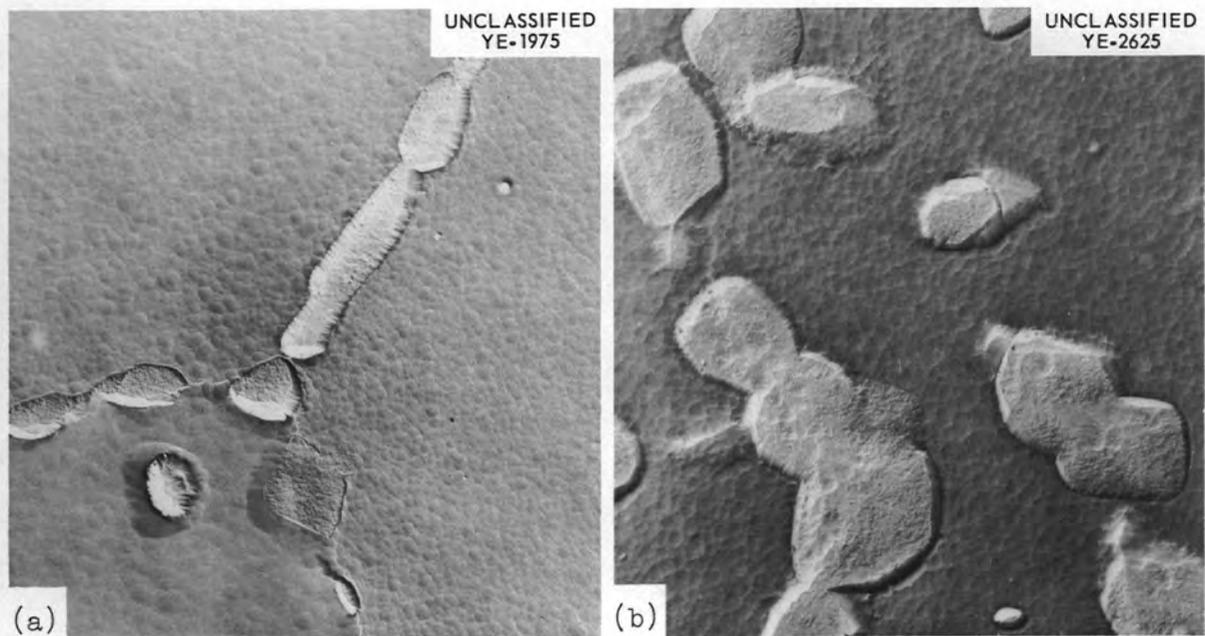


Fig. 18. Hastelloy B Heated 1600 hr at 980°C. The "pocked" surface is an etching artifact. Vacuum cathodic etch. (a) 3,500X. (b) 4,500X.

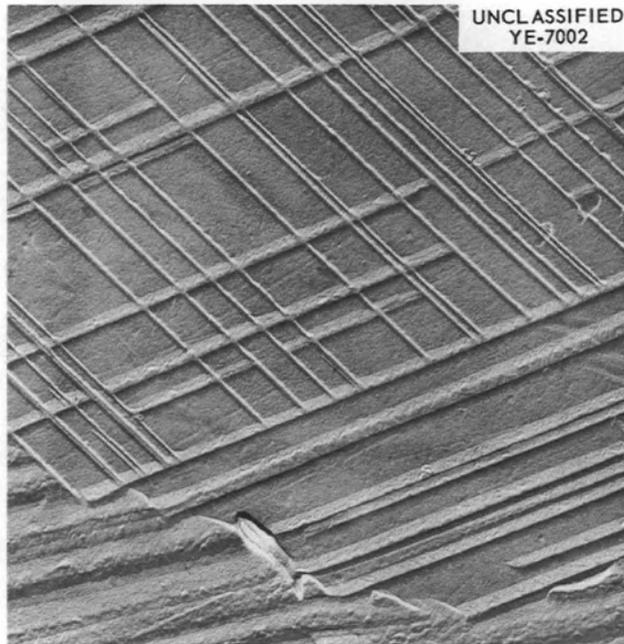


Fig. 19. Electron Micrograph of a Binary Ni-31 wt % Mo Alloy Aged 150 hr at 865°C. 3,600x.

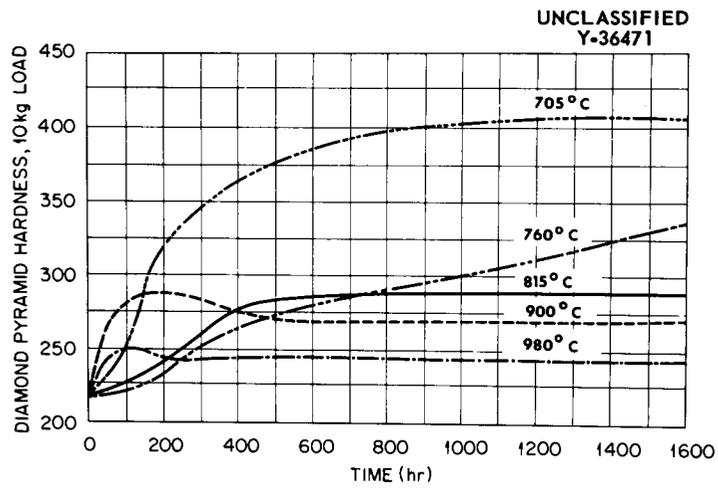


Fig. 20. Hardness Curves for Hastelloy B at Various Aging Temperatures.

(DPH + 400). From the microstructures and hardness values of the present investigation, this condition would appear to exist after 1600 hr at 705°C.

The double heat treatments to reduce the continuous network of fine platelets (see Fig. 12) were also evaluated by hardness measurements. The specimens that were heat treated 1200 hr at 760, 815, or 900°C and then heated 800 hr at 705°C had hardness values of 368, 351, and 336 DPH, respectively. These hardness values compare with 398 DPH for the specimen heated 800 hr at 705°C. Judging only from the microstructure and hardness, the prior heat treatments at 815 and 900°C would probably promote more satisfactory tensile and impact properties.

CONCLUSIONS

The microstructures and diamond-pyramid hardness values for Hastelloy B were determined for isothermal heat treatments at five temperatures from 705 to 980°C. The most microstructural precipitation and the highest hardness occurred at 705°C. The precipitate was characterized by a continuous network of fine Widmanstätten platelets and the specimen attained a hardness of over DPH 400. Although age hardening was evident at all temperatures, the hardness, the tendency to form Widmanstätten platelets, and the etching rates were observed to diminish with increasing heat treatment temperatures. The microstructure and hardness data produced by duplicate heat treatments on this alloy show that over aging at 900°C substantially retards the age hardening that occurs at 705°C.

Carbides appear as spheroidal particles in wrought Hastelloy B and they appear to be stable at all temperatures of this investigation.

ACKNOWLEDGMENTS

We are indebted to E. L. Long, Jr. for electron microscopy, to C. Hays for the development of a process to electrolytically extract carbides from Hastelloy B, and to R. M. Steele and O. B. Cavin for the x-ray diffraction work. We are particularly grateful to E. E. Stansbury for his many helpful comments and suggestions regarding the structures and phases in these materials.



APPENDIX A



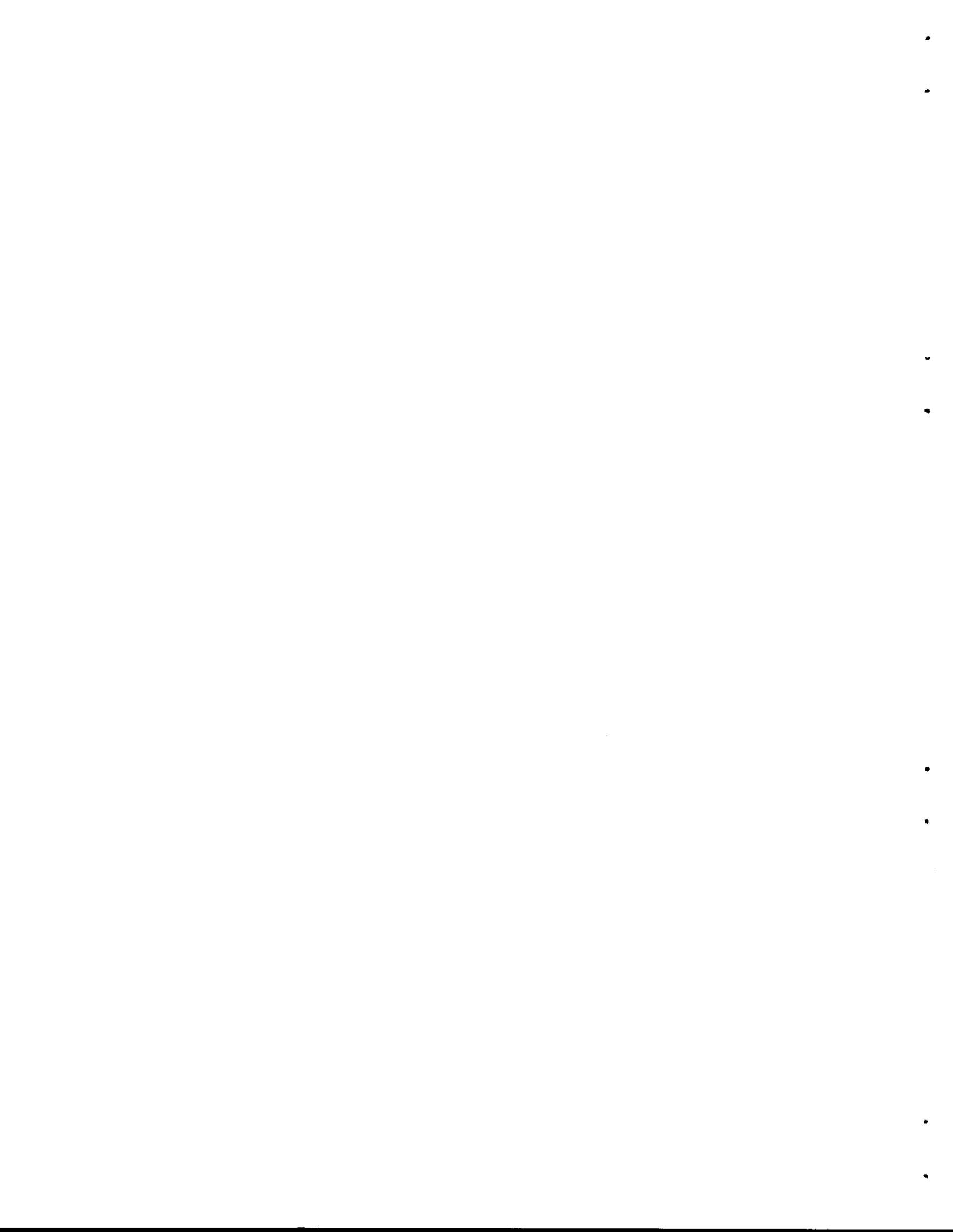
THE ELECTROLYTIC EXTRACTION OF CARBIDES FROM HASTELLOY B

Carbides were extracted from wrought and annealed Hastelloy B, using an electrolytic cell of the type recommended by Blickwede and Cohen.¹⁵ The cell was operated under the following conditions:

Electrolyte	10% HCl solution in water, replenished at a rate of 75% every 12 hr.
Volume	2000 cm ³
Cathode	Copper
Cell current	1.2 amp
Anode current density	0.2 to 0.5 amp/in. ²
Washing media	5% HCl solution in water, distilled water, and alcohol
Drying temperature	165°C max

The cell was operated for 68 hr to yield 0.5752 g of M₆C carbides from 49.67 g of dissolved metal. Upon completion of electrolysis, the electrolyte was decanted from the cell through a fine-porosity fritted-glass filter crucible. A rubber policeman was used to remove loose carbides from the sample. The carbide residue was washed two or three times with 100 cm³ of the 5% HCl solution, using an aspirator to aid in the removal of the solution. The HCl rinses were followed by a distilled water rinse and an alcohol rinse prior to drying.

¹⁵D. J. Blickwede and M. Cohen, Trans. AIME 185, 578 (1949).



APPENDIX B



DIFFRACTION ANALYSIS OF CARBIDES IN HASTELLOY B

The interplaner spacings of the carbides in wrought and annealed Hastelloy B obtained from powder diffraction patterns using chromium K_{α} radiation were:

<u>hkl</u>	<u>d (A)</u>
400	2.71
331	2.48
422	2.22
333, 511	2.09
440	1.92
600	1.81
622	1.635
711, 551	1.525
731, 553	1.42
800	1.36
733	1.33
822, 660	1.283
751, 555	1.256
911, 753	1.194
842	1.187
664	1.16

The interplaner spacings and the relative line intensities are in very good agreement with those for the M_6C carbide reported by Beattie and VerSnyder¹⁶ for high-alloy nickel-base alloys. The lattice parameter of the carbides in Hastelloy B is 10.88 A. This value is slightly lower than the lattice parameter reported by Beattie and VerSnyder, probably due to differences in the types of metal atoms composing the M_6C structure.

¹⁶H. J. Beattie, Jr., and F. L. VerSnyder, Trans. Am. Soc. Metals 45, 397 (1953).

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