Effects of 1600F Exposure on the Microstructure of MAR-M 421 Nickel-Base Alloy

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As-cast MAR-M 421 nickel-base superalloy was given a 2100F (2 hrs) air cool plus 1975F (4 hrs) air cool plus 1400F (16 hrs) air cool heat treatment and exposed at 1600F in both stressed and unstressed conditions for up to 2000 hrs to determine microstructural changes. Exposure in the stressed and unstressed conditions produced a coarsening and agglomeration of the gamma prime and formation of continuous gamma prime envelopes around all carbides and along the grain boundaries. In addition the MC carbide decomposed with a simultaneous precipitation of a chromium rich M23C6 carbide. The application of stress caused an acceleration of the carbide reaction and gamma prime growth, and also produced elongation of the gamma prime.

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INTRODUCTION

The requirements for nickel-base alloys to withstand higher temperatures and stresses for extended periods of time have resulted in greater additions of alloy elements to provide improved strength and stability(1-3). The resulting alloys are too complex to be readily hot worked and so are used in either the as-cast or as-cast plus heat treated condition. To date little information has been reported on the effect of long time elevated temperature exposures on the microstructure of such complex nickel-base superalloys. This investigation reports the effects of long time thermal exposure at 1600F on the microstructure of such a heat treated nickel-base superalloy, MAR-M 421.

EXPERIMENTAL PROCEDURES

A. MATERIAL, HEAT TREATMENT AND EXPOSURE

Oversize test bars were cast from a virgin heat. The chemistry of this heat is given in Table I. All castings were in the form of test specimens 4.75 inches long by 0.50 inches in diameter tapering to 0.30 inches at the center, with a uniform 1/16 in. to 1/8 in. equiaxed grain size.

The heat treatment and long time exposures were conducted in a furnace with a fluctuation of temperature found to be within ±10°F. The first two heat treatment cycles, 2100F(2 Hrs.) and 1975F(4 Hrs.) were accomplished in a retort with continuously circulating dry hydrogen gas. The dewpoint of the exit hydrogen gas was measured at -40°F. The third cycle of the heat treatment, 1400F(16 Hrs.), was performed in static air. All specimens were air-cooled to room temperature after each heat treatment cycle. Long time exposures at 1600F were conducted in static air and air-cooled upon completion of test.

Fully heat treated castings were furnace exposed at 1600F for 100, 250, 500, 750, 1000, 1250, 1500, 1750, and 2000 hours in static air and air-cooled. A single creep specimen with a two inch long by 0.250 inch diameter gage section was machined from each fully heat treated casting. All creep specimens were tested at 1600F and at various stress levels for periods of time from 100 to 2000 hours.

B. MICROSTRUCTURAL EXAMINATION

The microstructures of longitudinal sections of creep-ruptured and of exposed specimens were examined using both optical and electron microscopy. Specimens were prepared for examination by a combination of mechanical polish, electrolytic polish, and chemical immersion etching. Specimens were mounted in epoxy and rough polished on a belt sander followed by hand polishing through 120 to 600 silicon carbide papers. Samples were subsequently polished

on felt wheels impregnated with 6.0 and 1/4 micron diamond paste. Final mechanical preparation was accomplished on a wheel with Linde fine alumina abrasive. The samples were then immersed in Kallings Reagent (5 grams CuCl₂, 100 ml Ethyl alcohol and 100 ml H₂O) from 10 to 30 seconds, until the desired structure was obtained. Occasionally the samples were briefly repolished on Linde fine alumina and re-etched to remove the flow layer and obtain a more desirable microstructure. The final microstructure consists of the carbides and gamma prime in relief above the matrix gamma.

Specimens for electron microscope examination were prepared by electropolishing for 10-20 seconds at 25 volts in a 1 part $\rm H_2SO_4$ - 6 parts methanol solution, rinsed in acetone, and etched in Michigan B (29 parts CuCl₂, 36 parts acetic acid, 23 parts HCl, 5 parts $\rm H_2SO_4$, 7 parts Chromic acid) reagent for 5-10 seconds at 2-3 volts. Two stage replicas were then prepared by pressing a 0.001 in. thick, acetone softened, acetate film onto the prepared surface. After drying the acetate film was stripped from the specimen, shadowed with chromium at an angle of 35° and backed with carbon. The replicas were then sectioned and placed on 200 mesh stainless steel disks and placed in acetone to dissolve the acetate tape, producing a chromium-carbon replica.

C. X-RAY DIFFRACTION AND FLUORESCENCE STUDIES

Phase analysis studies were performed on electrolytically extracted residues from all samples. Extractions were accomplished for 2-3 hours at 0.5 amps/sq. cm. and approximately 5 volts in a 10% HCl methanol solution with the specimen as the anode and a platinum sheet as the cathode. Ten grams of tartaric acid per 150 cc of the 10% HCl methanol solution was added to prevent the formation of tungstic acid.

After completion of the process, the solution was centrifuged to ensure that all extracted particles were retained. On occasion an additional extraction was performed in which the solution was allowed to stand for 5-10 minutes after extracting and carefully decanted into another beaker to separate the fine and coarse particles. The solution was then centrifuged to settle the fine particles to the bottom of the beaker. Since the extraction solution dissolves both the matrix gamma and precipitated gamma prime, the resultant residues are carbides.

The extracted residues were mounted on glass slides and subsequently examined. Identification of phases was accomplished by X-ray diffraction using published and unpublished data on nickelbase superalloys. A chemical analysis of selected residues was also performed by means of a vacuum X-ray spectrograph.

RESULTS

The as-cast and cast plus heat treated microstructures of MAR-M 1 21 have been determined previously (1 4). The following results were obtained when heat treated MAR-M 1 421 was exposed at 1600F.

A. MICROSTRUCTURE

The microstructural results may be conveniently divided into two groups (stressed and unstressed) and into several sections (based) on exposure time).

Heat Treated and Unstressed Isothermal Exposure

100 Hrs.

A post heat treatment exposure at 1600F for 100 hours produced significant changes in the microstructure of MAR-M 421.

Formation of additional gamma prime along the grain boundaries was noticed, producing a continuous film about 0.5 to 1.5 microns wide (Figures 1-2). The matrix gamma prime was not significantly affected and remained about 0.3 to 0.6 microns in size (Figure 2). The fine gamma prime particles characteristic of heat treated MAR-M 421 (4) were not present in the microstructure and apparently coalesced during the aging cycle to form larger particles.

In addition to the existing MC carbide phase $(\frac{1}{4})$, a new carbide phase identified as M23C6, consisting of irregularly shaped particles, was observed in the microstructure located preferentially along the grain boundaries and around the MC carbides. Small amounts were occasionally seen in the matrix (Figures 1-2).

100 Hrs. to 500 Hrs.

The continuous gamma prime grain boundary film widened to 0.7 to 1.7 microns (Figures 3-4). The large gamma prime cubes coarsened to 0.6 to 0.8 microns in size while the fine gamma prime precipitate coarsened to 0.1 to 0.2 microns (Figure 3).

Both plate and irregular shaped $M_{23}C_6$ carbides were observed in the microstructure located predominantly in the grain boundaries (Figure 3) and around the larger smooth MC carbides (Figure 4). Smaller amounts of the $M_{23}C_6$ carbide were also found in the matrix (Figure 3).

500 Hrs. to 1250 Hrs.

The continuous grain boundary film showed a slight coarsening to 1 to 2 microns in width (Figure 5). The fine matrix gamma prime coarsened to about 0.1 to 0.3 microns in size while the larger gamma prime cubes were about 0.6 to 0.9 microns in size.

Small $M_{23}C_6$ carbide particles were located along the grain boundaries and surrounding the larger carbides (Figure 5) and were also randomly dispersed throughout the matrix. Both the MC and $M_{23}C_6$ carbides were surrounded by a gamma prime envelope.

1250 Hrs. to 2000 Hrs.

The gamma prime grain boundary film widened to 1 to 3 microns, while the fine and coarse gamma prime particles also showed a slight coarsening effect. The fine gamma prime particles were about 0.2 to 0.3 microns in size while the larger gamma prime cubes were about 0.6 to 1.0 microns in size after 2000 hours at 1600F (Figure 6).

Both plate and irregular shaped $M_{23}C_6$ carbide particles were present along the grain boundaries, surrounding the large MC carbides, and randomly dispersed throughout the matrix.

Heat Treated Plus Creep Tested at 1600F

Heat treated creep specimens were tested under various stresses at 1600F (see Table II) and the microstructures were examined after 89.0, 765.7, 1300.0, 1500.0, and 2000.0 hours.

1600F for 89.0 Hrs.

Exposure of heat treated MAR-M 421 at 1600F under stress for 89.0 hours after heat treatment produced a slight coarsening of the cubical matrix gamma prime (0.4 to 0.7 microns) and the formation of a secondary fine gamma prime approximately 0.1 micron in diameter (Figures 7-8). A gamma prime film also began to form around the primary MC carbide and along the grain boundaries (Figure 8).

A new irregularly shaped particle, identified as an M23C6 carbide, was also observed to form predominantly along the grain boundaries and around MC carbides. This carbide was also noticed in isolated areas of the matrix.

1600F for 765.7 Hrs.

Exposure under stress at 1600F for 765.7 hours after heat treatment produced additional changes in the microstructure. The matrix gamma prime coarsened to 0.4 to 0.9 microns and occasional elongated gamma prime particles were noted (Figure 9). Occasional fine matrix gamma prime particles were observed that were approximately 0.1-0.2 microns in diameter. A continuous grain boundary film about 0.3 to 1.0 microns wide had also formed.

The irregularly shaped M23C6 carbide was noticed in the grain boundaries, around MC carbides, and in isolated areas of the matrix. The M23C6 was also observed in a plate configuration as well as an irregularly shaped particle, both of which were surrounded by a gamma prime envelope.

1600F for 1300 Hrs.

Exposure under stress at 1600F for 1300 hours after heat treatment produced further elongation of the matrix gamma prime, with particles 0.5 microns wide and up to 2.0 microns long observed (Figure 10). A few fine matrix gamma prime particles about 0.2 microns in diameter were also observed.

A gamma prime envelope was observed along the grain boundaries and surrounding the primary MC and M23C6 carbides. The primary MC carbides continued to dissociate forming $M_{23}C_6$ carbides.

1600F for 1500 Hrs.

Continued exposure at 1600F under stress for 1500 hours after heat treatment produced more elongation of the matrix gamma prime with particles measuring 0.7 to 2.5 microns long and 0.5 to 0.8 microns wide (Figures 11-12). Occasional finer gamma prime particles were observed in the matrix which were about 0.2 microns in diameter. Occasional agglomerated gamma prime particles approximately one to two microns in size were observed in localized areas of the microstructure (Figure 12).

A gamma prime film was observed along the grain boundaries and around the MC and M23C6 carbides. Irregularly shaped and some plate shaped M23C6 carbides also became more predominant at grain boundaries, around MC carbides, and at localized sites in the matrix.

1600F for 2000 Hrs.

Exposure under stress at 1600F for 2000 hours after heat treatment produced a more severe elongation and also agglomeration of the matrix gamma prime. Matrix elongated gamma prime particles were 0.2-0.5 microns wide and up to 3.0 microns in length (Figures 13-14). Agglomerated gamma prime particles, most of which became elongated, were approximately 1.0 micron wide and varied from 1.0 to 3.0 microns in length. Continuous gamma prime envelopes were present at the grain boundaries and surrounding both the MC and M23C6 carbides. The M23C6 carbides were present in a plate phase but usually as irregularly shaped particles which were located in the grain boundaries, surrounding the primary MC carbides, and also at localized sites in the matrix.

B. PHASE IDENTIFICATION AND ANALYSIS

Carbides were extracted from each exposed sample. X-ray diffraction and fluorescence were used for identification of the extracted residues and results are given in Tables III and IV).

Unstressed Exposure

Isothermal annealing at 1600F for up to 2000 hours resulted in the formation of a new phase, a face centered cubic $M_{23}C_6$ carbide. The $M_{23}C_6$ carbide, as well as the MC carbide, was identified in all specimens aged at 1600F.

The most significant trend was the steady decrease in MC carbide with a corresponding increase in M23C6 carbide which was still continuing after 2000 hours (Figure 15). X-ray fluorescence indicated the MC carbide was rich in columbium with small amounts of molybdenum, zirconium, and tungsten substituting for the columbium. The M23C6 carbide was found to be rich in chromium with small amounts of tungsten, nickel, and molybdenum substituting for the chromium (Table IV). No significant changes in chemical content were noted for either carbide during the 1600F aging exposures. The lattice parameter was 4.39Å for the MC carbide and 10.69Å for the M23C6 carbide neither of which were altered by the 1600F exposures (Table IV).

Stress Exposure

Samples exposed under stress at 1600F for up to 2000 hours produced the same phases, face centered cubic MC and M23C6 carbides, as in the annealed samples (Table III). The lattice constants of the MC and M23C6 carbides remained constant out to 2000 hours and were the same as the heat treated and annealed samples.

A decrease in MC with a corresponding increase in $M_{23}C_6$ was found out to 2000 hours. The major difference between the annealed and stress exposed samples is shown in Figure 15. The $M_{23}C_6/MC$ ratio for the stress exposed samples increased at a faster rate than the annealed samples indicating the application of stress increased the decomposition of MC and formation of $M_{23}C_6$.

DISCUSSION

The microstructure of cast plus heat treated MAR-M 421 is typical of nickel-base superalloys of 15 pct. chromium content which are hardened by additions of aluminum and titanium(4). A cubical (blocky) matrix gamma prime with fine secondary gamma prime and only MC carbides would have been predictable from the heat treatment. The resultant behavior during 1600F exposure stems from the temperature-time effects on the gamma, gamma prime and MC phases.

A. EFFECTS OF ISOTHERMAL EXPOSURE AT 1600F ON MICROSTRUCTURE

During isothermal exposure at 1600F, in the unstressed condition, the primary MC carbide, which showed no appreciable changes during the initial heat treatment, decomposed to form M23C6 carbide. The reaction continued out to 2000 hours as noted by the increasing X-ray diffraction intensity ratio, IM23C6/IMC, of the extracted residues. The slopes of these curves indicated the reaction was still occurring after 2000 hours (Figure 15).

While the M23C6 carbides were being formed, the formation of Ni₃(Al,Ti) gamma prime envelopes also took place during the 1600F exposure. The decomposition of the columbium rich MC carbide enriches the surrounding matrix in columbium and carbon which simultaneously reduces the solubility for chromium in the matrix. The reduced solubility for chromium results in the subsequent precipitation of the M23C6 carbide according to the reaction.

MC +
$$\times$$
 \longrightarrow $M_{23}C_6$ + \times or CbC + Ni, Cr, Al, Ti \longrightarrow $Cr_{23}C_6$ + Ni₃(Al, Ti) + Cb

The gamma prime envelope which was usually observed surrounding the M23C6 carbide (predominantly Cr_{23}C_6) might suggest that the carbide may nucleate and grow within the gamma prime particles. However, since significant amounts of neither chromium or carbon are available within the gamma prime phase, this is not probable. It is believed this carbide forms in the matrix and in the grain boundaries at or near the 8-8' interface since the growth of the Ni₃(Al,Ti) depletes the 8-8' interface of nickel, aluminum, and titanium creating a higher concentration of chromium and carbon.

The resultant nucleation and growth of the $\rm Cr_{23}C_6$ carbide in turn depletes the carbide- $\bf x$ matrix interface of chromium and carbon thus creating higher concentrations of nickel, aluminum, and titanium for precipitation of a gamma prime envelope around the $\rm Cr_{23}C_6$ particles.

The imposition of a stress on as-cast plus heat treated samples during 1600F exposure generally accelerated the reactions observed during unstressed exposure. The MC decomposition and M23C6 nucleation and growth was found to proceed at a higher rate than in the unstressed samples as indicated by the Mo2C6/MC intensity ratio in Figure 15. In addition, the application of stress caused elongation of the gamma prime particles after 765.7 hours and, after approximately 1500 hours, resulted in the formation of agglomerated gamma prime particles which were approximately twice the size of the cubical matrix gamma prime present in the heat treated alloy. Agglomerated gamma prime particles were not observed in the annealed samples after 2000 hours; however, unstressed exposure at 1600F for 3000 hours did cause agglomerated gamma prime particles to form in the microstructure (Figure 16). It is interesting to note that agglomerated gamma prime particles required twice the time to form in the unstressed samples (3000 hours) compared to the stressed samples (1500 hours). This effect has been noted previously (1) and indicates the acceleration of the gamma prime growth due to the application of stress.

B. THE STABILITY OF SUPERALLOYS

MAR-M 421 appears to be conventional in its microstructural behavior. However, sigma phase (5,6) and M_CC carbides form in many of the higher strength wrought and cast nickel-base superalloys, but not in MAR-M 421. General empirical rules have been derived to delineate the occurrence of sigma (8,9) and M_CC(10) instabilities. For example, molybdenum content plus one-half the tungsten (wt. pct.) content must exceed 6 wt. pct. for M_CC to form (10). MAR-M 421 contains less than 6 adjusted wt. pct. molybdenum plus tungsten and thus M_CC should not be formed. Even if the 6 wt. pct. figure had been attained, the exposure temperature (1600F) was somewhat below the accepted range (1700-1900F) for M_CC formation.

Chromium, cobalt, nickel and molybdenum are found in most nickelbase superalloy sigma phases. Prior to formation of the sigma phase, these elements are partitioned in complex fashion amongst existing phases. Furthermore, some of these elements may be grossly segregated during the solidification process. Despite these problems, the techniques (8,9) for the prediction of sigma occurrence are of some value in assessing the relative sigma forming tendency of an alloy. A calculation of the sigma forming tendency (electron vacancy number-N $_{\rm V}$) for MAR-M 421 gave an N $_{\rm V}$ value of 2.25. Values of N $_{\rm V}$ > 2.45-2.50 are required for sigma to form. Thus no sigma would be expected in MAR-M 421, a fact which was confirmed by the experimental results.

CONCLUSIONS

Conclusions based on the results of this investigation are as follows:

- 1. Exposure at 1600F in both stressed and unstressed conditions of as-cast plus fully heat treated MAR-M 421 resulted in the decomposition of the MC carbide and simultaneous precipitation of a chromium rich M_{23} C6 carbide with $a_0 = 10.69$ Å, a coarsening and eventual agglomeration of the gamma prime phase, and subsequent nucleation and growth of continuous gamma prime envelopes around all carbides and along the grain boundaries.
- 2. The application of stress caused an acceleration of the above microstructural changes and, in addition, produced an elongation of the gamma prime particles.
- 3. Exposures at 1600F do not alter the chemistry or lattice parameter of either the MC or $M_{23}C_6$ carbide in MAR-M 421.
- 4. Sigma phase and M₆C carbides do not form in MAR-M 421 exposed at 1600F.

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TABLE I

COMPOSITION OF MAR-M 421 ALLOY TESTED

Element	Wt. Pct.	Element	Wt. Pct.			
C	0.18	W	3•33			
Si	0.04	Ti	1.64			
Min	0.10	Al	4.52			
Co	10.78	В	0.019			
Cr	14.96	Zr	0.04			
Fe	0.21	Cb	2.32			
Мо	1.82	Ni	Balance			

TABLE II

CREEP-RUPTURE TESTS ON HEAT TREATED AND MACHINED SPECIMENS

Temperature	Stress	Time in Test					
1600°F	45 ksi	89.0 hours					
1600	35	396.8					
1600	32	765•7					
1600	30	1300.0 (discontinued)					
1600	29	1500.0 (discontinued)					
1600	28	2000.0 (discontinued)					

TABLE III

X-RAY DIFFRACTION DATA ON EXTRACTED RESIDUES

Condition	Type Carbide	Lattice . Parameter Range (A)
Heat Treated plus Exposed 1600F (100 hrs.)	мс ^м 23 ^С 6	4.39 10.66-10.75
1600F (250 hrs.)	MC ^M 23 ^C 6	4.39 10.66 - 10.75
1600F (500 hrs.)	мс ^м 23 ^с 6	4.38-4.39 10.66-10.72
1600F (750 hrs.)	мс ^М 23 ^С 6	4.39 10.66-10.72
1600F (1000 hrs.)	MC ^M 23 ^C 6	4.39 10.66-10.72
1600F (1250 hrs.)	мс ^М 23 ^С 6	4.39 10.66-10.72
1600F (1500 hrs.)	мс ^м 23 ^с 6	4.39 10.66-10.72
1600F (1750 hrs.)	мс м ₂ 3с ₆	4.39 10.66-10.72
1600F (2000 hrs.)	мс ^М 23 ^С 6	4.39 10.66-10.72
Heat Treated plus Stress Exposed 1600F (89.0 hrs.)	MC M ₂₃ C ₆	4.39 10.66-10.75
1600F (396.8 hrs.)	MC M ₂₃ C ₆	4.39 10.66-10.72
1600F (765.7 hrs.)	MC M ₂₃ C ₆	4.38-4.39 10.66-10.72
1600F (1300 hrs.)	мс м 23°6	4.38-4.39 10.66 - 10.72
1600F (1500 hrs.)	мс ^М 23 ^С 6	4.39 10.66-10.72
1600F (2000 hrs.)	мс ^м 23 ^С 6	4.39 10.66-10.72

TABLE IV

X-RAY CHEMICAL ANALYSIS OF EXTRACTED RESIDUES

	Residu e Type		Normalized Relative Peak Intensities							
Condition	Size	Carbide	Cb	W	Ti	Мо	Cr	Zr	<u>Ni</u>	Co
Heat Treated and Exposed:										
1600F (100 hrs.)	coarse	MC	100	2.1	0.9	8.5	-	1.8	0.4	-
1600F (100 hrs.)	fine	M ₂₃ C ₆		19	0.8	9.2	100	-	11.2	2.0
1600F (2000 hrs.)	coarse	MC	100	1.7	-	7.5		2.5		
1600F (2000 hrs.)	fine	M ₂₃ C ₆	-	26	1.0	9.0	100	-	10.5	3.0

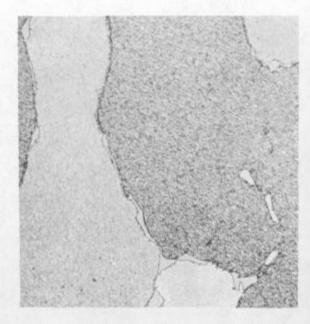


Fig. 1. Structure After 100 hrs Exposure Showing Formation of Gamma Prime in Grain Boundaries. Mag. 500X. Etchant-Kallings Reagent.

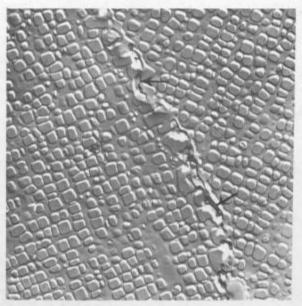


Fig. 2. Electron Microstructure After 100 hrs Exposure Showing Gamma Prime and M23C6 (arrows) in Grain Boundary. Mag. 4250X.

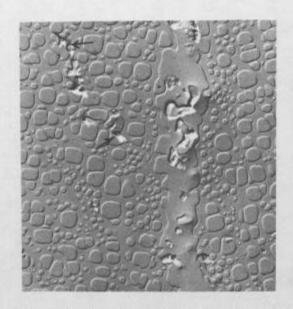


Fig. 3. Electron Microstructure After 500 hrs Exposure Note Coarsened Matrix Camma Prime, M23C6 (arrows) in Matrix, and Widened Grain Boundaries. Mag. 5000X.

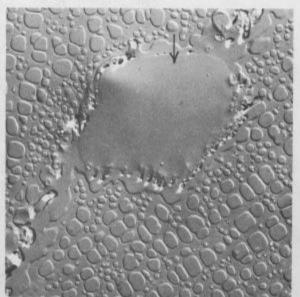


Fig. 4. Electron Microstructure After 500 hrs Exposure Showing M23C6 in Grain Boundary and Around Larger MC Carbide (arrow). Mag. 5000X.



Fig. 5. Electron Microstructure After 1250 hrs Exposure Showing Continued Coarsening of the Gamma Prime Grain Boundary Film. Mag. 4100X.

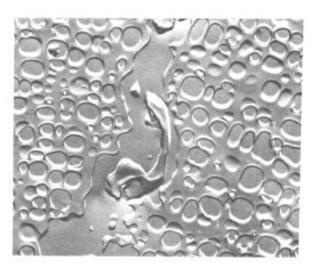


Fig. 6. Electron Microstructure After 2000 hrs Exposure. Mag. 4500X.

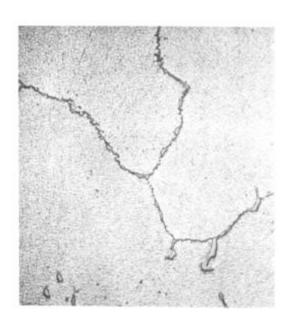
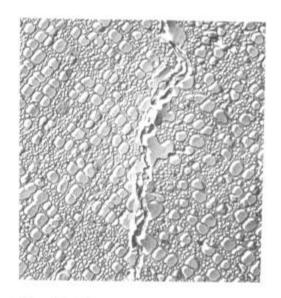


Fig. 7. Structure After 89 hrs Stress Fig. 8. Electron Microstructure Exposure Showing Formation of Gamma Prime in Grain Boundaries. Mag. 500X. Etchant-Kallings Reagent.



After 89 hrs Stress Exposure Showing Gamma Prime and M23C6 in Grain Boundary. Mag. 4500X.

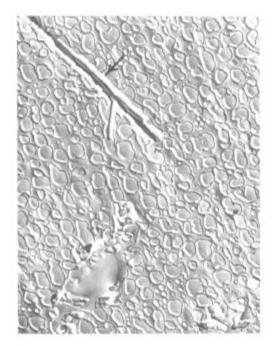


Fig. 9. Electron Microstructure After 766 hrs Stress Exposure Showing Occasional Elongated Gamma Prime Particles and Platelike M23C6 (arrow). Mag. 4500X.



Fig. 10. Electron Microstructure After 1300 hrs Stress Exposure Showing Elongated Gamma Prime Particles and Coarsened Gamma Prime Grain Boundary Film. Mag. 4500X.



Fig. 11. Electron Microstructure After 1500 hrs Stress Exposure Showing Elongated Gamma Prime, M23C6 with Gamma Prime Envelope (arrow) and MC with Peripheral M23C6 and Gamma Prime Envelope (lower right corner). Mag. 4250X.

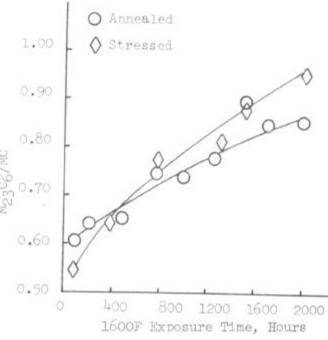


Fig. 15. Ratio of M23C6/MC X-Ray Diffraction Intensity vs. 1600F Exposure Time.



Fig. 12. Electron Microstructure After 1500 hrs Stress Exposure Showing Elongated and Agglomerated Gamma Prime. Mag. 4250X.



Fig. 13. Electron Microstructure After 2000 hrs Stress Exposure Showing Plate and Irregular Shaped M23C6 plus Gamma Prime Envelope Originating from Prior MC Carbide Area. Mag. 4250X.

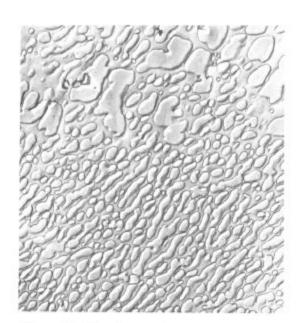


Fig. 14.Electron Microstructure After 2000 hrs Stress Exposure Showing Both Elongated and Agglomerated Gamma Prime Particles. Mag. 4250X.

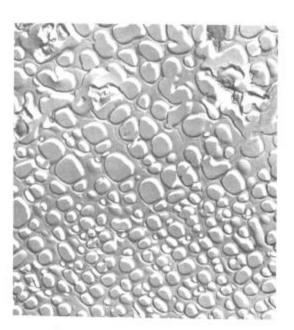


Fig. 16. Electron Microstructure After 3000 hrs Unstressed Exposure. Note Agglomerated Gamma Prime Particles. Mag. 3000X.