# THE EFFECT OF THERMOMECHANICAL HISTORY ON THE STABILITY OF ALLOY 718

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## Abstract

Previous studies showed that Ni<sub>3</sub>Cb precipitation behavior appears to follow a predictable pattern with increased strain in the precipitation range for this phase. Additional investigation was initiated in order to further the understanding of Ni<sub>2</sub>Cb precipitation under similar strain conditions and to develop some information on the effects of such conditions on the stability of gamma prime and the formation of sigma or other phases often considered detrimental to performance. Samples from a production heat were treated above the Ni<sub>3</sub>Cb precipitation range and subsequently strained between 0% RA and 35% RA at 1500°F. Light and electron microscopy and x-ray diffraction studies were performed on samples in the as-strained condition; after a 1750°F gamma prime solution treatment; after a 1750°F treatment followed by a double aging, gamma prime precipitation treatment and after a fully aged plus 1250°F-500 hour exposure. It was found that the quantity of Ni<sub>3</sub>Cb which precipitated during the 1750°F treatment step increases markedly with the degree of strain, that gamma prime stability is not significantly affected by strain or the quantity of Ni<sub>2</sub>Cb precipitate, and that sigma and similar phases do not form during prolonged exposure at the upper end of the useful temperature range for this alloy, even at the highest strain level investigated.

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#### INTRODUCTION:

In a previous paper $^{(1)}$ , an attempt was made to ascertain certain specific effects of Ni<sub>3</sub>Cb precipitation as a function of prior processing history. This preliminary work showed that precipitation behavior seems to follow a predictable pattern with increased time or reduction in the precipitation range. A definite variation in the amount of Ni<sub>3</sub>Cb precipitation proceeded as a function of reduction of area at 1500°F (without fracture) and the present work was initiated for further interpretation of these effects. Actually, there is relatively little known of the specific effects of NiaCb phase on the high temperature properties of Alloy 718. As a generality, Ni<sub>3</sub>Cb phase is not thought to contribute to strengthening and is typically observed in overaged structures (2). On the other hand, small amounts of grain boundary Ni<sub>3</sub>Cb have been observed to improve notch ductility in the  $1200^{\circ}\text{F}$  -  $1300^{\circ}\text{F}$  testing range (3). In the previously referenced paper (1), an initial effect of strain was first shown to decrease the amount of Ni<sub>3</sub>Cb precipitation with early small amounts of strain (10-30%) with increasing Ni<sub>3</sub>Cb precipitation on increasing strain up to about 57% reduction of area. Furthermore, there was some indication of preferred precipitation at specific sites (i.e., grain boundary, twin boundaries and areas containing relatively large numbers of carbides) and it was thought that perhaps a preferred order of precipitation existed as a function of strain.

Wide variations in chemistries and thermomechanical histories have shown a large variety of phases existing in Alloy 718(4). The phases observed in the as-cast structure are Laves,  $\rm M_6C$ , CbC, TiN and Ti\_2SC. The relative amounts and distribution of these phases are altered by the thermomechanical and overall processing history involved but generally these phases exist above  $1800^{\rm o}F$ .

High temperature exposures of 100 hours or less in the  $1500^{\circ}$ F to  $1800^{\circ}$ F range result in the presence of Ni<sub>3</sub>Cb with morphologies that vary from plates to spheroidal particles. With longer time exposures Ni<sub>3</sub>Cb can also form at lower temperatures.

The gamma prime phase, primarily responsible for strengthening, is found in the temperature range of  $1100^{\circ}\text{F}$  -  $1500^{\circ}\text{F}$ . However, gamma prime overages rapidly at high temperatures and a transition body centered tetragonal Ni<sub>X</sub>Cb forms (5). After more prolonged aging this transitional phase then converts to the Ni<sub>3</sub>Cb phase.

With extremely long exposures (thousands of hours) at  $1200^{\circ}$ F to  $1300^{\circ}$ F, the formation of Laves, Sigma and "J" phase is experienced (4). The "J" phase is similar to alpha chromium and has a body centered cubic structure. The influence and interaction of these structures is not fully indicated except that their presence is considered undesirable.

For high temperature applications, Alloy 718 is typically heat treated as follows:

1. 1700°F to 1800°F - 1 to 4 hours to solution gamma prime.

2. Double age at 1325°F and 1150°F for a combined time of 18 to 20 hours to age, precipitate gamma prime.

The above treatment has been found to develop the best combination of tensile and stress rupture properties at elevated temperatures (3).

The present investigation was instituted in order to gain a further advanced understanding of  $\mathrm{Ni_3Cb}$  phase precipitation and shed more light on some of the findings of the original study (1). As in the first paper, a thermomechanical program was instituted using a Duffers Gleeble machine which is capable of controlling heating and strain rates in such a manner as to simulate cycles that the forger experiences during processing and to study the  $\mathrm{Ni_3Cb}$  phase precipitation after various steps in these cycles. A further objective of the present work is a cursory examination of the possible effects of various amounts of  $\mathrm{Ni_3Cb}$  phase on the long time structural stability characteristics of 718 and to separate the specific effects of  $\mathrm{Ni_3Cb}$  on microstructure such as fine gamma prime and a previously observed  $\mathrm{Ni_3Cb}$  phase.

#### PROCEDURE:

The Alloy 718 material used in this study was from hot rolled bar from a commercial vacuum induction plus vacuum arc remelted heat. The samples were initially solution treated 4 hours at 1950°F followed by a water quench to approximate the structural conditions that exist prior to a finish forging operation. Following this the samples were reheated on a Gleeble to 1950°F, held one minute, allowed to free cool to 1500°F, held 5 minutes, and individual samples were then strained the appropriate amounts followed by a free cool to room temperature. The final samples represented strains of 0 to 35% reduction in area thus simulating the structural and strain conditions existing after finish forging. The detailed Gleeble thermomechanical procedure can be found in Appendix A. Figure 1 is a time-temperature portrayal of the thermomechanical treatments used and Table I shows the treatment of individual samples.

Samples with each reduction were then given the normal one hour,  $1750^{\rm O}{\rm F}$  gamma prime solution step followed by the regular  $1325^{\rm O}{\rm F}$  +  $1150^{\rm O}{\rm F}$  gamma prime aging treatment. Another set of samples was held for 500 hours at  $1250^{\rm O}{\rm F}$  to study the effects of strain on the overaging of gamma prime and the possible formation of the transition phase  ${\rm Ni_xCb}$ . The samples thus prepared were studied after each stage of heat treatment and exposure by light and electron microscopy as well as x-ray diffraction of extracted residues in accordance with the detailed procedures outlined in Appendix A.

## RESULTS:

# A. X-Ray Diffraction

The results of x-ray diffraction studies are given in Table

II. The 4 hour heat treatment at 1950°F has resulted in considerable CbC, TiN, and some Ti<sub>2</sub>SC particles being present. When this material is then treated at 1750°F for one hour, a precipitation of orthorhombic Ni<sub>3</sub>Cb occurs. Subsequent aging at 1325°F and 1150°F resulted in no apparent additional Ni<sub>3</sub>Cb formation. The 10% HCl-methanol electrolyte dissolves the gamma prime and thus this phase is not detected by x-ray analysis.

On the other hand, x-ray diffraction studies of the gamma prime residue extracted from the 35% reduction of area, fully heat treated sample (with  $HCl-H_2O$  electrolyte) shows that gamma prime is present and in the face-centered cubic form. A broadening of the gamma prime lines indicates a very fine particle size. During this type of extraction, only a portion of the  $Ni_3Cb$  plates are extracted and, as a consequence, this solution is not suited for a study of this phase.

If a strain is imposed on the material prior to the solutioning 1750°F step, the amount of Ni<sub>3</sub>Cb increases with increasing strain levels. The Ni<sub>3</sub>Cb is mostly contained in the "small" fraction of residue (see detailed procedure - Appendix A) and shows a fairly direct correlation to the percent reduction of area.

Extractions of the samples exposed for 500 hours at  $1250^{\rm O}{\rm F}$  gave the same increasing trend of more Ni<sub>3</sub>Cb with higher strain levels. This trend was expected as no Ni<sub>3</sub>Cb should form at  $1250^{\rm O}{\rm F}$  for 500 hours.

The extraction of the gamma prime in the exposed samples was more easily accomplished since changes can be expected in gamma prime size and composition due to the exposure. The x-ray data showed no new phase developing under the conditions of exposure; however, it is known that exposure under stress may form a transitional phase  $Ni_xCb(5)$ .

### B. Optical Microscopy

Samples were prepared for study by optical microscopy as discussed in Appendix A. Some microstructural observations are as follows.

Specimens with no prior strain (0% reduction in area) showed the presence of a discrete grain and twin boundary precipitate believed to be MC,  $\rm Ni_3Cb$  and/or both. Numerous grain and twin boundaries are free of any precipitation. A similar structure was observed in speciments treated at  $1750^{\rm o}$ F-lhr-AC, fully heat treated and exposed for 500 hours at  $1250^{\rm o}$ F.

When the material is strained 10%, there is an increase in the plate-like  $\rm Ni_3Cb$  precipitation on the twin boundaries and in the matrix near grain boundaries. There are areas that exhibit higher plate densities suggesting pre-existing microchemical segregation still remains in the material even though prior heat treatment should have leveled concentration gradients by diffusion.

Additional straining to the 20% level produces larger amounts of  $Ni_3Cb$  plates on the twin and grain boundaries as well as the usual heavy concentration of  $Ni_3Cb$  in the more solute rich areas. At this strain level, there is evidence that recrystallization is occurring but that the grains are not able to grow because of the impedance provided by the  $Ni_3Cb$  precipitation.

The microstructure of the 35% reduction in area samples showed more of the same type of structural changes seen in the 20% reduction in area sample. There appears to be a great deal more Ni<sub>3</sub>Cb precipitation and more evidence of recrystallization. As the strain levels approach 20% or more, the Ni<sub>3</sub>Cb precipitation on the twins indicates considerable deformation of the twin boundaries. The matrix in recrystallized areas exhibits lower Ni<sub>3</sub>Cb densities than the surrounding unrecrystallized areas.

## C. Electron Microscopy

# 1. Fully Heat Treated Condition

The 0% reduction in area sample shows the presence of Ni<sub>3</sub>Cb plates precipitated near CbC particles, on the ends of twins, and along grain boundaries, Figure 2. In addition to the Ni<sub>3</sub>Cb plates, there is evidence of a round necklace-like precipitate in the grain boundaries which is apparently associated with the CbC phase, Figure 3a. However, a repolish and etch reveals this necklace more clearly but not necessarily at grain boundaries, Figure 3b. The gamma prime phase is very small and is difficult to resolve even at 7500X.

At the 10% reduction in area level, the Ni<sub>3</sub>Cb plate phase is more easily detected in specific areas, more Ni<sub>3</sub>Cb is found in twins, and the necklace structure is still present, Figure 4. At a strain level of 20% reduction in area the quantity of Ni<sub>3</sub>Cb platelets increases and the maximum quantity is noted after 35% reduction in area. Certain areas show heavy Ni<sub>3</sub>Cb, Figure 5a, while others show the nucleation and growth of smaller particles, Figure 5b. Some twin boundaries which are in the heavily recrystallized 35% reduction in area sample show Ni<sub>3</sub>Cb plates growing perpendicular to the twin boundary in a zipperlike fashion, Figure 5c.

The gamma prime size and distribution did not materially change over the strain interval studied. This would seem to indicate the independence of gamma prime precipitation on the amount of Ni<sub>3</sub>Cb phase.

# 2. Exposed 500 Hours at 1250°F Condition

The samples given 500 hours exposure show no change in Ni<sub>3</sub>Cb formation; however, a change in gamma prime is noted. In the 0% reduction in area sample the gamma prime has started to show a fine plate-like structure in addition to the round particles, Figure 6. No depletion or denuding of the grain boundaries is evident in

this or any other sample.

The gamma prime has apparently grown more in the 10% reduction in area and higher strain samples, Figure 7. The twin boundaries show the Ni<sub>3</sub>Cb plates to be still discontinuous. Very little change occurs in the gamma prime at the 35% reduction in area level. The Ni<sub>3</sub>Cb plates are still present and discrete in twin boundaries, Figure 8b, and there is much evidence of recrystallization and lack of grain growth because of Ni<sub>3</sub>Cb plates, Figures 8a, 8b, 8c and 8d.

# DISCUSSION OF RESULTS:

The amount of Ni<sub>3</sub>Cb precipitated at  $1750^{\circ}F$  is dependent of the amount of strain imposed on the material prior to the  $1750^{\circ}F$  heat treatment. While the grain boundaries and twin boundaries nucleate and grow large Ni<sub>3</sub>Cb plates at  $1750^{\circ}F$ , the high strains imposed on the material keep the plate formation discrete in nature.

The Ni3Cb formation without strain is noticed primarily in grain and twin boundaries and near CbC particles which may have partially solutioned during the 1950°F treatment. The addition of strain induces Ni3Cb precipitation in banded areas which may be related to prior chemical segregation produced by solutioning of other CbC particles. The heavy concentration of Ni3Cb in such areas increases with higher strain levels.

The gamma prime reaction did not seem to be influenced greatly by any degree of Ni<sub>3</sub>Cb precipitation or strain level. Apparently, there is enough columbium in the matrix to generate the gamma prime precipitate regardless of the amount of Ni<sub>3</sub>Cb precipitated at 1750°F. There were no signs of depletion or denuding of the gamma prime near the grain boundaries.

The gamma prime does undergo changes in size and morphology during exposures of 500 hours at  $1250^{\circ}\mathrm{F}$ . The size and morphology seems to be affected by strain more after such exposure than in material that was aged but not subsequently exposed. The resultant small plates of gamma prime still showed a face-centered cubic structure. The transition phase  $\mathrm{Ni_{X}Cb}$  previously reported (5) was not detected after 500 hours at  $1250^{\circ}\mathrm{F}$ .

The speed of the gamma prime overaging reaction appears to be controlled more by the stresses superimposed during exposure than by stresses induced prior to exposure. The latter tends to affect Ni $_3$ Cb formation at 1750°F to a greater extent.

The presence of many particles of  $\mathrm{Ni_3Cb}$  tends to inhibit the growth of newly recrystallized grains formed at  $1750^{\mathrm{O}}\mathrm{F}$ . Recrystallization is seen with 20 and 35% strain levels. The use of higher solutioning or heat treatment temperatures would dissolve more  $\mathrm{Ni_3Cb}$  and thus grain growth probably would occur more readily.

The appearance of a necklace of fine particles at grain boundaries and across grains indicates that a film of CbC may have formed initially because of prior processing, the  $1950^{\circ}F$  treatment, and subsequent  $1500^{\circ}F$  treatment (6). CbC film formation would be followed by spheroidization during the  $1750^{\circ}F$  step.

It should be recognized that the precipitation behavior of the Ni<sub>3</sub>Cb and gamma prime may be altered if the material is subjected to thermomechanical histories different from those used in this study. Uniformity of Ni<sub>3</sub>Cb precipitation would be affected by the type and distribution of carbides and other phases present prior to the 1750°F heat treatment.

Although the quantity of  $Ni_3Cb$  generated is influenced by the amount of strain induced, no formation of phases normally considered detrimental to properties was noted even after prolonged exposure near the upper end of the useful temperature range for this alloy. This information along with the previously described TTT behavior of Alloy  $718^{(5)}$  could be useful in designing time-temperature-deformation procedures in finish forging for the purpose of optimizing the strengths and reliability of this important alloy.

#### REFERENCES

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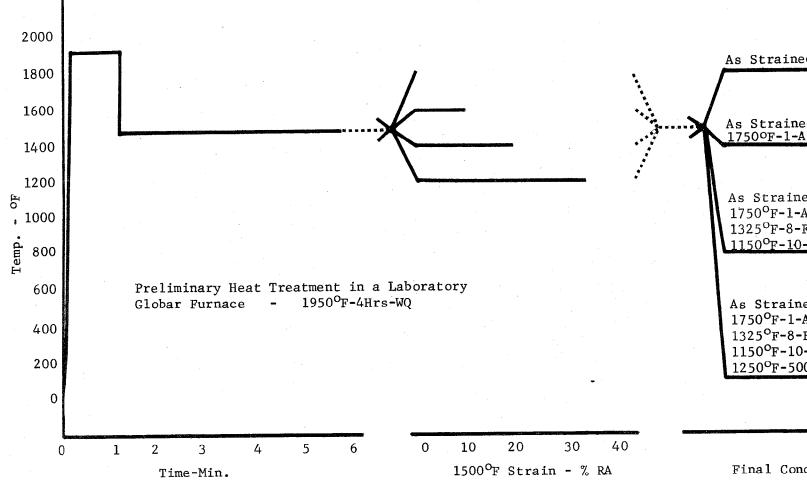
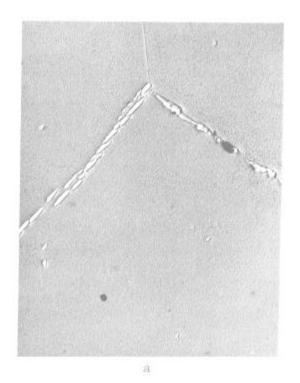
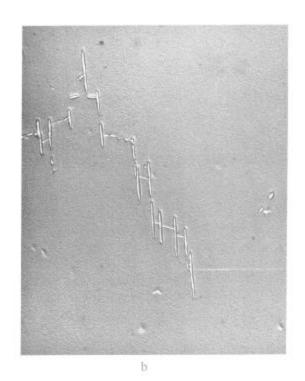


Figure 1 - Thermal, Strain and Heat Treatment Programs Applied to Test Samples





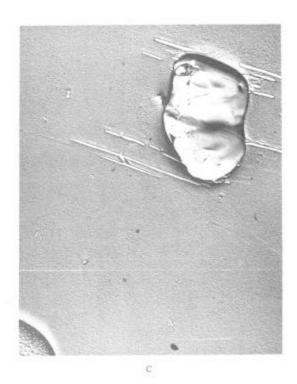


Figure 2 - Ni<sub>3</sub>Cb Precipitation Along Grain Boundaries(a); At Twin Boundaries(b); and At CbC Particles(c). 0% RA + Fully Heat Treated. X3000



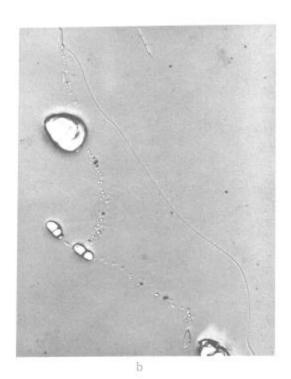


Figure 3 - Necklace-Like Precipitate at Grain Boundaries(a); and Within Grains(b). 0% RA + Fully Heat Treated. X3000



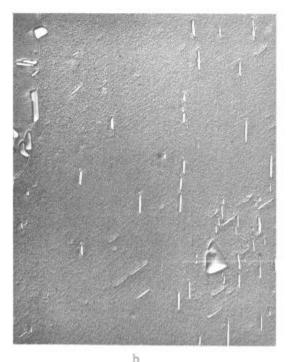




Figure 4 - Ni<sub>3</sub>Cb Precipitate Within Grains and At Grain and Twin Boundaries(a),(b),(c); and Necklace-Like Precipitate at Grain Boundary(c).

10% RA + Fully Heat Treated. X3000





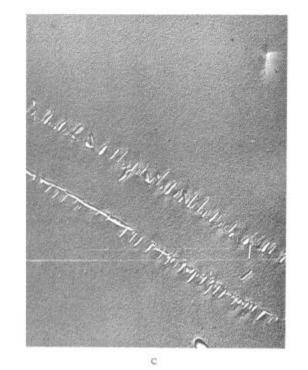


Figure 5 - Variations in  $Ni_3Cb$  Precipitate Density and Particle Size(a)(b); and Zipper-Like Precipitate Form at Twin Boundaries(c). 35% RA + Fully Heat Treated. X3000







Figure 6 - Visible Fine Spheroidal and Plate-Like Precipitate of Gamma Prime; No Visible Gamma Prime Depletion Adjacent to Grain Boundaries.

0% RA + Fully Heat Treated + 1250°F-500 Hours. X3000



b



Figure 7 - Slight Growth of Gamma Prime Precipitate as a Result of Prior Strain.
10% RA + Fully Heat Treated + 1250°F-500 Hours. X3000

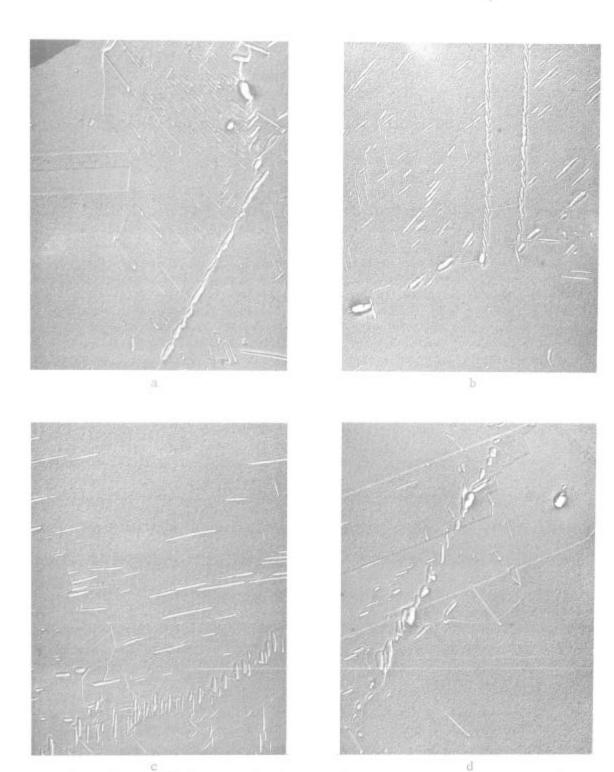


Figure 8 - Slight Growth of Gamma Prime Precipitate as a Result of Prior Strain; Incipient Recrystallization and Grain Growth; The Obstruction of Grain Growth by Ni<sub>3</sub>Cb Precipitate Particles; Reduced Ni<sub>3</sub>Cb Precipitate Density in Recrystallized Areas (a)(b).

35% RA + Fully Heat Treated + 1250°F-500 Hours. X3000

TABLE I - Thermal, Strain and Heat Treatment Programs Applied to Test Samples Studied (1)

Heat Treatment Prior	Thermomechanical Cycle Applied W	With Gleeble Strain - %RA	Heat Treatment After Gleeble Treatment
1950°F-4hrs-WQ	1950°F-1min-Cool to 1500°F-5min	0%	1750°F-1hr-AC
			1750°F-1hr-AC+1325°F-8hrs- Cool to 1150°F-10hrs-AC
n n	<b>n</b>	n	1750°F-1hr-AC+1325°F-8hrs-Cool to 1150°F-10hrs-AC+12
1950°F-4hrs-WQ	1950°F-lmin-Cool to 1500°F-5min	10%	 1750 <sup>O</sup> F-1hr-AC
11	11	11	1750°F-1hr-AC+1325°F-8hrs- Cool to 1150°F-10hrs-AC
<b>11</b>	<b>n</b>	. <b>11</b>	1750°F-1hr-AC+1325°F-8hrs- Cool to 1150°F-10hrs-AC+12 500hrs-AC
1950°F-4hrs-WQ	1950°F-1min-Cool to 1500°F-5min	20%	 1750 <sup>0</sup> F-1hr-AC
ii e	<b>II</b>	ii	1750°F-1hr-AC+1325°F-8hrs- Cool to 1150°F-10hrs-AC
;	en e		1750°F-1hr-AC+1325°F-8hrs- Cool to 1150°F-10hrs-AC+12 -500hrs-AC
1950°F <b>-4</b> hrs-WQ	1950°F-1min-Cool to 1500°F-5min	35%	 1750°F-1hr-AC
н	u	11	1750°F-1hr-AC+1325°F-8hrs- Cool to 1150°F-10hrs-AC
11	H		1750°F-1hr-AC+1325°F-8hrs- Cool to 1150°F-10hrs-AC+12 500hrs-AC

(1) Samples, in Triplicate, Were Prepared for Each Condition Shown

TABLE	II	_	X-Ray Diffraction Data on Extracted	1 Residues

	4	node II nay bilizaction baca	Structures (Intensity) (1)			
	Туре					
Sample	Residue	Sample Condition	MC	TiN	Ni3Cb	T12SC
Extracted w	vith HC1-Methanol	for 2 Hours				
A-Standard	Tota1	1950°F-4hrs-AC	20	5		1
B-Standard	Tota1	1950°F-4hrs-AC+1750-1-AC	25	3½	3	1
C-Standard	Tota1	1950°F-4hrs-AC+FHT(2)	25	3	2	1/2
1	Total	1950°F-4hrs-AC 0% Strain	19	9 .		
5	Tota1	1950°F-4hrs-AC 10% Strain	20	10		1
9	Total	1950°F-4hrs-AC 20% Strain	20	13		12 12 12
13	Total	1950°F-4hrs-AC 35% Strain	20	- 8		$ar{1}_{2}^{-}$
2	Large Fraction	0% Strain+1750°F-1hr-AC	19	3	3	
6	Ħ	10% Strain+1750°F-1hr-AC	19	2	4	
10	11	20% Strain+1750°F-1hr-AC	20	5	2	
14	<b>11</b>	35% Strain+1750°F-1hr-AC	20	3	4	
3	: <b>!!</b>	0% Strain+FHT	17	. 8	1/2	
7	11	10% Strain+FHT	18	6	$\bar{2}$	
11		20% Strain+FHT	- 19	3	5	
15	· 11	35% Strain+FHT	19	2	12	
3	Small Fraction	0% Strain+FHT	. 3		4	· · · ·
7	· 11	10% Strain+FHT	6		13	
11	11	20% Strain+FHT	5		12	
15	11 ,	35% Strain+FHT	6		20	
Extracted w	ith HC1-Methanol	for 4 Hours		•	4	
D-Standard	Tota1	FHT+500hrs@1250 <sup>0</sup> F	20	2	5	, <b></b>
4	Tota1	0% Strain+FHT+500hrs@1250°F	20	4	1	-,-
8	Total	10% Strain+FHT+500hrs@1250°F	19	2	. 8	
12	Tota1	20% Strain+FHT+500hrs@1250°F	19	1	11	
16	Tota1	35% Strain+FHT+500hrs@1250°F	19	1	12	

Table II Cont'd.

			Structures (Intensity) (1)				
Sample	Type Residue	Sample Condition	MC	TiN	Ni <sub>3</sub> Cb	Ti <sub>2</sub> SC	
Extracted wit	h HC1-Water						
15		35% Strain+FHT	1		2		
E-Standard		Std+FHT+500hrs@1250 <sup>o</sup> F	·	,	,		
4		0% Strain+500hrs@1250 <sup>O</sup> F		_ ==	(3)		
8		10% Strain+500hrs@1250 <sup>O</sup> F	1/2		present		
12		20% Strain+500hrs@1250 <sup>O</sup> F	1 2		present		
16		35% Strain+500hrs@1250°F	1		present		

<sup>(1)</sup> Intensity Units of Strongest "d" Line in X-Ray Pattern

<sup>(2)</sup> Full Heat Treatment - 1750°F-1hr-AC+1325°F-8hrs-FC to 1150°F-10hrs-AC

<sup>(3)</sup> Gamma Prime and Ni<sub>3</sub>Cb Strongest "d" Lines are Nearly in Coincidence and Separation is Difficula

# Appendix A

#### DETAILED PROCEDURES

# 1. Material:

Material used for this study was in the form of 5/8" diameter hot rolled bar from a commercial size vacuum induction plus vacuum arc remelted heat. All samples were heat treated  $1950^{\circ}F$  - 4 hours in a laboratory Globar furnace and then water quenched.

# 2. Thermomechanical Treatment:

Samples 4-1/2" long and 0.25" in diameter were machined from the heat treated barstock. The thermomechanical procedure on the Gleeble involved resistance heating at about  $100^{\circ}\text{F/sec.}$  to  $1950^{\circ}\text{F}(1)$ , holding one minute, cooling at about  $1000^{\circ}\text{F/sec.}$  to  $1500^{\circ}\text{F}$  and holding five minutes. At this point samples were:

- a) Cooled at about 1000°F/sec. to room temperature.
- b) Strained about 10%, 20% and 35% (reduction in area) at 1500°F (at a load ram speed of 1"/sec.) and then cooled to room temperature.

The thermal cycle was controlled and recorded through the output of a 0.020" diameter Chromel-Alumel thermocouple that was percussion welded to the surface of the sample in such a way as to assure physical bonding with minimum effect on the structure in the weld area. The amount of strain was varied as desired by using crosshead stops to control the travel of the load ram. With this technique a heat-affected zone approximately 1/2" long was obtained with an approximately 0.2" uniform temperature zone. After treatment on the "Gleeble" some samples were prepared for structural studies,

- a) As thermomechanically treated,
- b) With a post heat treatment of 1750°F-1hr-AC,
- c) With a post heat treatment of 1750°F-1hr-AC+1325°F-8hrs-Furnace Cool to 1150°F-10hrs-AC, and
- d) With a post heat treatment of 1750°F-1hr-AC+1375°F-8hrs-Furnace Cool to 1150°F-10hrs-AC+1250°F-500hrs-AC.

Thus samples, in triplicate, were generated with four different conditions of strain and four different conditions on heat treatment for each strain condition.

<sup>(1)</sup> All temperatures on the Gleeble require a correction factor of +75°F to +100°F due to the type of temperature measurement inherent in the equipment. Any attempts to relate these temperatures to those used for forging or heat treatment should take this into consideration.

# Appendix A Cont'd.

Pg. 2

# 3. Optical Microscopy:

Samples were prepared from the 1/2" long heat-affected zone generated during the thermomechanical cycle. These were ground longitudinally through 600 grit paper and then polished, on wheels, with 6 micron diamond followed by 0.3 micron alumina and finally with 0.05 micron alumina. The polished samples were swab etched with Kallings reagent (50 ml HCl - 50 ml methanol - 2.5 grams CuCl<sub>2</sub>).

## 4. Electron Microscopy:

Samples were cut into about 3/4" length sections with the desired heat-affected zone of 1/2" centered in the section. The samples were ground longitudinally through 600 papers, polished with Linde A abrasive on a wheel, and then electropolished in a  $\rm H_2SO_4$ -methanol solution. The effects of various amounts of strain on the  $\rm Ni_3Cb$  and gamma prime precipitation were studied using the electron microscope. Only the areas located directly opposite the thermocouple point were replicated.

The samples exposed for 500 hours at 1250°F were examined in a similar way as described above. Care was used to study and photograph representative areas.

All samples were replicated with Collodion, shadow cast with chromium and backed with a thin deposit of carbon. Structures were photographed at 1500X.

# 5. X-Ray Diffraction of Extracted Residues:

A second set of samples was cut off at one boundary of the heat-affected zone and the area of interest was ground free of oxides. The sample was then cleaned free of the flow layer by a three minute electrolytic attack in HC1-methanol. The freshly prepared surfaces were then immersed to the desired height in a 10% HC1-methanol electrolyte and the phases were extracted for a period of two hours.

The floating residue was allowed to settle for two hours and then the electrolyte was decanted off into a separate beaker. The fine precipitation in the electrolyte was allowed to settle for 24 hours prior to final decanting and washing. The sample residues thus obtained were labeled "large", two hour settle, and "small", 24 hour settle.

The resultant residues were mounted on a glass slide and x-ray diffraction patterns were obtained with a Philips x-ray unit using Cu radiation, a monochromator and a PHA system.

# Appendix A Cont'd.

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The same procedure was carried out on the 500 hour exposed samples and an additional extraction in 10% HCl-H<sub>2</sub>O was performed in order to study gamma prime changes. The 10% HCl-methanol electrolyte dissolves the gamma prime phases.

In addition to the various strained areas, extractions were carried out on material away from the heat-affected zones for each heat treatment condition. These structural studies served as a check on the material behavior without strain and are designated as A, B, C, D & E Standards.