PRECIPITATION REACTIONS AND STABILITY OF Ni_3Cb IN INCONEL ALLOY 718

W. J. Boesch*
H. B. Canada**

Abstract

Data presented in current literature attribute many desirable properties of Inconel Alloy 718 to sluggish precipitation of the columbium rich intermetallics. Original time-temperature-transformation curves indicate the precipitation of Ni_3Cb initiated in five to ten hours in the temperature range commonly used for solution heat treatment. This presentation shows that Ni_3Cb precipitates in less than 0.2 hours at 1700°F. The chemical composition and structure for Ni_3Cb are presented. The chemical and morphological stability of this phase are discussed. The change from the spheroidal hardening precipitate to the acicular Ni_3Cb is also shown.

The presentation also discusses the composition and stability of the MC carbide and indicates the precipitation of CbC. The precipitation of Ni_xCb and its upper stability limit are discussed.

---

* W. J. Boesch is Senior Research Metallurgist, Special Metals Corporation, New Hartford, New York

** H. B. Canada is Senior Research Chemist, Special Metals Corporation, New Hartford, New York

Introduction:

Precipitation reactions in Inconel Alloy 718 have been described as sluggish. Eiselstein(1) was the first to prepare time-temperature-transformation curves to explain the behavior of the major precipitation reactions. These data were reviewed and the Ni_3Cb, Laves phase and gamma prime transformation curves are reproduced in Figure 1. All specimens used by Eiselstein were aged in an essentially strain-free condition which was produced by a one hour solution heat treatment at 2100°F. As depicted the Ni_3Cb phase has a relatively slow aging response and begins to precipitate in approximately 5 hours at 1650°F. Time-temperature-transformation data presented by Wagner(2) indicate an even slower aging response with initiation of Ni_3Cb precipitation in about 10 hours at 1650°F. According to the Eiselstein curves, Laves phase has a faster aging response and begins to develop in about 50 minutes at 1750°F. If the commonly used one hour solution heat treatment in the temperature range of 1700°F to 1750°F is considered, the early transformation curves predict freedom from Ni_3Cb precipitation. In addition the data predict the formation of small amounts of Laves phase with the same heat treatment. The curves also show the gamma prime reaction to be the fastest with precipitation occurring in about 6 minutes at 1400°F.

Recent metallographic observations(3) are inconsistent with the predictions from the early transformation curves. Various quantities of Ni_3Cb were detected in specimens subjected to heat treatments of less than 5 hours at 1700°F. Therefore, this investigation was initiated to study the aging behavior of Ni_3Cb in essentially strain-free material with the objective of developing a more precise time-temperature-transformation curve which might resolve the observed discrepancies. During the execution of the program, other related reactions were detected and studied. In addition, the stability of various phases during long time elevated temperature exposure was determined.

Experimental Procedure:

Standard commercial 19/32-inch diameter hot rolled bar was used in this investigation. The original ingot was a product of a vacuum induction melting plus vacuum arc remelting technique. The ingot was cogged to reroll stock and then rolled to finished bar at a starting temperature of 2050°F and a finishing temperature of 1850°F. The chemical composition of the product which is listed in Table I is well within the compositional range defined in many Inconel Alloy 718 specifications. To assure that the material used is representative of commercial quality, its properties were compared with the requirements of an arbitrarily selected specification. The tensile data presented in Table I confirm that the material has acceptable mechanical strength. Metallographically examined samples were found free of deleterious banding and segregation.

Upon completion of quality assurance tests, the bar was cut into three inch long heat treatment specimens. All bars were solution heat treated for 4 hours at 1950°F and air cooled to produce an essentially strain-free condition which was verified by x-ray. Thermocouples embedded in the bars indicated a cooling rate of 2.7°F per second. One specimen was retained in the as-solution heat treated condition as a control and the remainder were aged in accordance with the program depicted in Figure 2. All specimens aged for one hour or more were heat treated in air in a conventional laboratory furnace. Specimens aged for less than one hour were heat treated in a salt pot furnace to obtain fast heating rates and thus minimize timing error.

After heat treatment the specimens were sectioned to provide light microscopy, electron microscopy, and phase extraction samples. The light microscopy samples were also used as hardness samples.

Two bars approximately 6 inches in length were solution heat treated for 4 hours at 1950°F and air cooled followed by a gradient (1100°F to 2040°F) heat treatment for 48 and 100 hours respectively. These bars were stress-free ground and hardness tested to provide supplementary data.

Samples for both light and electron microscopy were mechanically ground through 600 grit paper and polished with Linde A alumina abrasive. This was followed by a ten second electropolish in a solution of one part sulfuric acid to six parts methanol. The majority of samples were electro-etched in 10 v/o hydrochloric acid-methanol solution for ten to thirty seconds, depending upon the quantity of intermetallic compounds present in the structure. Samples containing only large amounts of Ni_3Cb plates were electro-etched for two to four seconds in a solution of chromic acid in dilute sulfuric acid. This technique gave better resolution of the Ni_3Cb plates.

All light microscopy samples were photographed at 1000X using an oil immersion lens with a 1.25 numerical aperture. Samples for electron microscopy were replicated with Collodion, shadow cast with chromium and backed with a thin deposit of carbon. The representative surfaces were photographed at 1500X and enlarged to 7500X.

The procedures for extracting the precipitated phases are similar to those used to extract phases in Udimet 700 and are described in a previous publication(4). A chemical attack with a 10 v/o bromine-methanol solution was employed for the isolation of (CbTi)C carbide, TiN, and Ti_2SC. The Ni_3Cb and carbide were coextracted electrolytically in a 10 v/o hydrochloric acid-methanol solution using a current density of 0.07 amps per square centimeter. An electrolytic extraction at 0.1 amps per square centimeter in a solution of 2 parts hydrochloric acid, 9 parts methanol, and 9 parts water was used to extract the transition phase Ni_xCb. Extraction times were limited to a four hour period or less depending on the amount of residue required for analysis. Samples for all extraction techniques were weighed before and after the extraction. The residues were collected on a tared solvent-resistant membrane filter, washed in methanol, air dried, and weighed. The electrolytic extraction apparatus consists of water cooled cells equipped with Teflon stop-cocks at the bottom for draining the residue and electrolyte as illustrated in Figure 3.

To detect trace amounts of Ni_3Cb, the electrolyte and residue from the hydrochloric acid-methanol extraction were allowed to settle for approximately two hours. The solution containing the small Ni_3Cb particles was decanted from the settled MC carbide and filtered through a solvent-resistant membrane filter. The filter pad containing the residue was mounted on a glass slide using double adhesive cellophane tape. The residue was sealed to the slide with 10 mil Mylar. A Phillips Diffractometer equipped with a monochromator was used to obtain traces for identification of phases.

Small portions of the extracted residues were mounted on glass slides using a 1 v/o solution of Formvar-ethylene dichloride as specimens for light microscopy. Standard chemical procedures were used to analyze the residues. The MC chemistries were determined from the bromine residues while the combined MC plus Ni_3Cb chemistries were determined from the electrolytic residues. The Ni_3Cb chemistries were calculated by difference.

Discussion of Results:

The major phases detected in this study by microscopy, x-ray, and/or chemical analysis were Ni_3Cb, Ni_xCb, MC, and gamma prime. The compositions and structures of these phases are listed in Table II. The morphologies of some phases are depicted in Figure 4. The plate-like form of Ni_3Cb is confirmed in Figure 4C. This residue from a hydrochloric acid-methanol extraction also contains (Cb,Ti)C and TiN particles. Typical (Cb,Ti)C particles are shown in Figure 4B. This residue which was extracted from a solution heat treated specimen also contains TiN particles. These particles have a more cubic morphology than the carbides and are bright yellow-orange in color. Chemical analysis of the residue indicates that the residue contains about 2 w/o TiN. Some particles are probably of the carbo-nitride type since they are lightly tinted orange. Specimens solution heat treated and then aged develop a fine almost plate-like precipitate (Figure 4C) which is recovered in the bromine-methanol extracted residue. This phase is believed to be CbC as reported by Cremisio et al(5). Gamma prime was detected only by metallographic techniques. The structure of this phase has not been resolved and has been reported as both BCT(2,6) and FCC(1,7). Gamma prime was not isolated in this investigation because primary interest centered upon Ni_3Cb reactions.

The data given in Figure 5 show the weight percent of Ni_3Cb determined in each heat treated condition. A trace amount of Ni_3Cb was detected after 0.2 hours exposure at 1700°F. No Ni_3Cb was found at 1850°F and above or at 1200°F and below even at 2000 hour exposures. At 1700°F and above only Ni_3Cb forms. There is one area which is outlined near the 48 hour-1800°F position. In this area the amount of

Ni_3Cb detected by x-ray and chemical analysis does not agree with microscopic observations. The x-ray diffraction shows a slight change in lattice parameters but there is no detectable change in chemistry of the extracted residue. Although there is no clear explanation for this observation, the region is probably a zone in which the Ni_3Cb has a slight change in structure.

Typical structural changes which occur during the 1700°F exposure are shown in Figure 6. Ni_3Cb first precipitates at twin or grain boundaries and often grows in a cell-like manner. A full Widmanstätten structure develops during a 24 hour exposure. Beyond 24 hours exposure there is an obvious growth in plate size. Aside from the minor structural change previously described, Ni_3Cb is stable and has a constant chemistry to at least 1000 hours exposure at 1600°F.

The 48 hour and 100 hour hardness curves from the gradient bars are the same as shown in Figure 7. Hardness behavior is normal except in the temperature range 1575°F to 1675°F. This plateau and the abrupt drop in hardness at 1675°F indicates a phase change. At 1600°F there is a marked decrease in the amount of $Ni3Cb$ recovered by extraction and the amount observed metallographically. As shown in Figure 8 this is caused by a fine plate-like phase which was identified by x-ray diffraction as the BCT phase Ni_xCb. The composition of this phase is currently under investigation. Specimens exposed for 500 hours or more at 1600°F had no Ni_xCb. A line placed at 1650°F on the $Ni3Cb$ TTT curve in Figure 5 indicates the approximate Ni_xCb stability limit.

Structural details of changes in gamma prime, Ni_xCb, and $Ni3Cb$ morphologies are shown in Figure 9. After 1 hour exposure at 1600°F the predominant phases are fine gamma prime and Ni_xCb. A moderately large amount of Ni_3Cb can be seen at the grain boundaries. After 100 hours exposure at 1600°F, the predominant phases are coarse Ni_xCb and Ni_3Cb plates. Only a few very large gamma prime particles remain in the structure. Figure 10 reveals a large amount of gamma prime, Ni_xCb, and Ni_3Cb after 100 hours exposure at 1450°F. Only very fine gamma prime is present after 100 hours exposure at 1250°F.

Summary and Conclusions:

The MC phase has the composition $(Cb_{.85}Ti_{.15})C$ and is stable within the parameters studied. There is an indication of CbC precipitation in less than 2 hours exposure at 1600°F.

The structures indicate that the spheroidal gamma prime changes to Ni_xCb which in turn changes to Ni_3Cb at temperatures above 1200°F and below about 1650°F. The approximate upper stability limit for Ni_xCb was determined. In contrast with previous publications(1) no Laves phase was found to precipitate within the time-temperature parameters studied.

Ni_3Cb has the composition $Ni_3(Cb_{.8}Ti_{.2})$ and precipitates much faster than indicated in previous investigations as shown in Figure 11.

Since Ni_3Cb may precipitate in strain-free samples in about 6 minutes at 1700°F, it may be present in material commercially heat treated in this temperature range. Therefore, heat treatments below 1850°F should not be considered absolute solution heat treatments for Inconel Alloy 718.

REFERENCES

1. H. L. Eiselstein, Metallurgy of a Columbium-Hardened Nickel-Chromium-Iron Alloy, ASTM Special Technical Publication No. 369, 1965, pp. 62-79.

2. H. J. Wagner and A. M. Hall, Physical Metallurgy of Alloy 718, Defense Metal Information Center, Report No. 217, Battelle Memorial Institute, Columbus, Ohio, June, 1965.

3. W. B. Aufderhaar and R. S. Cremisio, Some Structural Aspects of Thermomechanically Treated 718 Alloy, paper presented at the AIME Thermomechanical Treatment of Nickel-Base Superalloys Symposium at the National Metals Congress, Cleveland, Ohio, October 17, 1967.

4. W. J. Boesch and H. B. Canada, Phases Present in the Wrought Superalloy Udimet 700, Journal of Metals, 20, April 1968, pp. 46-50.

5. R. S. Cremisio et al, The Effect of Thermomechanical History on the Stability of Alloy 718, paper presented at the International Symposium on Structural Stability in Superalloys, Seven Springs, Pennsylvania, September 4, 1968.

6. E. L. Raymond, Effect of Grain Boundary Denudation of Gamma Prime on Notch-Rupture Ductility of Inconel Nickel-Chromium Alloys X-750 and 718, Transactions of the Metallurgical Society of AIME, 239, 1967, pp. 1415-1422.

7. J. F. Barker et al, Effect of Long Time Exposure on Inconel 718, paper presented at the ASM Thermal Stability Symposium at the National Metals Congress, Cleveland, Ohio, October 16, 1967.

Table I

CHEMISTRY AND PROPERTIES OF HEAT 7-3516

Chemical Composition

Ni	Cr	Mo	Cb+Ta	Ti
53.8	18.0	3.00	5.24	1.00

Al	C	S	B	Fe
0.59	0.05	0.003	0.004	Bal.

Tensile Properties

	Temp. (°F)	UTS (Ksi)	0.2 YS (Ksi)	Elong. (%)	R.A. (%)
Spec.	R.T.	180.0	150.0	12	15
7-3516	R.T.	206.0	172.0	25.6	44.0
Spec.	1200	145.0	125.0	10	15
7-3516	1200	166.0	142.0	28.3	32.6

Table II

IDENTIFICATION OF PHASES

Phase	Composition (Atomic Ratio)	Structure
Ni_3Cb	$Ni_3(Cb_{.8}Ti_{.2})$	Orthorhombic
MC	$(Cb_{.85}Ti_{.15})C$	FCC
Ni_xCb	----	BCT
Gamma Prime	$Ni_3(Al,Ti,Cb)$	FCC-BCT

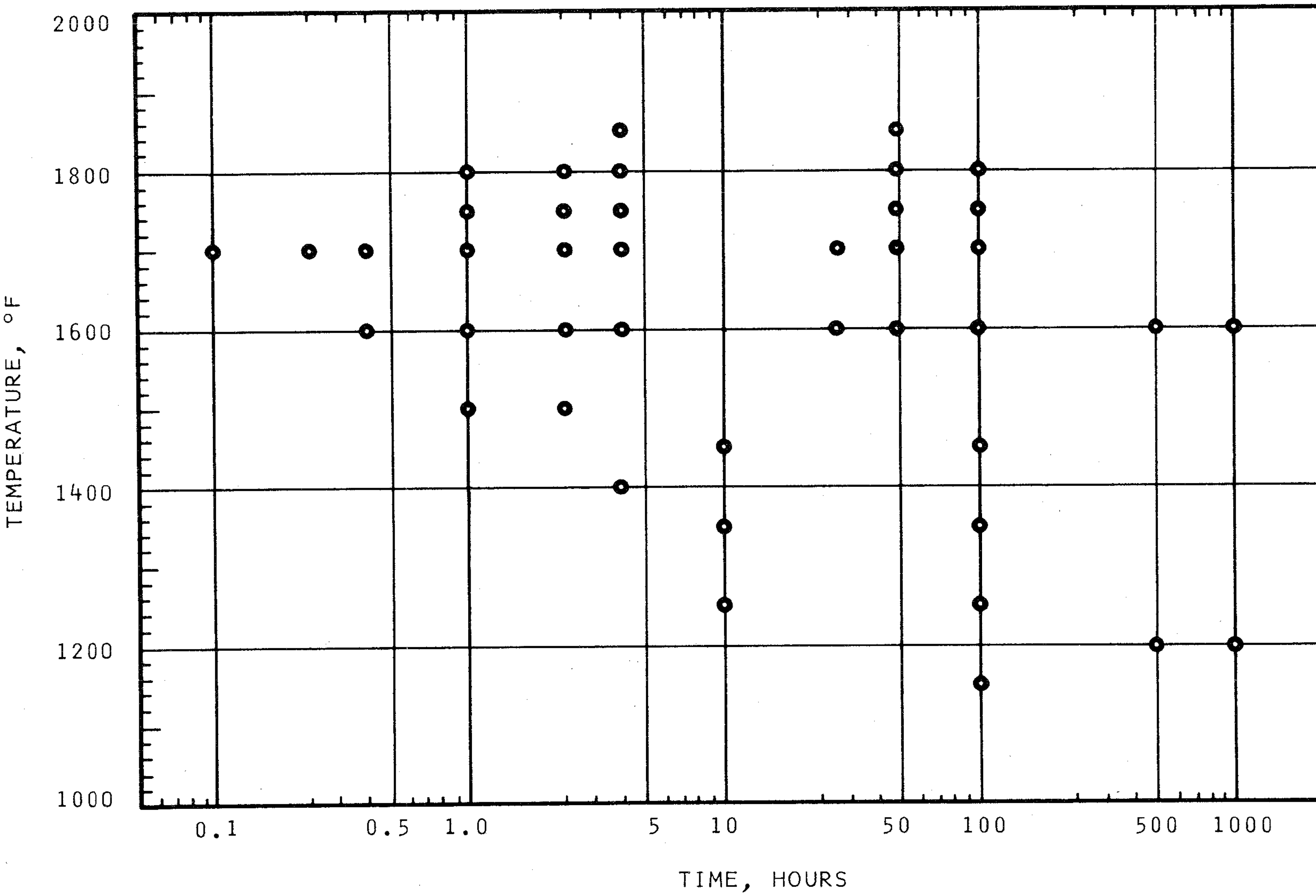


FIGURE 2 - DIAGRAM OF HEAT TREATMENT PROGRAM
(DOTS REPRESENT TIME-TEMPERATURE AGING PARAMETERS)

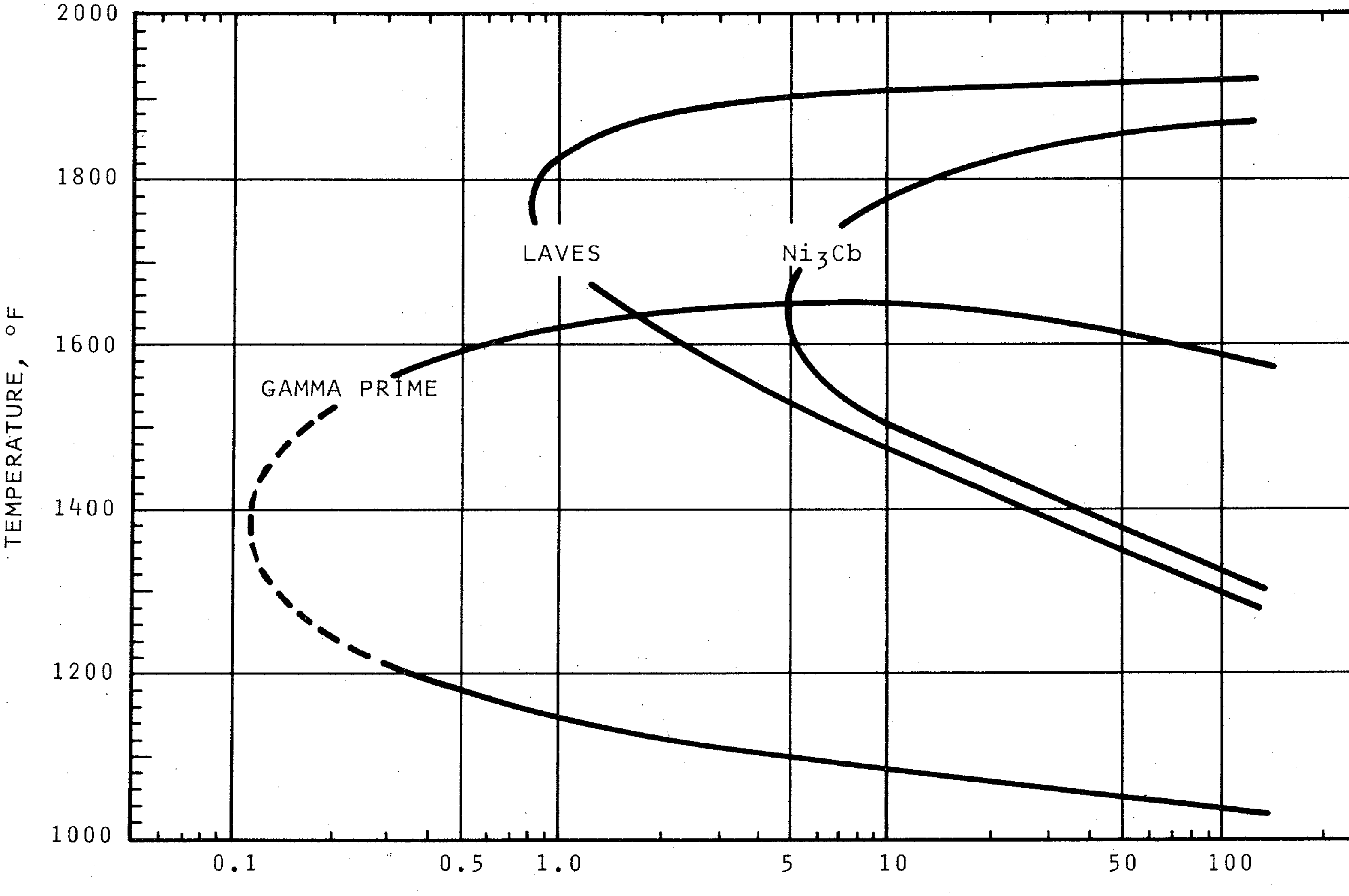


FIGURE 1 - ABRIDGED TIME-TEMPERATURE-TRANSFORMATION DIAGRAM (REFERENCE 1)

H-72144

FIGURE 3
ELECTROLYTIC EXTRACTION
APPARATUS

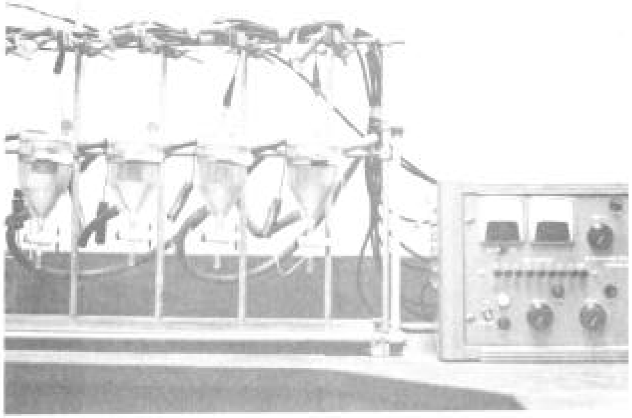
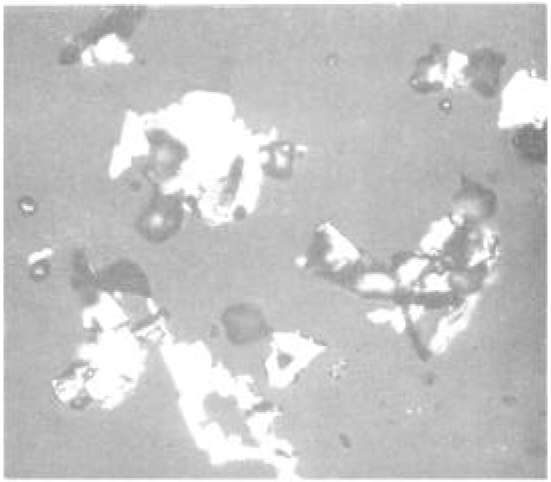


FIGURE 4 - EXTRACTED RESIDUES

4A Ni_3Cb, (Cb,Ti)C, AND TiN; HCl-METHANOL EXTRACTION

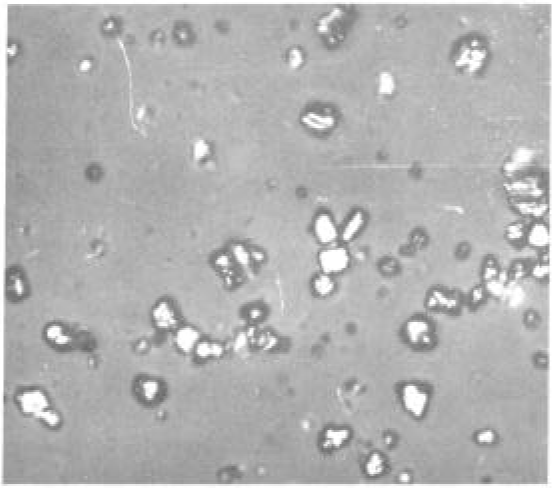
4B (Cb,Ti)C, AND TiN; Br_2-METHANOL EXTRACTION

4C (Cb,Ti)C, TiN, AND CbC; Br_2-METHANOL EXTRACTION



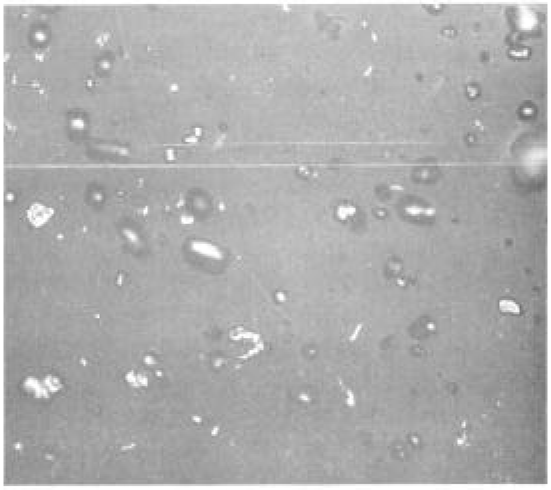
4A 500X

1950°F-4HRS-AC+1700°F-100HRS-AC



4B 500X

1950°F-4HRS-AC



4C 500X

1950°F-4HRS-AC+1600°F-2HRS-AC

H-72144

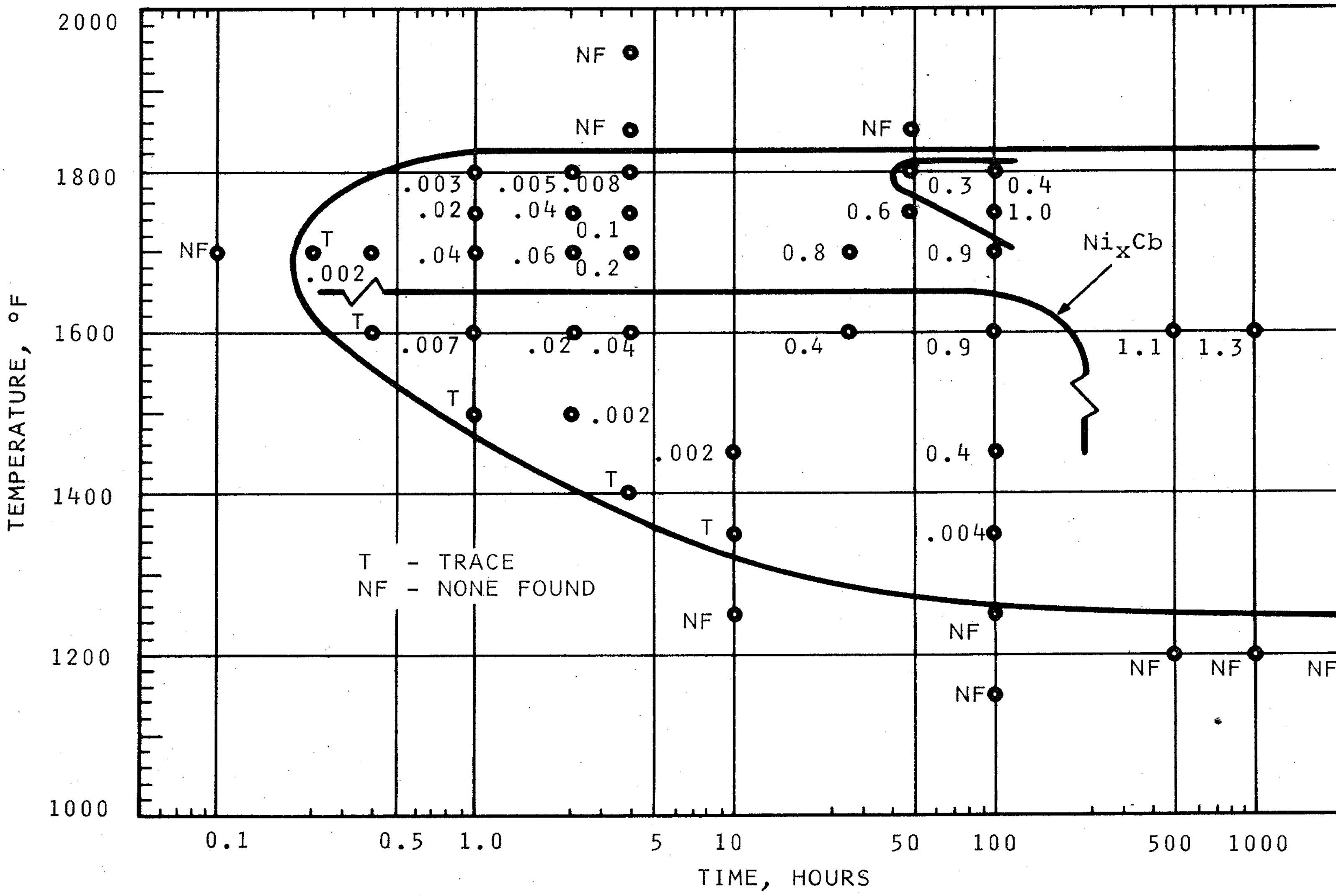
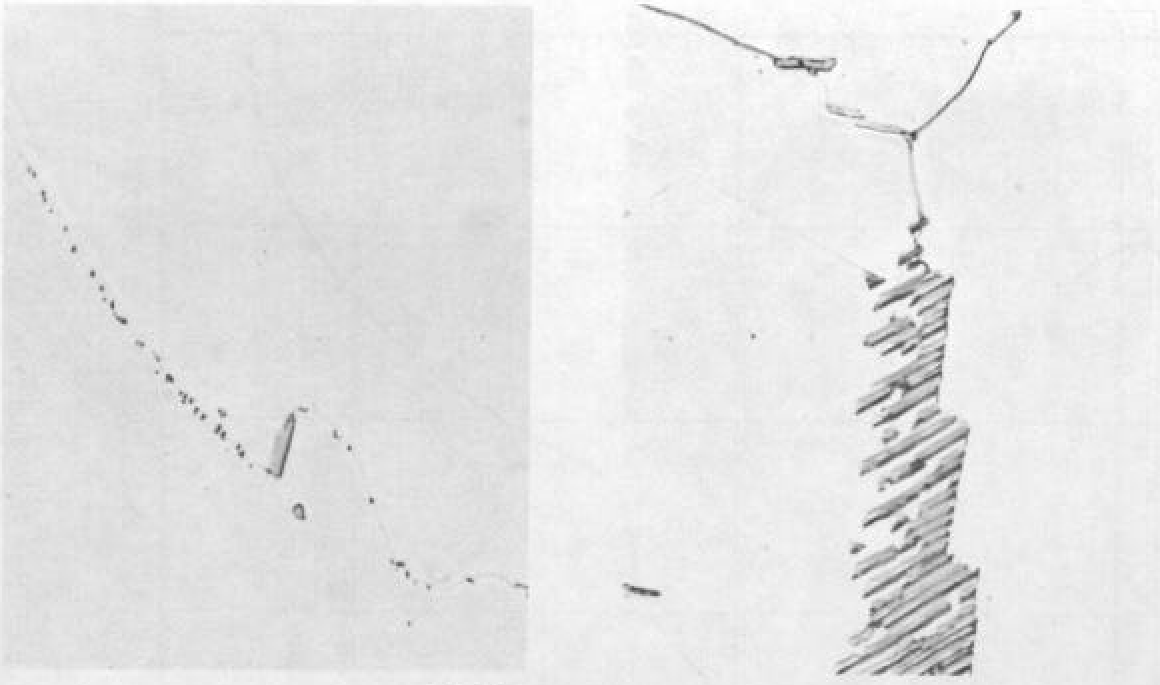


FIGURE 5 - Ni_3Cb TTT CURVE AND QUANTITATIVE EXTRACTION DATA

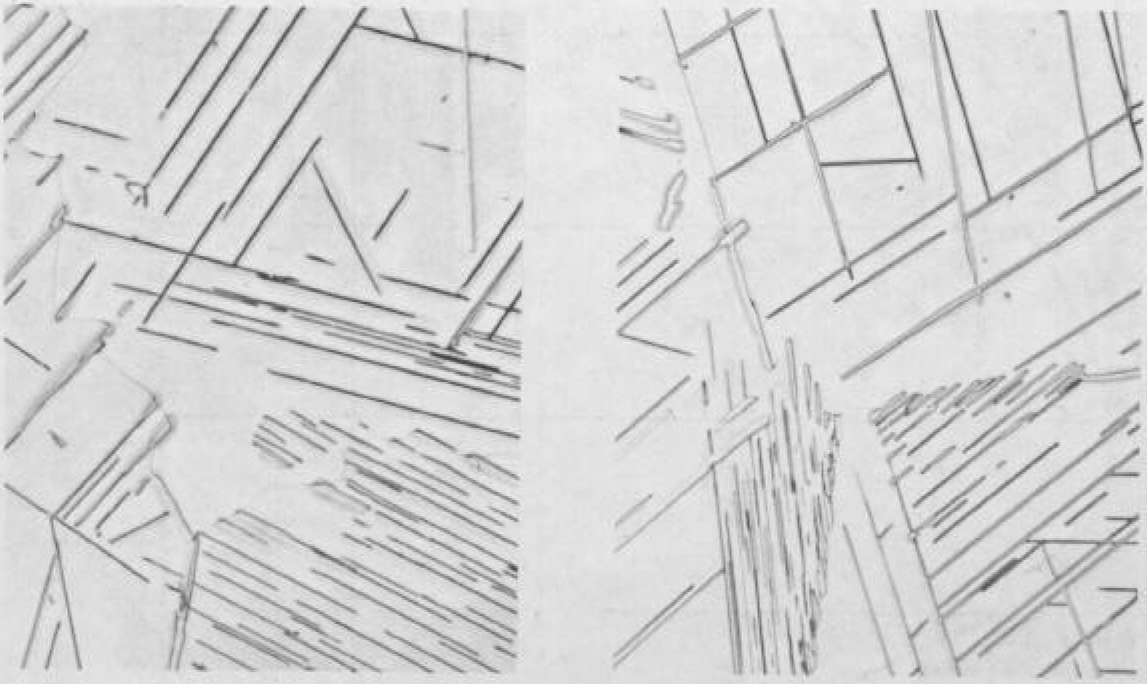
203-383-2000

H-72145



1000X
0.2 HOURS

1000X
4 HOURS



1000X
24 HOURS

1000X
100 HOURS

FIGURE 6
EFFECT OF 1700°F EXPOSURE UPON Ni_3Cb PRECIPITATION

H-7214

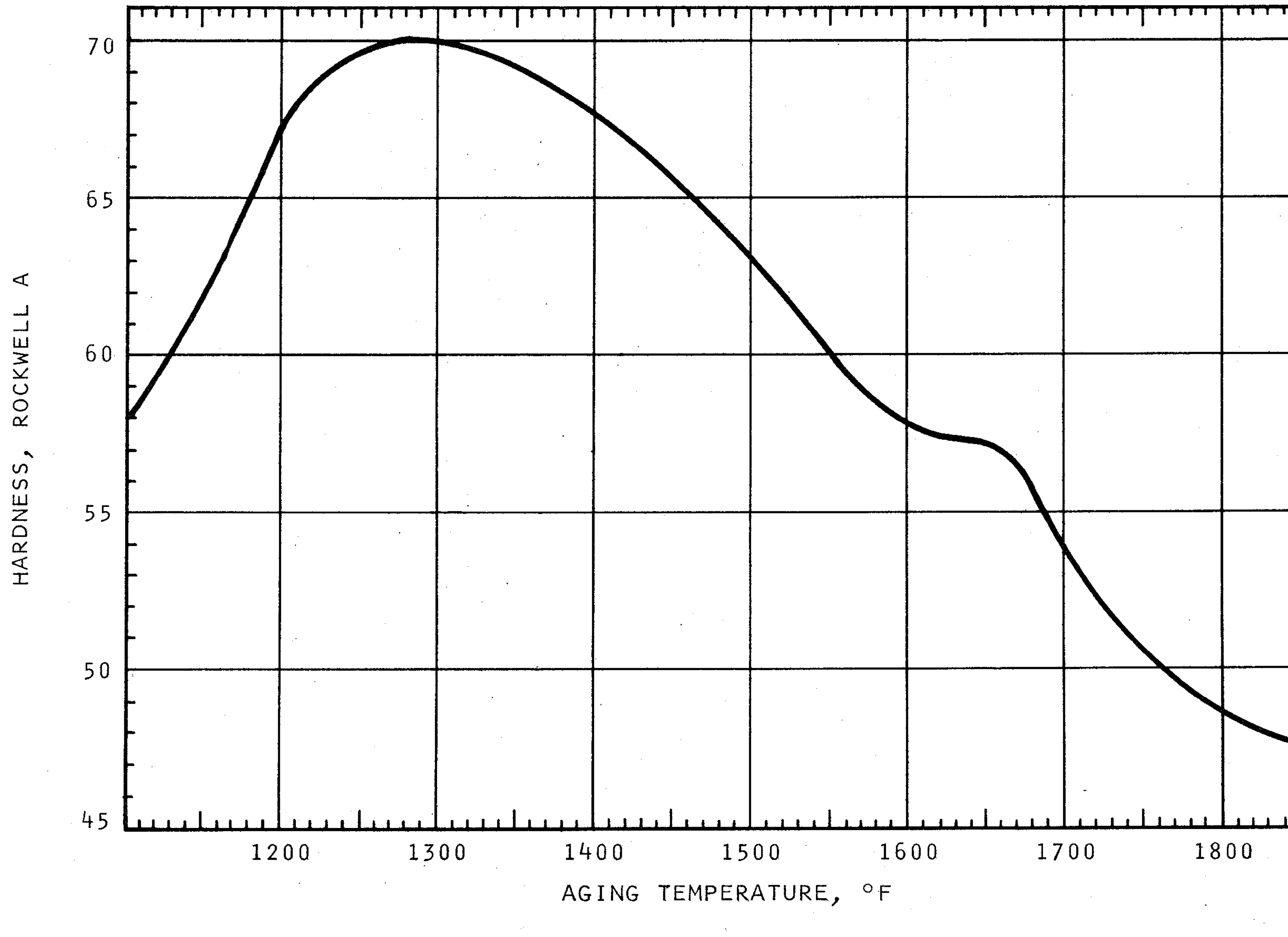
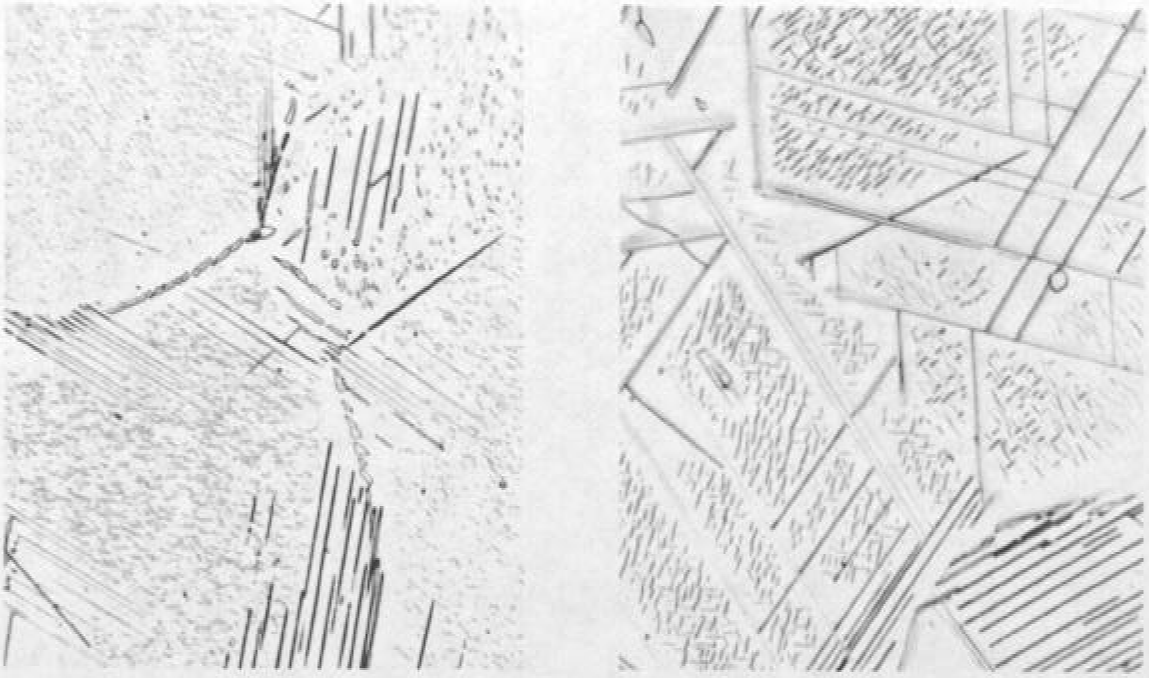
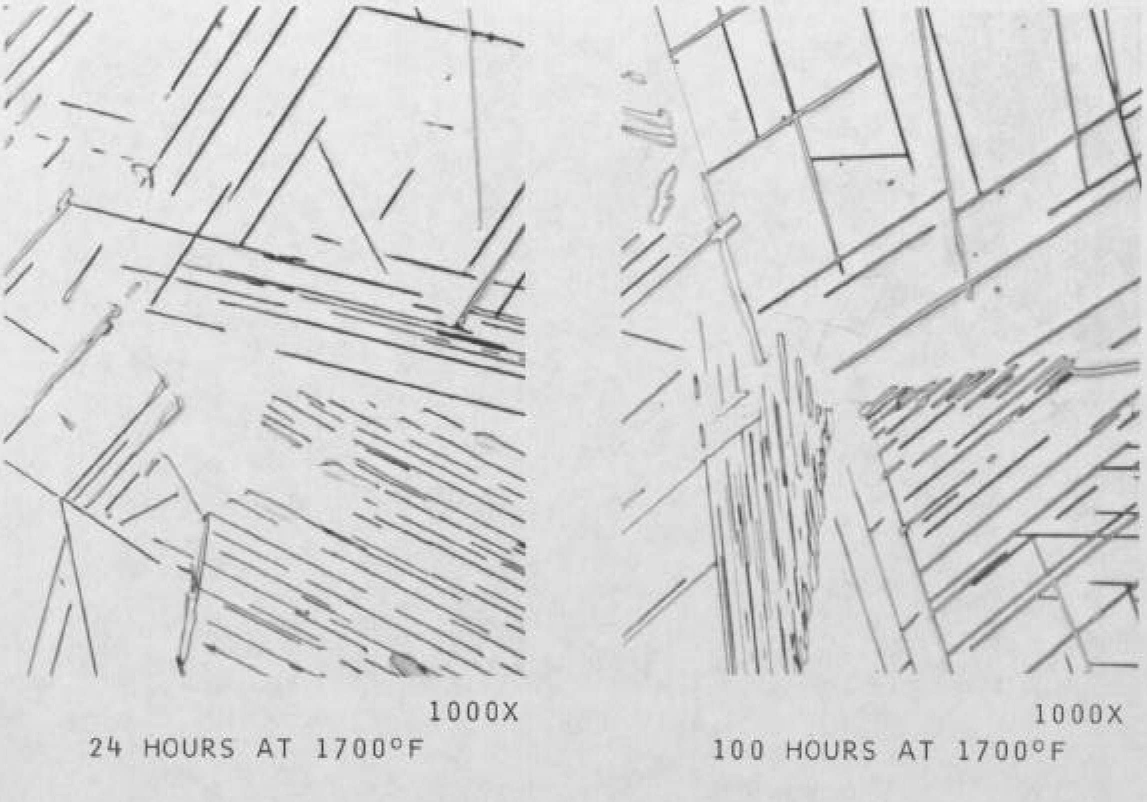


FIGURE 7 - HARDNESS OF GRADIENT HEAT TREATED BARS

H-72146


1000X
24 HOURS AT 1600°F

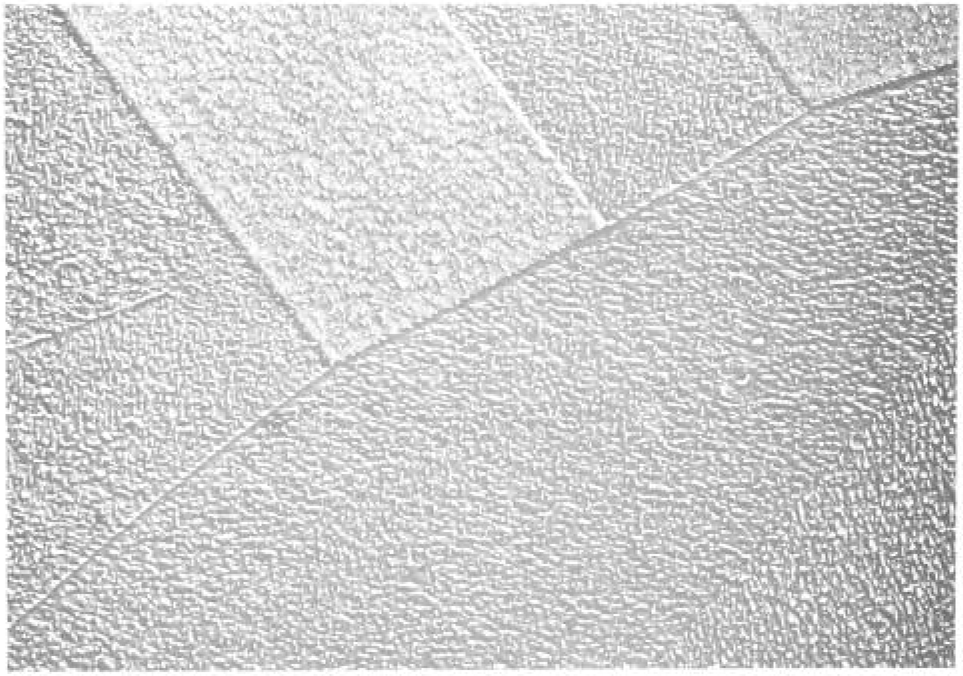
1000X
100 HOURS AT 1600°F


1000X
24 HOURS AT 1700°F

1000X
100 HOURS AT 1700°F

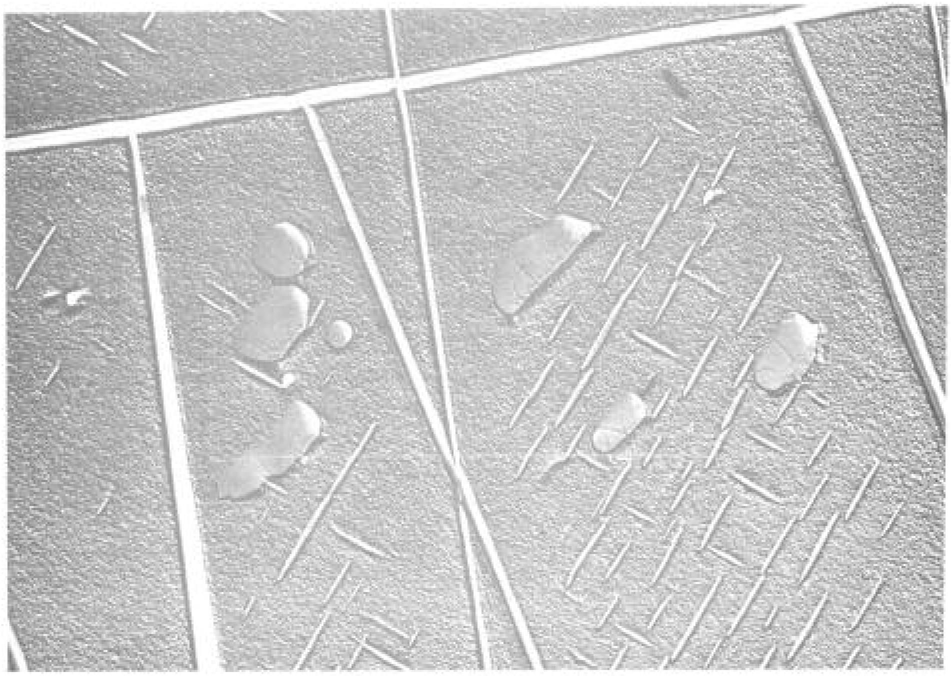
FIGURE 8
EFFECT OF Ni_xCb PRECIPITATION UPON Ni_3Cb FORMATION

H-7214



7500X

1 HOUR EXPOSURE

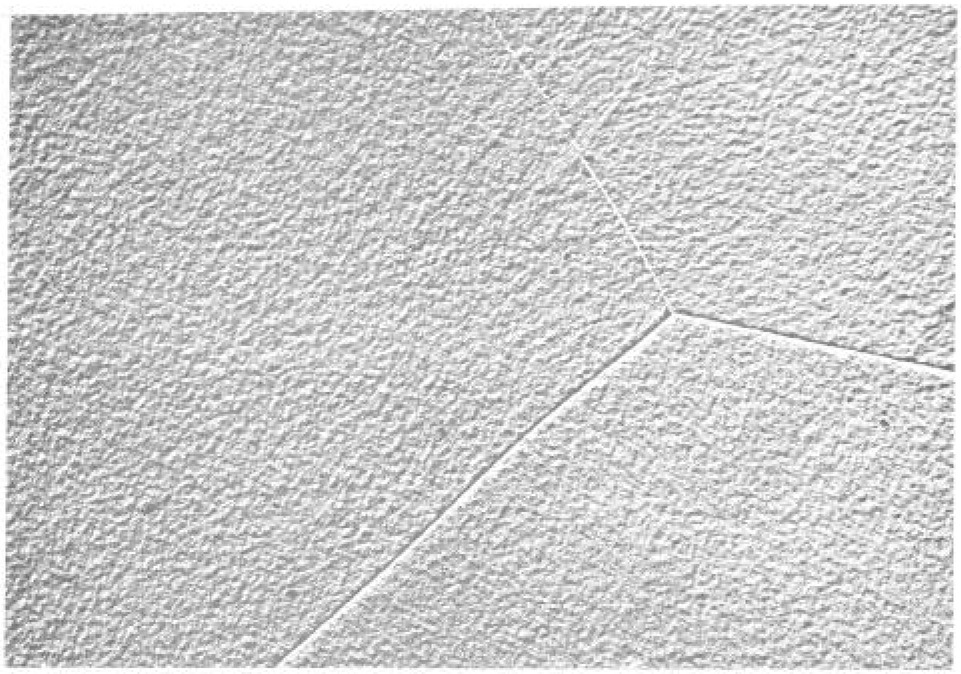


100 HOUR EXPOSURE 7500X

FIGURE 9
CHANGE IN GAMMA PRIME, Ni_xCb, AND Ni_3Cb
MORPHOLOGY AT 1600°F

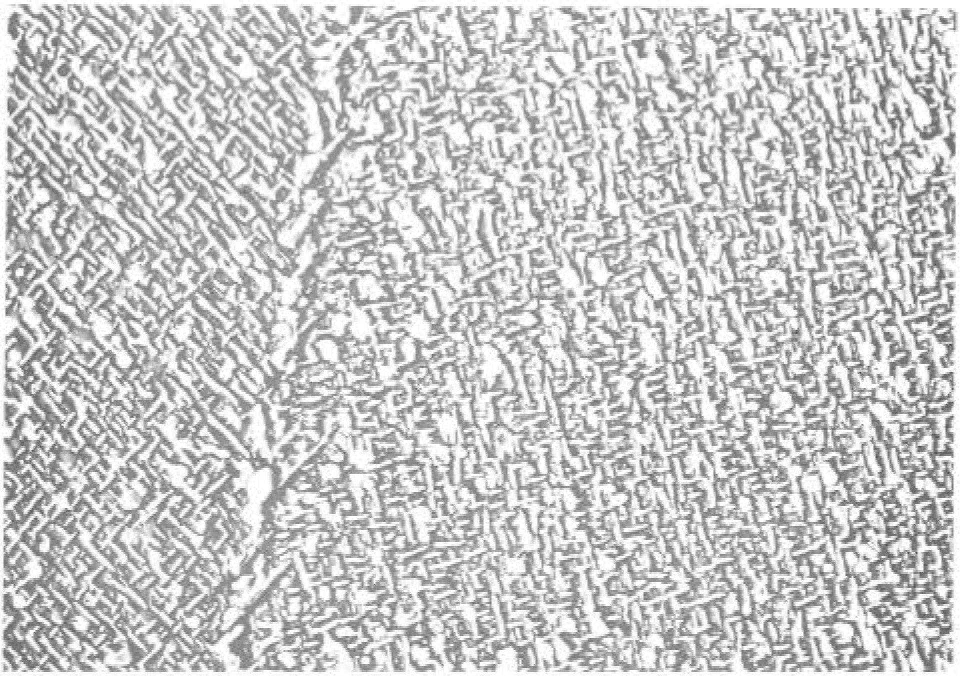
H-72147

H-72147



7500X

100 HOURS AT 1250°F



7500X

100 HOURS AT 1450°F

FIGURE 10
EFFECT OF MODERATE TEMPERATURE EXPOSURE UPON
INCONEL ALLOY 718 STRUCTURE

H-72148

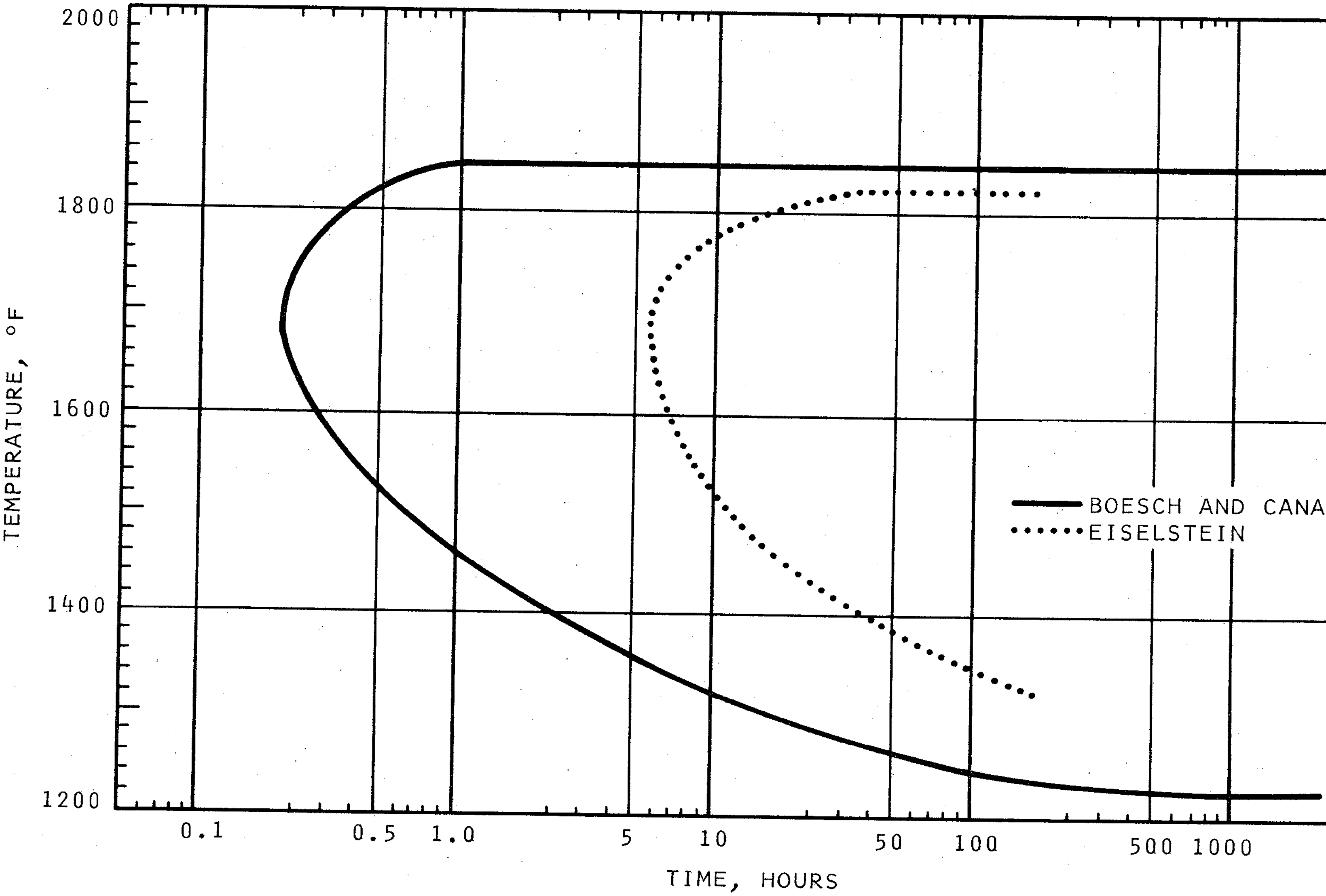


FIGURE 11 - COMPARISON OF Ni_3Cb TTT CURVES