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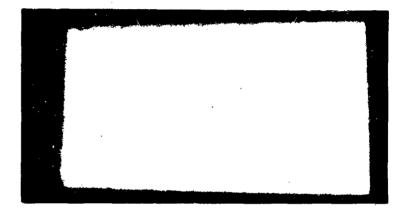
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EFFECT OF VARIOUS
ANNEALING CYCLES
ON INCONEL 718
MICROSTRUCTURE

REPORT	A470	SERIAL NO.	20

MCDONNELL

This report was prepared under Contract Number AF33(657)-11215 and BPSN: 63-6899-7381-738103. Additional information pertaining to any data contained herein may be obtained from the Directorate of Materials and Processes (ASRCEM-1), Aeronautical Systems Division, Air Force Systems Command, United States Air Force, Wright-Patterson Air Force Base, Ohio, or McDonnell Aircraft Corporation, St. Louis, Missouri

INDEX.	
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DATE 12 December 1	MCDONNELL ST. LOUIS, MISSOURI	
EVISED	LABORATORY REPORT	PAGEA
	(Ni-4)(I-e)(IV-e)	FINAL REPORT
	LABORATORY: Structures	
	OF INCOMEL 718 MICROSTRUCTURE	CLES
	ABSTRACT	
of various anneal	ographic studies were performed to i ling cycles on grain size and precip el chromium alloy.	immestigate the effect pitate morphology in
	pecimens of .040-inch material were then annealed 15 minutes at 1500, 1 and 2150F.	
Before determined and pl	and after aging, the DFH hardness of hotomicrographs at 250% and 2000% we	of all material was ere prepared.
satisfactory rest	ing temperatures of 1500 and 1600F fults. Annealing between 1700 and 18 es so that subsequent aging produces	BOOF adequately diameter
completely disso	ing overaged material at 1900F for l lve precipitate phases without alter bsequent aging or encouraging excess	ring precipitation be-
grain growth can	nnealed at temperatures greater than be expected. Precipitation behaviours different with grain boundary fil	or during subsequent
PREPARED BY Rest		Denkaliza enior Engineer, Materials ethods, Metallurgical Grou

FIRAL REPORT

ST. LOUIS, MISSOURI		
	PAGE	2
	REPORT	A470
		ST. LOUIS, MISSOURI PÄGE

1. INTRODUCTION

Incomel 718 is a precipitation hardening, nickel base alloy which offers excellent strength up to 12007 and good weldability (reference 1). Bomimal composition of the alloy is 52 Mi, 19 Cr, 3 Mo, 5 Cb + Ta, 0.8 Ti, 0.6 Al, 0.05C, and the remainder iron. Incomel 718 differs from other super alloys of its class in that aluminum and titanium are not the principal hardening elements. Instead, it is strengthened by a gamma prime type of precipitate containing a considerable amount of columbium as well as molybdenum, titanium, and possibly aluminum.

The precipitation hardening reaction of the alloy is relatively sluggish, a feature of the alloy attributed to relatively slow diffusion rates of the elements which produce strengthening. This slow aging response is of benefit to both fabrication and welding processes. Distortion is minimized while formability and weldability can be maintained at high levels because low annealed hardness is obtained by air cooling from annealing temperatures.

Incomel 718 is being used and planned for many applications at McDonnell. Because the principal hardening phase differs from other superalloys, precipitation behavior during heat treating should be expected to differ. At the present time information on the effect of commercial heat treatments on the precipitate morphology is not available in the literature. To investigate this factor, metallographic studies were performed to observe precipitation behavior after annealing at various temperatures.

The approximate formula of the principal strengthening phase in Incomel 718 is Wi₃(Cb, Mo, Ti). Previous work (references 2 and 3) indicates the phase to be metastable in nature and possesses a face centered cubic structure which is coherent with the austenitic matrix. X-ray diffraction work has shown the lattice parameter of Wi₃(Cb, Mo, Ti) precipitate is approximately 0.8% greater than the matrix which produces the strengthening lattice strain. The compound has been found to precipitate heavily during aging between 1300 and 1400F.

Aging for long times or at higher temperatures results in formation of stable Ni₂Cb compound with a hexagonal close-packed structure that is non-coherent with the matrix. Formation of Ni₂Cb relieves lattice strain and results in overaging. The compound is commonly observed as a coarse, acicular precipitate formed after aging long times at temperatures from 1300 to 1700F. It probably dissolves at temperatures in excess of 1850F.

As in most super alloys, the carbon present is tied up by columbium and some titanium as (Cb, Ti) C. The primary carbide phase occurs as coarse blocky particles randomly dispersed and relatively insensitive to temperature. Some solutioning of (Cb, Ti)C may occur around 2200F and permit reprecipitation during aging as a grain boundary film. In addition to the primary carbide, TiN

DATE 12 December 1962	ST. LOUIS, MISSOURI		
REVISED	LABORATORY REPORT	PAGE	3
KEVIJEO		REPORT	A470
REVISED			

1. INTRODUCTION (CONT'D.)

is also frequently observed as distinctly angular, randomly dispersed particles that are not dissolved at temperatures as high as 2250F.

FINAL REPORT

While not normally observed in wrought Inconel 718, cast material has been found to possess additional phases. A Laves phase, M₂ (Cb, Ti), is commonly observed where M can be nickel, iron, or chromium and molybdenum may replace columbium and titanium in the radical (Cb, Ti). When wrought materials are solution treated at temperatures in excess 2250F M₂ (Cb, Ti) can be formed by incipient melting at the grain boundaries. In castings, acicular Hi₂Cb is observed to occur abundantly around M₂ (Cb, Ti) and form with greater rapidity than in wrought material. On this basis, it has been suggested that the presence of M₂ (Cb, Ti) may hasten the formation of acicular Hi₂Cb. Another carbide, Cr₇C₃, has also been found in cast materials associated with M₂ (Cb, Ti) and acicular Hi₂Cb. It is probable that Cr₇C₂ is formed only after appreciable amounts of columbium are tied up as M₂ (Cb, Ti) and/or Hi₂Cb. While these phases are not normally found in wrought Inconel 718, consideration will have to be given them in evaluating the welding behavior of the alloy.

This test was conducted by the Structures Laboratory, McDonnell Aircraft Corporation, during the period 2 July 1962 through 28 September 1962.

2. TEST NATERIALS

The material evaluated was Inconel 718 in the form of 0.040-inch sheet. Material was received in the mill-annealed temper with a pickled finish.

3. TEST SETUP AND PROCEDURE

Test specimens 0.040 x .05 x 1.0-inch were sheared from the as-received material. Except for retaining a sample of as-received material, all specimens were "overaged" at 1400F for 30 hours.

To determine the extent to which various annealing temperatures would eliminate the overaged structure, duplicate specimens were annealed 15 minutes at 1500, 1600, 1700, 1750, 1800, 1900, 2000, 2100 and 2150F. After annealing the duplicate specimens, one specimen was allowed to cool in still air, while the other was quenched in water.

All specimens were then sectioned and one-half mounted, DFH hardness was determined, and the specimens prepared for metallographic examination. Photomicrographs were then prepared of as-received material, overaged material, and the overaged material which was annealed at the 9 annealing temperatures.

The same procedure was repeated after aging per PS 15603.

DATE 12 December 1962	ST. LOUIS, MISSOURI		
REVISED		PAGE	4
		REPORT	A470
REVISED			

FINAL REPORT

5. TEST SETUP AND PROCEDURE (CONT'D.)

Overaging of the material which was subsequently annealed, and aging of the annealed material were performed in a Westinghouse circulating air heat-treating furnace. All annealing was performed in tubular furnaces normally used for mechanical property determinations. The specimens to be annealed were dropped into furnaces, at the desired temperature, along with a dummy specimen containing chromel-alumel thermocouples. Annealing time was assumed to start when temperature of the dummy specimen reached furnace temperature.

DPM hardness was determined using a Vickers Microhardness Tester and a 10 KG load.

All specimens were prepared for metallographic examination using normal mechanical polishing techniques. The specimens were etched electrolytically using an electrolyte of 50cc ECl, 50cc E₂0, and 14cc 3% E₂0₂ with a platinum cathode and open circuit potential of 2 volts.

4. RESULTS

Table 1 on page 10 gives the DPH hardness of the material after the various heating cycles. The same results are shown graphically in Figures 1A and 1B on pages 11 and 12. As-received hardness of the material was DPH 188 or Rg 91. Overaging 30-hours at 1400F resulted in a hardness increase to DPH 358 or Rc 39. Annealing at 1700 to 1800F restored the material to its original hardness of DPH 184 to 188 or Rg 90 to 91. Annealing at 1900F produced slightly lower annealed hardness (DPH 179 to 180 or Rg 89) while annealing at 2000 to 2150F produced significantly lower annealed hardness (DPH 151 to 157 or Rg 76 to 77). Water quenching or air cooling the .040-inch material from annealing temperatures, failed to produce any significant variation in hardness.

Aging the annealed material produced similar results. As-received material aged to a hardness of DPH 438 or $R_{\rm c}$ 46. The overaged material also increased in hardness after aging to DPH 401 or $R_{\rm c}$ 43. Material aged after annealing at 1700 to 1900F possessed hardness of DPH 432 to 442 or $R_{\rm c}$ 46, which is equivalent to the as-received material. Material annealed at 2000 to 2150F had a slightly lower hardness after aging, ranging from DPH 427 to 433 or $R_{\rm c}$ 43. However, considerable scatter exists in these results due to the coarse grain size which prevailed. Annealing at 1500 or 1600F appears unsatisfactory. Hardness remains at intermediate values between that of overaged and as-received material in both the annealed and aged conditions.

Figure 2 on page 15 shows the microstructure of as-received material 250% and 2000%.

Figure 3 on page 14 shows equivalent microstructumes of material overaged for 30-hours at 1400F.

DATE 12 December 1962	ST. LOUIS, MISSOURI		
REVISED		PAGE	5 A470
251/1452		REPORT	A470

FINAL REPORT

4. RESULTS (CONT'D)

Microstructure of the material after the various annealing and subsequent aging cycles are shown in:

Figure	Page	Annealing Temperature
4 and 5 6 and 7	15 - 16 17 - 18	1500 1600
8 and 9	19 - 20	1700
10 and 11	21 - 22	1750
12 and 13	23 - 24	1800
14 and 15	25 - 26	1900
16 and 17	27 - 28	2000
18 and 19	29 - 30	2100
20 and 21	31 - 32	2150

Figures 22 and 23 on pages 53 and 54 show the microstructure at 250% of specimens aged after being annealed at 1800, 1900, 2000, 2100, and 2150F. Comparison of these photomicrographs shows the grain growth occurring after annealing at temperatures in excess of 1900F.

Average grain diameter of the material annealed at 1900F or below was 0.025 to 0.035 mm. Annealing at higher temperatures resulted in average grain diameters of 0.120 mm after 2000F, 0.150 after 2100F, and 0.150 to 0.200 after 2150F.

5. DISCUSSION OF RESULTS

In the mill-annealed condition, the microstructure of Incomel 718 consists of equiaxed grains showing extensive twinning which is characteristic of the austenitic matrix. The primary carbides, (Cb, Ti)C, can be observed as randomly dispersed particles. Also observable are networks of apparently spherical particles which seem to transcend the present equiaxed structure but delineate grain boundaries in a previous deformed structure.

Overaging 30 hours at 1400F results in precipitation that produces appreciable shading within the grains and visible precipitation at the grain boundaries. These effects are probably visible evidence of Ni₃Cb formation.

Annealing the overaged material 15 minutes at: 1500F removes some of the shading from inside the grains, although it subsequently returns after aging, and induces coarser agglemeration of Ni₃Cb at the grain boundaries.

Annealing 15 minutes at 1600F removes most of the shading from the grains, although it again returns after aging, and produces extremely coarse, acicular agglomeration of Ni₃Cb at the grain boundaries.

DATE 12 December 1962	ST. LOUIS, MISSOURI		
REVISED		PAGE	6
REVISED		REPORT	A470

FINAL REPORT

5. DISCUSSION OF RESULTS (CONT'D.)

Material ammealed from 1700 to 1800F and the as-received material show essentially the same structure when observed on the optical metallograph. All shading within the grains appears eliminated, even after subsequent aging, and the networks of small particles again can be observed to wander through the present structure. Appreciable amounts of coarse Ni₃Cb precipitate can still be observed at the grain boundaries. The size and amount of the Ni₃Cb, precipitate appears to decrease slightly as annealing temperature increases from 1700 to 1800F.

Annealing at 1900F for 15 minutes appears to eliminate any coarse grain boundary precipitation of MigCb. The networks of small particles through the structure still remain along with (Cb, Ti) C and some TiM.

Annealing at 2000 and 2100F for 15 minutes results in appreciable grain growth in the material. Except for the (Cb, Ti) C and TiE, all phases observable optically appear to have been completely taken in solution. However, subsequent aging produces visible precipitation at grain boundaries. This could be reprecipitation of the phase observed as the wandering networks in material annealed at 1900F and below. Annealing at 2150F produces noticeably heavier visible precipitation at grain boundaries after aging. In addition to the effects noted in material annealed at 2000 and 2100F, the 2150F anneal might partially dissolve the (Cb, Ti) C and redeposit it as films along grain boundaries and at twin bands.

6. CONCLUSIONS

Annealing aged Incomel 718 from 1700 to 1800F for 15 minutes will dissolve sufficient Ni3Cb precipitate to allow subsequent aging to maximum hardness. While some coarse precipitate may still remain in the microstructure it does not appear to occur in sufficient quantities to affect mechanical properties and may serve a useful purpose by inhibiting grain growth.

Annealing aged Incomel 718 for 15 minutes at 1900F appears to completely take into solution all coarse Mi₃Cb present without affecting precipitation behavior during subsequent aging, or encouraging grain growth.

Excessive grain growth may be expected if annealing temperatures exceed 1900F, and subsequent precipitation behavior during aging may be altered enough to deleteriously affect mechanical properties.

No variation in hardness or microstructure was found to result from air cooling or water quenching from annealing temperatures.

7. RECOMMENDATIONS

An attempt should be made to identify the random networks observed in the microstructures, as well as the grain boundary precipitation resulting

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7. RECOMMENDATIONS (CONT'D))	
to determine the effect of h	in excess of 2000F. It would also be design temperature annealing cycles on ductive in these areas will aid in understanding.	llity
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REVISED		PAGE	9
REVISED		REPORT	A470

FINAL REPORT

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- (2) M. Kaufman and A. R. Palfy, "The Phase Structure of Incomel 718 and 702 Alloys", Trans. AIME, Vol. 221, December, 1961
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REPORT	A470

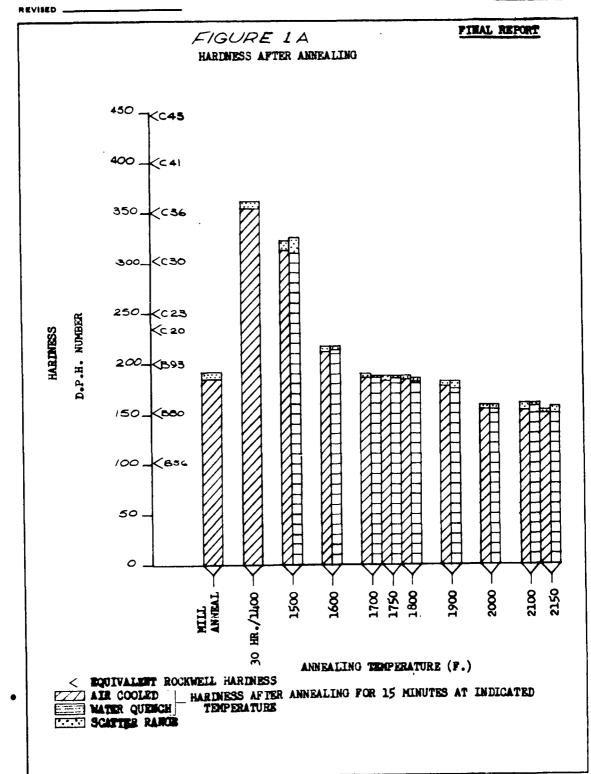
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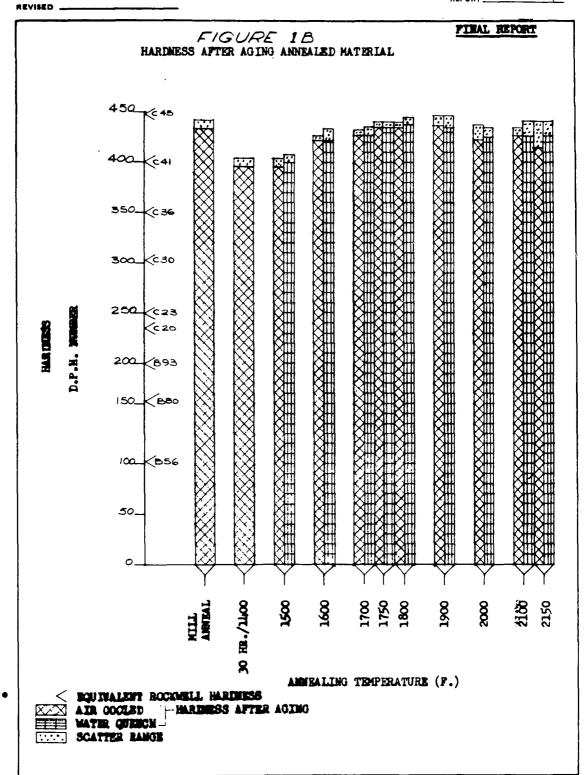
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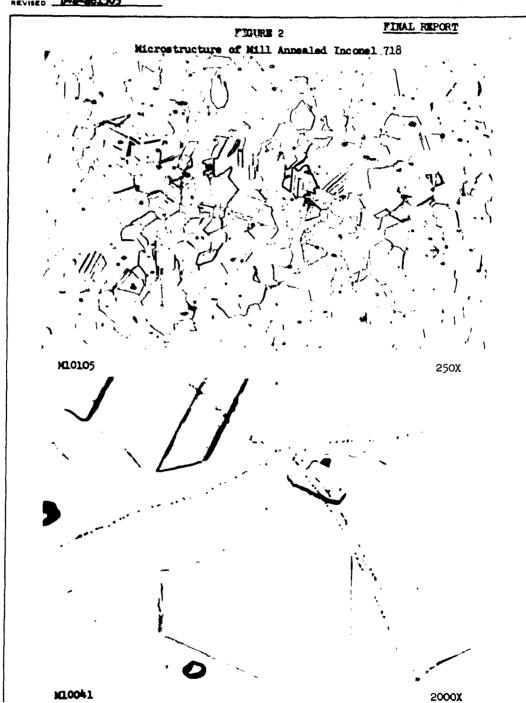
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PAGE 13

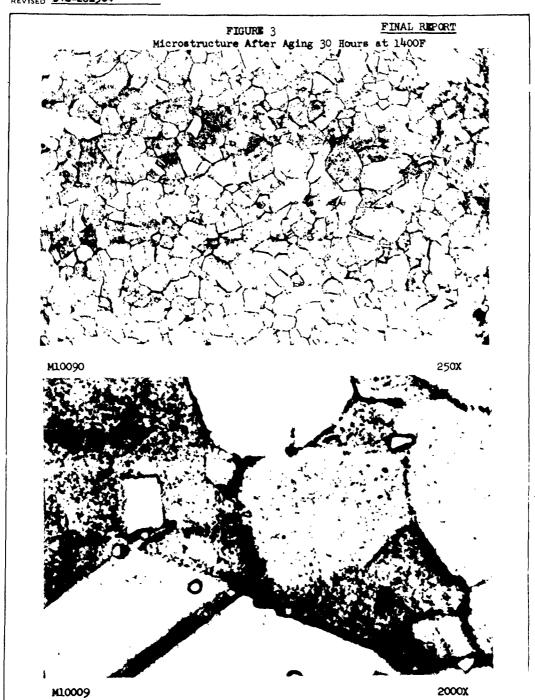
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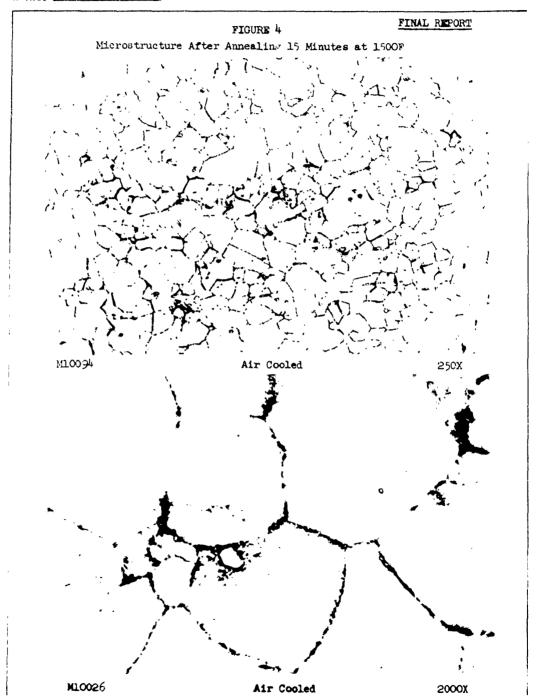
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REPORT A470



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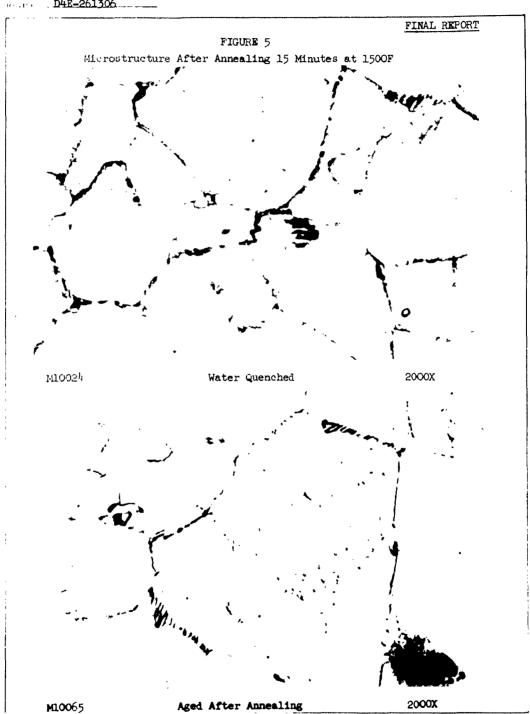


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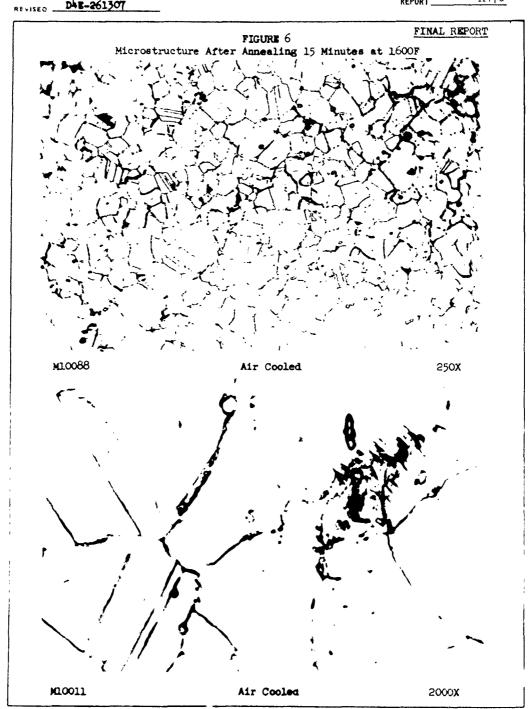


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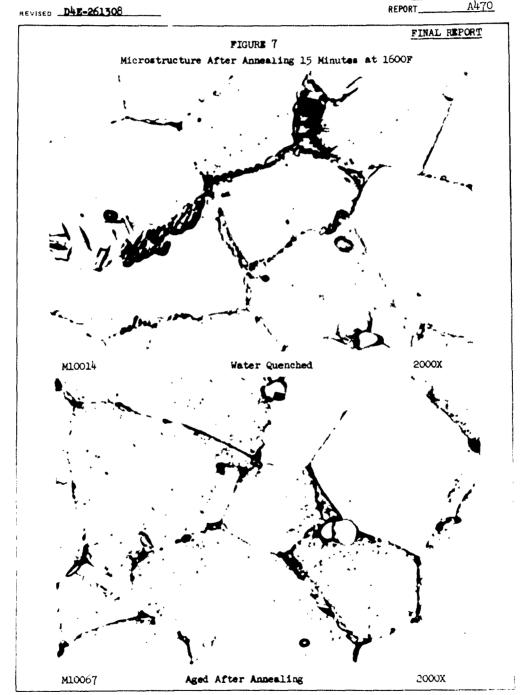
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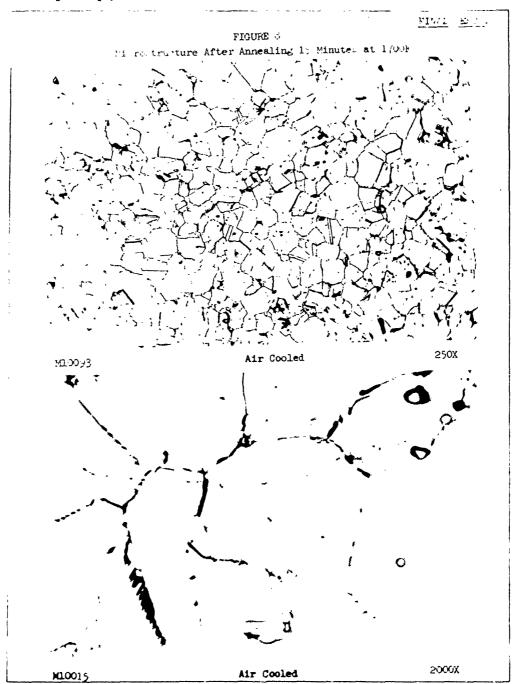


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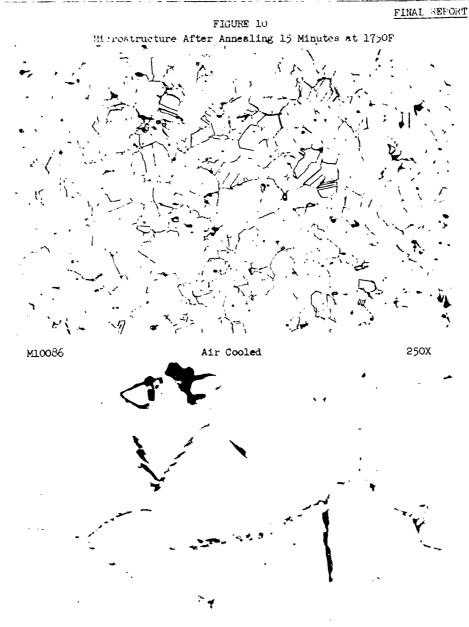


PAGE 20 REPORT A470

1845-261310 FINAL AND CAT FIGURE 5 Mr. roll-racture After Annealing 1 - Minutes at 1700F M10008 Water Quenched 2000X M10069 Aged After Annealing

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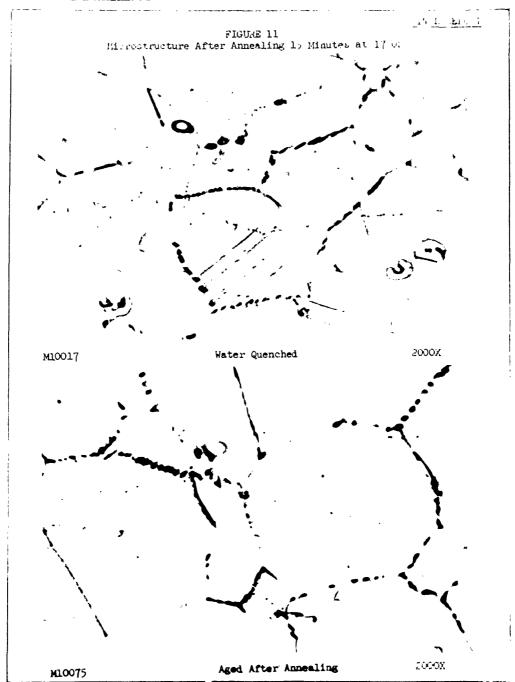
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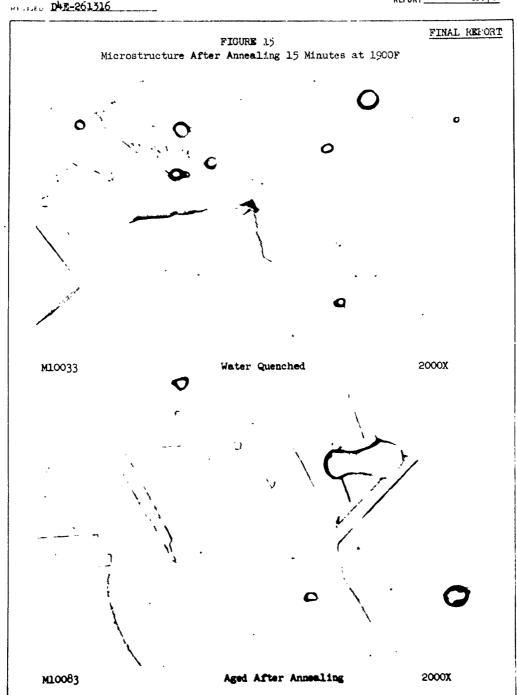
CIRT EL I FIGURE 12 Microstructure After Annealing 15 Minutes at 1800F 250X Air Cooled м10085 2000X Air Cooled M1:0022

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FINAL REPORT FIGURE 13 Microstructure After Annealing 15 Minutes at 1800F 2000X Water Quenched M10081 Aged After Annealing 2000X

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Pt. (156)		PAGE	26
when over you		REPORT	A470



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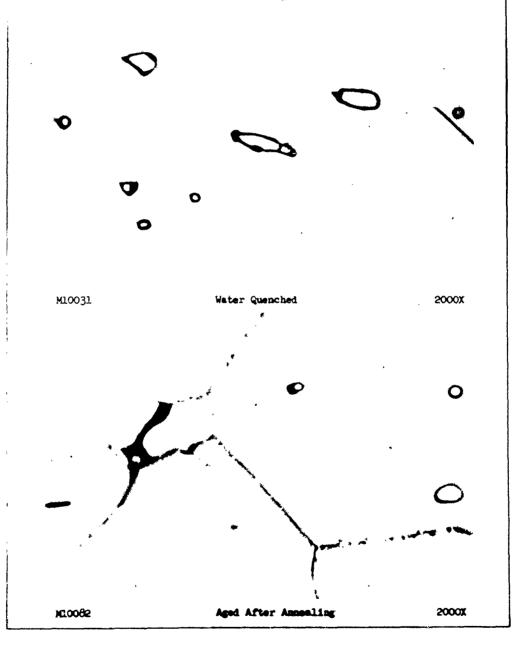
PAGE 28
REPORT A470

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FIGURE 17

FINAL REPORT

Microstructure After Annealing 19 Minutes at 2000F

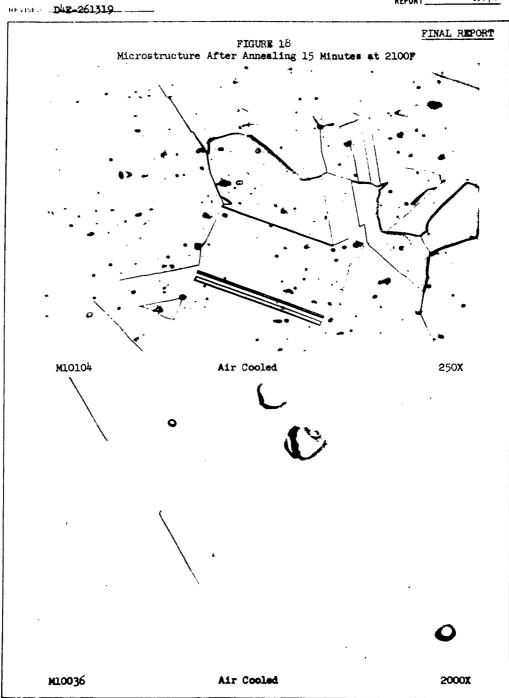


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PAGE 29

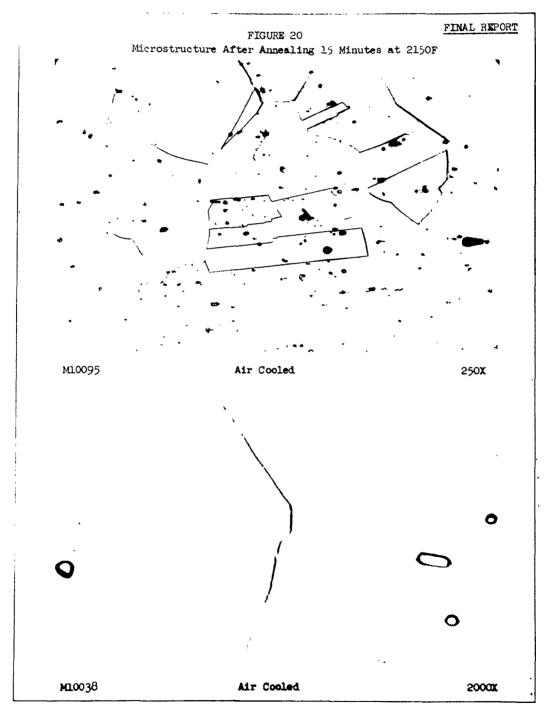
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	Microstructur	FIGURE 19 re After Annealing 15 Minutes at	<u> </u>
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		,	
	м10044 Ф	Water Quench	2000X
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	и10078	Aged After Annealing	2000X

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FINAL REPORT

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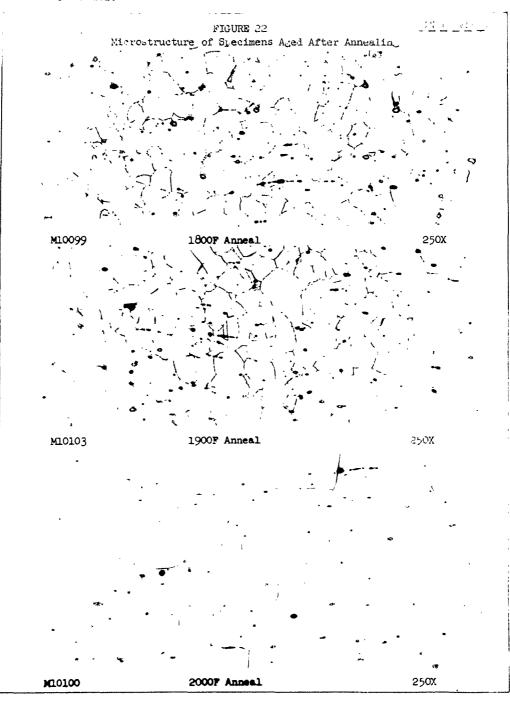
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FIGURE 21

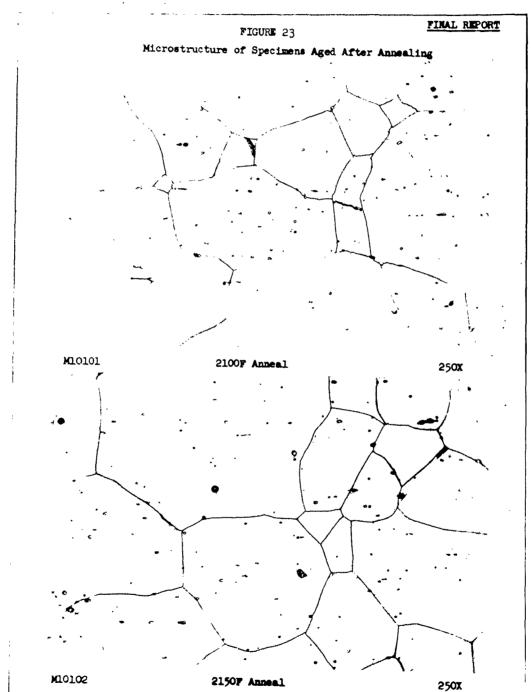
Microsorusture After Annealing 15 Minutes at 2150F

M10030 Water Quenched 2000X Aged After Annealing 2000X M10077

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TEST REQUEST

PAGE	35
REPORT	A470

TITLE	SFFET	OF	VARIOUS	ANNEALING	CYCLES	ON	INCONEL	718

MICROSTRUCTURE

OK for IDEP

WORK REQUESTED (Ni-4)(I-e)(IV-e)

OBJECTIVE (GIVE PURPOSE OF TEST, WORK AND DATA REQUIRED, INCLUDING SERVICE HISTORY AND BACKGROUND INFORMATION

1.0 OBJECT

To investigate the effect of various annealing cycles on grain size and precipitate morphology in the nickel-chromium alloy Inconel 718.

2.0 HISTORY

Inconel 718 is being used and planned for many applications at MAC. The alloy normally requires annealing and aging heat treating cycles during fabrication and cycles used have been based on recommendations from the International Nickel Co. and internally generated mechanical property data.

3.0 JUSTIFICATION

Metallographic studies of Inconel 718, subjected to various annealing cycles, will provide background information needed to deviate, if necessary, from normal annealing cycles when difficulties are encountered during fabrication.

4.0 MATERIALS

38 pieces - 0.040" x 0.5" x 1.0" nickel-chromium alloy, Inconel 718, cold rolled, annealed, temper, pickled finish (Material available from previous procurement)

ROW A ADDS ACTIONS

REFERENCES OR ENCLOSURES

Applicable to IDEP unless final report is classified. Report Summary Sheet, MAC 1008TRS required. Complete Report Summary Sheet and forward along with vellum copy of complete report to MAC IDEP Coordinator Dept. 261, Bldg. 33.

5.0 IDENTIFICATION

Identify, by steel stamping, all pieces from 4.0 as follows:

- 5.1 Al, A2 (2 specimens, to be examined as overaged)
- 5.2 Bl, B2, B3, B4 (4 specimens, 1500F anneal)
- 5.3 Cl, C2, C3, C4 (4 specimens, 1600F anneal)
- 5.4 Dl, D2, D3, D4 (4 specimens, 1700F anneal)
- . 5.5 El, E2, E3, E4 (4 specimens, 1750F anneal)
 - 5.6 H1, H2, H3, H4 (4 specimens, 1800F anneal)
 - 5.7 Kl, K2, K3, K4 (4 specimens, 1900F anneal)
 - 5.8 Ll, L2, L3, L4 (4 specimens, 2000F anneal)
 - 5.9 Ml, M2, M3, M4 (4 specimens, 2100F anneal)
 - 5.10 Pl, P2, P3, P4 (4 specimens, 2150F anneal)

6.0 HEAT TREATMENT (All temperatures ± 25F)

- 6.1 Overage all specimens from 5.0 by furnace heating at 1400F for 30 hours, air cool.
- 6.2 Anneal the B specimens from 5.2 at 1500F for 15 minutes. Air cool 1 and 2; water quench 3 and 4.
- 6.3 Repeat 6.2 with C specimens using 1600F anneal.
- 6.4 Repeat 6.2 with D specimens using 1700F anneal.
- 6.5 Repeat 6.2 with E specimens using 1750F anneal.
- 6.6 Repeat 6.2 with H specimens using 1800F anneal.
- 6.7 Repeat 6.2 with K specimens using 1900F anneal.
- 6.8 Repeat 6.2 with L specimens using 2000F anneal.
- 6.9 Repeat 6.2 with M specimens using 2100F anneal.
- 6.10 Repeat 6.2 with P specimens using 2150F anneal.

PAGE	 37
REPORT	 A470

TEST REQUEST NO. 513-336

7.0 METALLOGRAPHIC EXAMINATION

- 7.1 Send all specimens identified with 1's and 3's to L. Flanigan, Dept. 272, Bldg. 33.
- 7.2 Section and prepare for metallographic examination by electrolytic etching only, all specimens identified with 2's and 4's.
- 7.3 Prepare photomicrographs (highest magnification available) of all mounts from 7.2 showing the microstructure resulting from each thermal treatment.
- 7.4 Determine grain size of all specimens from 7.2 in accordance with ASTM standards.
- 7.5 Compare the relative quantities of grain boundary precipitate observed in all specimens from 7.2 using the over-aged sample (A2) as a reference. (Use optical metallographic technique)
- 7.6 Compare the relative quantities of intragranular precipitate of all specimens from 7.2 using the overaged sample (A2) as a reference.
- 7.7 Resolve and compare the precipitated phases as completely as possible using optical metallographic techniques.

8.0 DATA REQUIRED

- 8.1 Actual heat treat cycles
- 8.2 Photomicrographs of all specimens examined
- 8.3 Grain size of all specimens per ASTM standards
- 8.4 Relative quantities of precipitates as compared to the "over-aged only" specimen (A2), percent remaining
- 8.5 Comparison of precipitated phases