

Effect of Heat Treatment Variations on the Hardness and Mechanical Properties of Wrought Inconel 718

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Abstract

The effect of heat treat variations within current specification tolerances on the hardness of wrought Inconel 718 was evaluated. The statistically designed experiment identified cooling rate from solution heat treatment as having the greatest impact on hardness with increasing cooling rate increasing fully heat treated hardness. A specimen test program was then initiated to determine the effect of fast and slow cooling rates on mechanical properties. Slower cooling rates resulted in reduced creep rupture and yield strengths with no effect on low cycle fatigue and fatigue crack growth properties. After thermal exposure (100 hours at 732°C) both sets of material exhibited comparable strength and stress rupture properties.

Introduction

Wrought Inconel 718 has evolved into a workhorse alloy for turbine engine applications for many reasons including affordability, weldability and a good balance of high temperature mechanical properties. However, some of the characteristics desirable for engine applications can result in producibility difficulties. If processed to increased hardness, the alloy can be extremely difficult to machine (ref 1). In addition, complex, welded assemblies can be prone to distortion when rapidly cooled from the solution heat treat cycle. To control distortion, the use of slower cooling rates is pursued, but there is frequently very little margin relative to specification requirements to air cool the alloy (AMS 5663).

The use of statistically designed experimental matrices has proved to be an efficient technique in material process development (ref 2). They permit the screening of a large number of process variables, and when combined with a confirmation step, result in identification and control of the critical processing variables. The Taguchi based approach to experimental design (ref 3) has proven to be extremely valuable. This approach was utilized to assess the sensitivity of Inconel 718 hardness on variations that can occur in the heat treatment of Inconel 718. The processing steps in the heat treat cycle and levels of variation allowed by the specification are presented in Table I. Material hardness - R_C (Rockwell C) was selected as the response variable for the experiment.

Table I
AMS 5663 Specification Heat Treatment Variables and Typical Ranges

| Heat Treat Step | AMS5663 | Typical Range |
|---|----------------|----------------|
| Solution Temperature | 954°C/1 hour | +/- 14°C |
| Cooling Rate | Air Cool | Not Controlled |
| Heat Up Rate to Age Cycle | Not Controlled | Not Controlled |
| 1 st Precipitation Temperature | 722°C | +/- 14°C |
| 2 nd Precipitation Temperature | 621°C | +/- 14°C |

Details

A heat of AMS 5662 was obtained (heat PLNJ) and sectioned into heat treat cubes. The chemistry for the heat is

Table II
Chemistry of AMS 5663 Heat PLNJ used for The Heat Treatment Processing Study.

| Element | Weight % |
|---------|----------|
| Cr | 18.2 |
| Fe | 17.7 |
| Mo | 3.1 |
| Nb + Ta | 5.24 |
| Ti | .79 |
| Al | .36 |
| C | .07 |
| Si | .22 |
| B | < .003 |
| S | .013 |
| Co | .097 |
| Ni | Bal |

presented in Table II. A Taguchi L8 experimental matrix was selected for the experiment and the variables were assigned to assess the main effect and some of the interactions of the factors listed in Table I. Details of each experimental run are presented in Table III. After heat treatment, the material was metallographically prepared and direct Rockwell C hardness tests conducted (5 per sample). Hardness results are presented in Table IV. Review of the hardness results shows very little scatter within each run with a range of up to 2.6 Rc between the lowest and highest hardness runs. Statistical analysis of the hardness results identified all factors except solution

Table III
Heat Treatment Processing Parameters¹ for Taguchi L8 Experimental Runs

| Run # | Solution Temp | Cooling Rate | 1 st Precipitation | 2 nd Precipitation | Heat Up Rate |
|-------|---------------|----------------|-------------------------------|-------------------------------|--------------|
| 1 | 941°C | <6°C / minute | 704°C | 607°C | 278°C / hour |
| 2 | 941°C | <6°C / minute | 732°C | 635°C | 556°C / hour |
| 3 | 941°C | >22°C / minute | 704°C | 635°C | 556°C / hour |
| 4 | 941°C | >22°C / minute | 732°C | 607°C | 278°C / hour |
| 5 | 968°C | <6°C / minute | 704°C | 607°C | 556°C / hour |
| 6 | 968°C | <6°C / minute | 732°C | 635°C | 278°C / hour |
| 7 | 968°C | >22°C / minute | 704°C | 635°C | 278°C / hour |
| 8 | 968°C | >22°C / minute | 732°C | 607°C | 556°C / hour |

1) Material processed as follows:

Solution heat treat at temperature listed followed by cooling at the rate listed.

Heat to the 1st precipitation temperature listed at the heat up rate listed and hold for 8 hours.

Furnace cool at 56°C/hour to the 2nd precipitation temperature listed and hold for 8 hours.

Air cool to room temperature

Table IV
Measured Hardness for the Heat Treatment Runs Listed in Table III.

| Run # | Hardness (Rockwell C) | |
|-------|-----------------------|------------------------------|
| | Average | Individual Readings |
| 1 | 41.5 +/- .2 | 41.3, 41.3, 41.5, 41.5, 41.8 |
| 2 | 40.6 +/- .2 | 40.4, 40.4, 40.6, 40.8, 40.9 |
| 3 | 42.3 +/- .1 | 42.2, 42.2, 42.4, 42.4, 42.5 |
| 4 | 41.7 +/- .4 | 41.2, 41.6, 41.7, 42, 42.2 |
| 5 | 41.1 +/- .3 | 40.8, 40.9, 41.1, 41.3, 41.5 |
| 6 | 40.3 +/- .3 | 39.9, 40.1, 40.5, 40.5, 40.6 |
| 7 | 42.6 +/- .3 | 42.3, 42.4, 42.6, 42.9, 42.9 |
| 8 | 42.9 +/- .3 | 42.5, 42.6, 42.9, 43.1, 43.2 |

temperature and heat up rate to the precipitation temperature as having a statistically significant effect on fully heat treated hardness. Of the factors identified, cooling rate from solution heat treatment had the greatest effect (1.5 R_C difference) with less than .5 R_C difference between the low and high levels (Table V) for the other variables.

Table V
Average Hardness at the Low and High Levels
for the Experimental Factors Evaluated in Table III.

| Variable | Low Level | High Level |
|---|---------------------|---------------------|
| Solution Temperature | 41.6 R _C | 41.7 R _C |
| Cooling Rate | 40.9 R _C | 42.4 R _C |
| Heat Up to Age Cycle | 41.6 R _C | 41.7 R _C |
| 1st Age Temperature | 41.9 R _C | 41.4 R _C |
| 2nd Age Temperature | 41.9 R _C | 41.5 R _C |
| Solution Temp / Cooling Rate ¹ | 41.4 R _C | 41.9 R _C |
| Solution Temp / 1st Age Temp ¹ | 41.5 R _C | 41.8 R _C |

1) Interaction between the variables listed.

Two verification heat treatments were defined to confirm the effects of the factors identified; the first was aimed at maximizing hardness (“hard” run) with the second aimed at minimizing hardness (“soft” run). The two cycles are compared in Table VI. Hardness testing (Table VII) verified the expected effect of the heat treat cycles with a difference of slightly more than 2 R_C measured between the two processes. To further verify the processing effects, the material processed through the hard heat treat was re-heat treated to the soft cycle. Hardness testing showed a 2 R_C reduction relative to the initial hard heat treatment results. The two heat treat cycles were also verified in a shop furnace with test panels processed through both the hard and soft heat treat cycles. A difference of almost 3 R_C was observed between the two cycles in the shop trials.

Table VI
Confirmation Heat Treatment¹ Process Parameters.

| Run # ² | Solution Temp | Cooling Rate | 1 st Precipitation | 2 nd Precipitation | Heat Up Rate |
|--------------------|---------------|----------------|-------------------------------|-------------------------------|--------------|
| soft | 968°C | <6°C / minute | 732°C | 635°C | 278°C / hour |
| hard | 941°C | >22°C / minute | 704°C | 607°C | 556°C / hour |
| soft ³ | 968°C | <6°C / minute | 732°C | 635°C | 278°C / hour |

1) See Table III note 1.

2) Soft run aimed at minimizing hardness and hard run aimed at maximizing hardness.

3) Material originally processed through hard parameters than re-heat treated to soft process parameters.

Table VII
Measured Hardness for Material Processed Through Heat Treatment Cycles in Table VI

| Run # ¹ | Hardness (Rockwell C) | |
|--------------------|-----------------------|------------------------------|
| | Average | Individual Readings |
| soft | 41.2 +/- .1 | 41.1, 41.1, 41.2, 41.2, 41.3 |
| hard | 43.4 +/- .3 | 43, 43.1, 43.4, 43.7, 43.7 |
| soft ² | 41.4 +/- .4 | 40.8, 41.4, 41.4, 41.7, 41.8 |

1) See Table VI note 2.

2) See Table VI note 3.

Because cooling rate from solution heat treat appeared to be the most important factor, a series of cooling rate trials were conducted to study the impact on fully heat treated (per AMS 5663) hardness. The results are presented in Table VIII and show a strong trend between hardness and cooling rate.

Table VIII
Effect of Cooling Rate from Solution Heat Treatment on Inconel 718 Hardness.
(Balance of Heat Treatment per AMS 5663 Nominal Levels)

| Cooling Rate from Solution | Average | Range of Data |
|----------------------------|---------------------|---------------|
| 111°C / hour | 39.6 R _C | 38.9 to 40.2 |
| 333°C / hour | 40.3 R _C | 39.9 to 40.9 |
| 666°C / hour | 40.9 R _C | 40.2 to 41.4 |
| >1111°C / hour | 42.3 R _C | 42 to 43.2 |

While variations in heat treat temperatures can be observed, it is far more likely to experience a variation in cooling rate. In some instances slower cooling rates are used to minimize part distortion. Because of this and the results from the designed experiments, the effect of slower cooling rates from solution heat treatment on Inconel 718 properties was evaluated. A ring case was obtained and sectioned into 2 segments. A hole was drilled into each segment for measurement of cooling rates during heat treat. Both segments were heat treated in a vacuum furnace with the air cooled segment cooled by backfilling the chamber with Argon. The slower cooled segment used a programmed cooling rate of 11°C / minute. A comparison between the two heat treat cycles is presented in Figure 1. Cooling rates ranged from 11°C / minute to 28°C / minute. Typical microstructures from each of the heat treat cycles are presented in Figure 2. No evidence of overaging is observed in the slower cooled segment. Hardness testing showed about a 1.5 R_C difference in hardness between the two pieces.

Table IX
Specimen Test Matrix used to Characterize the Effect of Cooling Rate
from Solution Heat Treatment on the Mechanical Properties of AMS 5663

| Property | Conditions | # of Specimens | |
|------------------------------|--------------------------------|-------------------|-------------------|
| | | 11°C/minute | 28°C/minute |
| Tensile | 20°C, 371°C, 649°C | 5 per temperature | 3 per temperature |
| Stress Rupture | 649°C/689.5 MPa | 5 | 3 |
| Creep | 621°C/689.5 & 724 MPa | 1 per stress | 1 per stress |
| Notch LCF ¹ | 371 °C & 593°C/1241 MPa | 4 per temperature | 4 per temperature |
| Fatigue Crack Growth | 371 °C & 593°C | 1 per temperature | 1 per temperature |
| Stability (100 hrs at 732°C) | 649°C Tensile & Stress Rupture | 3 per test | 2 per test |

1) Tested at a concentrated stress of 1241 MPa.

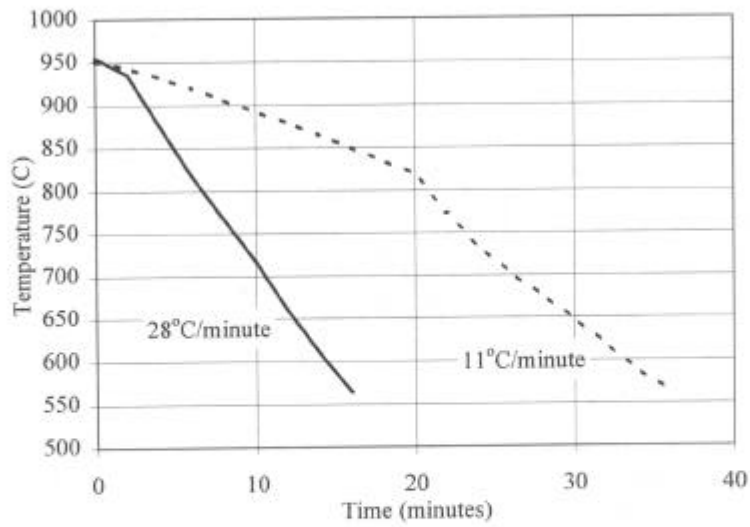
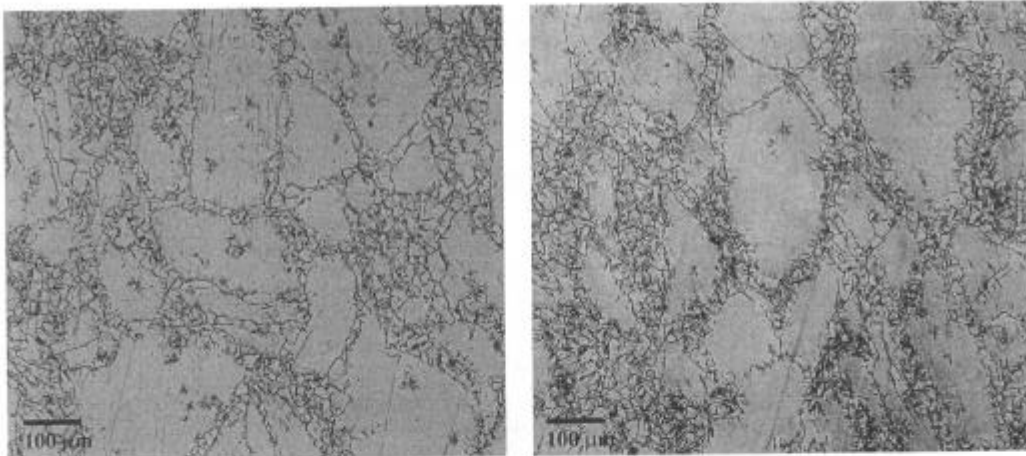


Figure 1
Cooling Rate Curves for AMS 5663 Material Processed for this Test Program



Cooled 28°C/Minute

Cooled 11°C/Minute

Figure 2
Typical Microstructures Observed in Wrought Inconel 718
Cooled at 28°C / Minute and 11°C / Minute from Solution Heat Treatment.
Etch 10% Oxalic Acid

Tensile, combination (smooth/notch) stress rupture, notched LCF, fatigue crack growth and exposure testing was conducted on both ring segments. The test matrix used is presented in Table IX.

Table X
Effect of Cooling Rate from Solution Heat Treatment on the Tensile Properties of AMS 5663

| Temp | Yield Strength (MPa) | | UTS (MPa) | | EL (%) | | RA (%) | |
|-----------------------------|----------------------|--------|-----------|--------|--------|--------|--------|--------|
| | 28°C/m | 11°C/m | 28°C/m | 11°C/m | 28°C/m | 11°C/m | 28°C/m | 11°C/m |
| 20°C | 1103.9 | 1048 | 1296.9 | 1296.3 | 17 | 18 | 23.9 | 23.4 |
| 371°C | 992.9 | 954.3 | 1136.3 | 1137.7 | 17.3 | 18.4 | 29.2 | 26.3 |
| 649°C | 916.3 | 882.6 | 1055.6 | 1051.9 | 19.8 | 20.8 | 37.6 | 38.1 |
| AMS 5663 ¹ 20°C | 1034 | | 1241 | | 10 | | 12 | |
| AMS 5663 ¹ 649°C | 862 | | 965 | | 10 | | 12 | |

1) AMS 5663 specification requirements.

Tensile results are summarized in Table X. The slower cooling rate resulted in reduced yield strength and equivalent ultimate strength and ductilities. Both cooling rates achieved AMS 5663 specification minimums. The reduced yield strength suggests that the material is experiencing overaging as a result of the slower cool from solution heat treat.

The combination (smooth / notch) stress rupture testing was conducted at the specification conditions of 649°C / 689.5 MPa. The results are presented in Table XI. All specimens failed in the smooth section and conformed to AMS 5663 requirements. The slower cooled material exhibited about a 25% decrease in rupture life with a 50% improvement in rupture elongation. Creep testing was also conducted at slightly lower temperatures and the results are summarized in Table XI. Similar to the stress rupture results a 25% to 40% reduction in creep life was observed for the slower cooled material.

Table XI
Effect of Cooling Rate from Solution Heat Treatment
on the Creep Rupture Properties of AMS 5663

| Cooling Rate | 649°C / 689.5 MPa | | 621°C / 689.5 MPa | 621°C / 724 MPa |
|-----------------------|-------------------|--------|-------------------|-----------------|
| | Life (hrs) | EL (%) | Hours to .5 % | Hours to .5% |
| 28°C/ minute | 184.7 | 7.3 | 545 | 313 |
| 11°C/ minute | 136.8 | 10.4 | 420 | 222 |
| AMS 5663 ¹ | 23 | 4 | | |

1) AMS 5663 specification requirements.

Notched LCF testing was conducted at a concentrated stress of 1241 MPa at temperatures of 371°C and 593°C. The results are presented in Figure 3. The slower cooled material exhibited fatigue capability equivalent to the air cooled material at lower temperatures and slightly better at higher temperatures.

Fatigue crack growth testing was conducted at both 371°C and 593°C. The results are presented in Figure 4. There was no difference in fatigue crack growth rates between the two cooling rates.

To determine if the use of a slower cooling rate from solution heat treatment resulted in overaging, the effect of thermal exposure on tensile and stress rupture properties was evaluated. Fully heat treated material was processed through a 732°C / 100 hour isothermal exposure. Tensile and stress rupture specimens were then machined and tested. A comparison between the exposed and unexposed results for the two cooling rates is presented in Table XII. The results show no difference in exposed tensile capability between the air cooled and slow cooled material. The exposed stress rupture capability of the slow cooled material was improved over the unexposed but was still only 90% of the exposed air cooled material levels. Metallographic characterization showed both the slow cooled and air cooled material to exhibit an overaged structure after the 732°C exposure (Figure 5). The testing suggests that both materials overaged to the same extent.

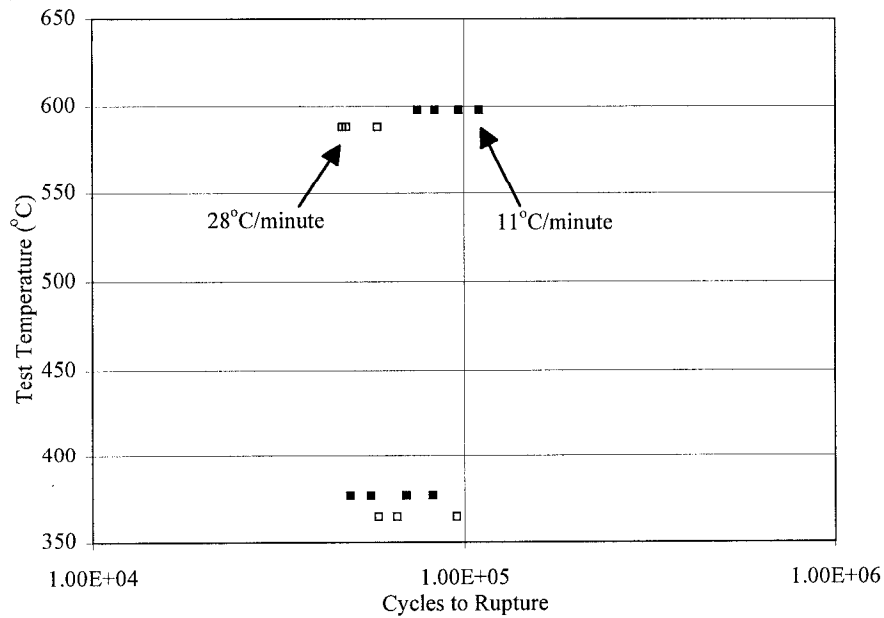


Figure 3
 Notched Low Cycle Fatigue Properties of Wrought Inconel 718 Cooled at 11°C / Minute or 28°C / Minute from Solution Heat Treatment. Specimens Tested at a Concentrated Stress of 1241 MPa at 317°C and 593°C.

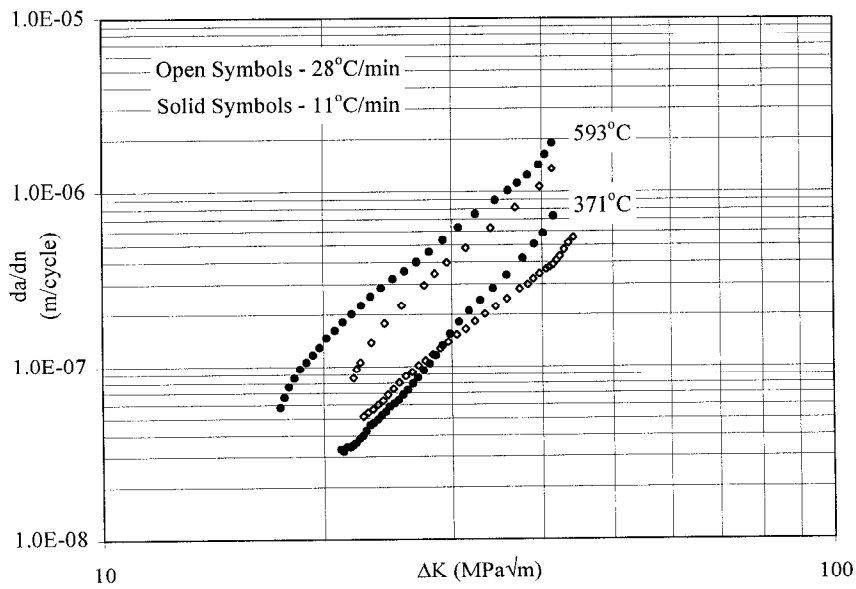


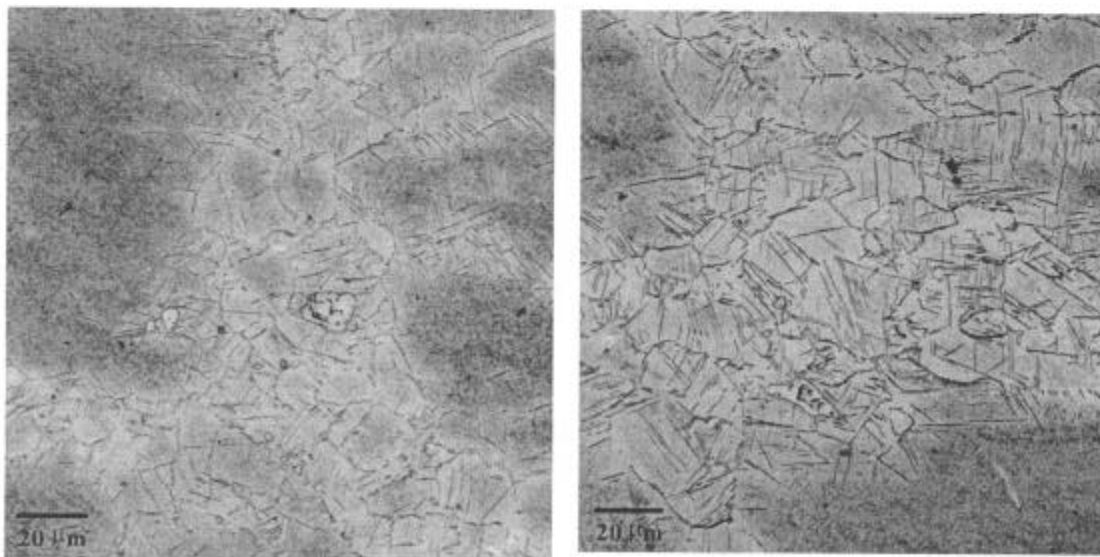
Figure 4
 Fatigue Crack Growth Rate of Wrought Inconel 718 Cooled at 11°C or 28°C from Solution Heat Treatment

Table XII
Effect of Cooling Rate from Solution Heat Treatment and Isothermal Exposure on the Tensile and Creep Rupture Properties of AMS 5663

| Cooling Rate | 649°C Tensile | | | | | | 649°C / 689.5 MPa Stress Rupture | | | |
|--------------|------------------|--------------------|-----------|--------|--------|-------|----------------------------------|-------|--------|-------|
| | YS (MPa) | | UTS (MPa) | | EL (%) | | Life (hours) | | EL (%) | |
| | Exp ¹ | Unexp ² | Exp | Unexp | Exp | Unexp | Exp | Unexp | Exp | Unexp |
| 28°C/m | 873.6 | 916.3 | 1076.5 | 1055.6 | 22 | 19.8 | 196 | 184.7 | 8 | 7.3 |
| 11°C/m | 861.9 | 882.6 | 1077.7 | 1051.5 | 23 | 20.8 | 170.3 | 136.8 | 11 | 10.4 |

1) Exposed 100 hours at 732°C prior to specimen machining and test.

2) Baseline results from this test program.



Cooled 28°C/Minute

Cooled 11°C/Minute

Figure 5

Microstructure of Inconel 718 Cooled at 11°C/Minute or 28°C/Minute from Solution Heat Treatment, Fully Heat Treated and then Exposed 100 Hours at 732°C. Both Structures Exhibit Evidence of Overaging.

Discussion

The results of this test program indicate that the mechanical properties of Inconel 718 are affected by heat treatment at the extremes of currently allowed specification limits, particularly by the cooling rate from solution heat treatment. Slower cooling rates resulted in reduced hardness and monotonic properties relative to faster cooled material. The results suggest that the slower cooling rate is producing a slightly overaged structure not readily observed by optical metallography. After extended thermal exposures both cooling rates appear to exhibit similar amounts of overaging. Consistent with other experience the use of statistically based experimental matrices provides an efficient process development tool. In the course of this work hardness proved to be an effective screening measurement of the impact of processing on material properties.

References:

- 1) J. J. Schirra & D. V. Viens; "Metallurgical Factors Influencing the Machinability of Inconel 718"; *Superalloys 718, 625, 706 and Various Derivatives*; (1994), Ed by E. A. Loria; pgs 827-839.
- 2) J. J. Schirra & R. W. Hatala; "Development of a Damage Tolerant Heat Treatment for Cast + HIP Incoloy 939"; *Superalloys 1996*; (1996), Ed by Kissinger et al; pgs 137-145.
- 3) G. Taguchi; *Introduction to Quality Engineering*; American Supplier Institute - Dearborn, MI; 1986