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METALLURGICAL EVALUATION OF WELDING AND HEAT TREATING PROCEDURES FOR THE INCONEL 718 PBF IN-PILE TUBE

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The solving of the problems encountered during fabrication of the in-pile tube was a coordinated effort between the Metallurgy and Hot Cell Branch, Power Burst Facility Program Operations of Aerojet Nuclear Company (ANC) and the tube fabricator, Standard Press Steel Company.

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ABSTRACT

Problems were encountered during welding and heat treating qualification testing and fabrication of the Power Burst Facility in-pile tube which was made of Inconel 718. Areas of localized low ductility in the welds were detected by bend testing. Solution heat treat temperatures high enough to dissolve and redistribute the Laves phase in the welds resulted in inadequate parent metal strength.

To solve the problems, the welds were given a higher localized solution heat treatment (1 hour at 2025°F) and the entire assembly was then solution annealed 1 hour at 1925°F and duplex aged, at 1325°F and 1150°F, for a total of 42 hours.

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SUMMARY

Although much information has been reported in the literature about the welding and heat treating of Inconel 718, problems were encountered when the Power Burst Facility in-pile tube was fabricated from this material.

Inconel 718 is precipitation hardened by body centered tetragonal $\text{Ni}_3(\text{Cb}, \text{Ti}, \text{Al})$ called gamma prime. The precipitation of the gamma prime is sluggish which reduces the tendency to strain age crack. Alloy 718 is said to be one of the most readily weldable of the precipitation hardened nickel-base alloys. However, it has a large number of alloying elements and is metallurgically complex.

The literature indicates that an 8% elongation in weldments (a requirement for the in-pile tube) which have been solution heat treated at 1925F followed by standard duplex aging, should easily be obtainable. Indeed, these values were attainable using a one inch gauge length tensile test, but when standard 5T bend tests with longitudinal welds (which results in deformation of a relatively long portion of weld metal) were tested, localized areas of Laves phase embrittlement caused pre-mature fracture at a bend radius as low as 30 degrees. Hence, the first problem encountered was inadequate localized ductility in the weld metal.

Higher solution heat treat temperatures are known to improve ductility in the welds, but for this particular heat of Inconel 718 difficulty was experienced in achieving adequate strength for the main tube, 150 ksi yield, even with the 1925F solution heat treat temperature. So the second problem encountered was inadequate strength in the parent metal.

The problems were solved by giving the welds a separate localized solution heat treatment at 2025°F before giving the entire assembly a 1925°F solution heat treatment followed by longer than the normal aging times. The assembly was first aged for the recommended 8 hours at 1325°F furnace cooled to 1150°F and held at 1150°F for 8 hours. When this proved to be inadequate the assembly was re-aged for 8 hours at 1325°F, furnace cooled to 1150°F and held at 1150°F for 14 hours to give a total age plus re-age time of 42 hours. Since the welds were located on the inlet and outlet nozzles which were only required to have a yield of 127 ksi, the higher localized solution heat treatment of the welds was tolerable. The final minimum yield strength achieved for the main tube was 160 ksi.

I. INTRODUCTION

The Power Burst Facility (PBF) in-pile tube (IPT) is a cylindrical pressure vessel designed for use in the PBF reactor under transient conditions. The basic functions of the IPT are to provide a means of transporting experiments from the canal to the reactor and to provide the temperature, pressure, flow, coolant chemistry, and nuclear environment necessary for experimental programs. The overall configuration of the tube is shown in Figure 1. There is an inlet and an outlet nozzle which were welded to the main tube. The design requires a minimum of 150 ksi yield strength in the main tube and 127 ksi yield strength in the nozzle with at least 8% elongation in both. Because of thermal stress considerations and space available the main tube was designed to 0.9 of the required yield strength. The purpose of this investigation was (1) to evaluate the metallurgical condition of the nozzle weldments and main tube as a result of failures in the welder qualification bend tests and (2) to conduct tests and make recommendations concerning heat treatments and welding procedures that would insure adequate strength and ductility.

The material selected for the IPT and nozzles is an age-hardenable nickel-chromium alloy called Inconel 718. It has been known that⁽¹⁾ impact strength and ductility in welds of this material can be improved by the use of higher post weld solution heat-treatments but that higher solution heat-treatments result in lower strength. The metallurgical problem encountered was to obtain adequate strength in the main tube without the loss of ductility in the nozzle welds.

The nozzles and IPT both come from the same heat of material. Their composition and that of the weld rod are shown in the accompanying table:

TABLE I

CHEMICAL ANALYSIS									
	C	Mn	P	S	Si	Cr	Ni	Ti	Mo
IPT & Nozzle	.05	.06	.006	.007	.20	17.82	54.48	.95	2.86
Weld Rod	.04	.07	.005	.002	.14	17.84	53.27	1.06	3.17
	Al	Co	Cb	Fe	Cu	B			
IPT & Nozzle	.48	.04	4.96	18.40	.06	.0038			
Weld Rod	.54	.11	5.10	18.03	.04	.0035			

Ingots of this material were forged in various steps starting at furnace temperatures of 2050°F and ending at 1900°F resulting in maximum reductions from an 18" square to a 10" octagon. What was to be the upper portion of the tube was forged to a 11 1/2" octagon. A bulk head 11 1/2" x 17" was formed where the nozzles were to be attached.

Samples from the forgings and similar heats of material had been welded and heat treated to produce material qualification test samples to verify a single solution heat treatment of 1925°F followed by double aging at 1325°F and 1150°F. Some of the bend tests of these welded single solution heat treated materials fractured indicating a problem with low localized weld ductility. This report contains information obtained from the examination of the failed bend tests, plus the evaluation of various welding and heat treating procedures which led to a solution to the problems. It also discusses what was done when tensile specimens, which were attached to the tube during its actual heat treatment, did not develop the required yield strength values.

II. LOCALIZED WELD EMBRITTLEMENT

When a series of welder and material qualification tests were run on the Inconel 718 material to be used for the IPT, two of the face 5T bend tests produced by one welder using TIG, which contained welds longitudinal to the sample, fractured completely showing low ductility. One sample fractured after a 30 degree bend and the other after 120 degrees. Other TIG face bend samples by another welder passed the test with no failure after a 180 degree bend. Calculations show that if such a bend test would be completed to an angle of 180 degrees the outside fiber would have an elongation of about 9%.

Although weld metal tensile samples, 8 transverse and 4 longitudinal, were pulled before the bend tests were run the problem of low localized ductility was not detected*. The transverse samples all broke in the parent metal exhibiting elongations of 10 to 13%. The longitudinal samples exhibited elongations ranging between 18 and 21%. The bend test served the purpose of exposing a long section of weld metal to deformation so that areas of localized low ductility could be detected. Such areas were not detected in tensile samples with 1" long gauge lengths.

A material examination was performed which mechanically and metallogically compared the 30 degree bend sample to the 180 degree sample. An incipient crack in the 120 degree sample well away from the main fracture was also investigated. Samples of both the 30 degree material and the 180 degree material were made into tensile and impact specimens to check for differences in mechanical properties. It was found that variations in the amount of an interdendritic phase, called Laves phase, existed within the 30 degree and 120 degree material and to a lesser extent in the 180 degree material. In areas of high concentration (at fractured faces and incipient cracks) the Laves phase is stringered along the dendritic grain boundaries, providing a path of reduced strength, reduced ductility and reduced toughness.

*See appendix "A".

Figure 1 - Longitudinal section of the PBF -In-Pile Tube.

1. FRACTOGRAPHY AND METALLOGRAPHY OF FAILED BEND SAMPLES

The macrophotography of the fractured face of the 30 degree bend specimen in Figure 2 shows a symmetrical pattern about a point on the outside fiber of the bend near the center of the weld face, suggesting the fracture originated in this region. There was no indication of a large inclusion or excessive porosity at this location or other areas of the fractured surface. Examination of the fractured surface at higher magnifications in the scanning electron microscope (SEM) (Figures 3 and 4) revealed a channeling or coring effect on the fractured face which produced striations at an interval of about 0.0004". At higher magnifications cracked particles of the Laves phase can be seen embedded in the fracture surface. Figure 5, a longitudinal metallographic section which included the face of the weld as one edge and the fractured surface as another edge, and Figure 6 at higher magnification, show that the fractured face follows the Laves phase stringering.

Metallographic viewing, as well as extensive SEM observation, has shown that the fractures which occurred in the 30 degree and 120 degree samples, causing complete separation, were associated with an area of oriented Laves-phase stringering. This stringering consistently retained its orientation in the various weld beads and was roughly perpendicular to the weld face and perpendicular to the axis of the weld.

A similar effect is seen in Figures 7 and 8 in an incipient crack from the 120 degree sample. The difference between this and the fractured area of the samples is that the Laves stringers do not continue through the material. They existed only in the surface weld beads. The crack terminated in the recrystallized material just beyond where the stringers terminated.

Although the 180 degree bend test samples exhibited small areas of stringered Laves phase, consistent orientation from weld bead to weld bead was not found and the quantity of Laves phase was much less. A typical longitudinal metallographic view of the 180 degree sample is shown in Figure 9.

The ASTM standard grain size shows considerable variation in these materials and ran from medium to coarse, or size 4-5 for the 180 degree weld metal, and size 3-5 for the fractured weld metal sample. The only evidence of microcracking was associated with the Laves phase in stressed areas.

2. MECHANICAL PROPERTIES OF BEND TEST MATERIAL

Four tensile samples and four Charpy V impact samples were prepared from the bend test weld metal material. The results are shown in Table II.

TABLE II

MECHANICAL PROPERTIES OF BEND TEST MATERIAL

Sample Designation	Yield Strength psi	Ultimate Strength psi	% Elongation in 1"	R.A. %	Charpy V ft. lb.
30° (A)	-----	-----	---	--	19.8
30° (B)	153,000	173,000	4.5	8	---
30° (C)	155,000	188,000	20	26	---
30° (D)	-----	-----	---	--	22.2
180° (A)	165,000	197,000	22	33	---
180° (B)	-----	-----	---	--	26
180° (C)	160,000	192,000	20	39	---
180° (D)	-----	-----	---	--	27

The 30 degree samples which had the more extensive, stringered and oriented Laves phase also had the poorer strengths, elongation, reduction of area and impact properties. An acceptable sized inclusion, 80 mils, was observed on the fractured face of one of the tensile samples (30°B) which showed a 4.5% elongation. The 180 degree samples by comparison which had less Laves phase had more consistent and better properties.

The fractography and metallography of the tensile and impact samples produced from the 30 degree bend test material Figures 10, 11 12, and 13 again show the effects of larger quantities of Laves phase compared to the 180 degree material Figures 14 and 15. It had been observed throughout the metallographic examinations that areas of high Laves phase did not show recrystallization. A special tinting etch was employed to illustrate this effect, Figure 16, on the impact specimens.

3. DISCUSSION OF LOCALIZED WELD EMBRITTLEMENT

There is considerable discussion in the literature about Laves phase in Inconel 718^(2, 3, 4), its effect on strength, ductility, toughness and what promotes its formation and how to get rid of it. It is reported that Laves phase in castings is not affected by heat

treatment below 2100°F. Weldment ductilities are known to be benefited by a factor of 3, by lowering welder heat inputs by a factor of 2 before a 1900°F solution anneal and double age. Also heat treatments of 1900°F and above show improvement in impact strength. A pseudobinary diagram (4), Figure 17, shows the relationship between columbium plus tantalum content and the formation of Laves phase as a function of temperature. From this diagram one would expect that the higher the columbium content and the slower the cooling rate from the melt to ~2100°F the greater is the amount of Laves phase produced.

One would expect that some combination of low heat input during welding to promote rapid cooling and/or solution heat treat temperatures in excess of 1900°F, but below 2100°F, would have been adequate to insure the minimum 8% elongation. Even though strength is a function of columbium content, the amount of work in an ingot, and aging temperatures (1400 vs 1325 and 1200 vs 1150), one might also have expected that adequate strength, 150 ksi, yield could be combined with adequate ductility at some point in this solution heat treat range. In general, this initial attempt wherein the welds were solution heat treated at 1925°F and aged at 1325°F for 8 hours and 1150°F for 8 hours (furnace cooling between the two aging temperatures) met these requirements when evaluated by tensile test results. However, the bend test evaluation showed that there were isolated areas of stringered Laves phase providing a path of weakness and low ductility and that the stringered Laves phase was associated with areas where recrystallization had not occurred during heat treatment of the welds. The welds produced by one of the welders (whose designation was W-2) showed almost complete recrystallization while the welds of the other welder (whose designation was W-4) using nominally the same technique retained extensive dendritic areas. Since a stringer-free microstructure was not reliably produced, it was elected to undergo a weld and heat treating procedure evaluation to characterize these particular heats of material, if possible overcome the human factor, and the interaction between the two.

Macrophotography
3X
Figure 2 - Macrophotograph of fractured surface of the 30 degree
bend test. The point of fracture origin is indicated (arrow).

SEM Fractography

200X

Figure 3 - Scanning electron microscope fractography of fractured surface of 30 degree bend test.

SEM Fractography

1000X

Figure 4 - Scanning electron microscope fractography of fractured surface of 30 degree bend test at higher magnification.

*HCL-Methanol Etch

50X

Figure 5 - Photomicrograph of fractured 30 degree bend test material. The section is longitudinal to the weld and roughly perpendicular to the fractured surface. The arrow indicates the fractured surface.

*10% HCl in Methanol

HCl-Methanol Etch

500X

Figure 6 - Higher magnification view of fractured surface shown in Figure 5. The fracture followed the Laves phase stringers, arrow.

HCl Methanol Etch

50X

Figure 7 - Incipient crack from the 120 degree sample.

HCl-Methanol Etch

500X

Figure 8 - Higher magnification of incipient crack shown in Figure 7. The fracture followed the Laves phase stringers.

HCl Methanol Etch

500X

Figure 9 - Typical metallographic structure of the 180 degree bend sample at the face of the weld.

Macrophotograph

~ 10X

Figure 10- Fractured surface of tensile specimen (30°B) containing the 80 mil inclusion. Striations resulting from Laves phase stringering is evident.

HCl-Methanol Etch

500X

Figure 11 - Metallography of tensile specimen (30°C), transverse to the dendrites and including the fractured surface. Note the microcracking at the Laves stringers, arrow.

Macro photograph

~8X

Figure 12 - Fractured surface of impact sample (30°A).

HCl-Methanol Etch

50X

Figure 13 - Metallography including the fractured surface of (30°A) impact sample.

Macro photograph

~8X

Figure 14 - Fractured surface of impact sample (180°D).

HCl-Methanol Etch

50X

Figure 15 - Metallography including the fractured surface of (180°D) impact sample.

HCl-Methanol Tint
Sample 30°A

100X

HCl-Methanol Tint
Sample 180°D

100X

Figure 16 - Metallography of impact samples showing degree of recrystallization using $K_2S_2O_5$ as a tinting agent in the etchant.

Figure 17 - Pseudobinary diagram for the formation of Laves phase.

III. WELDING PROCEDURES

In order to determine why the W-4 welds performed differently from the W-2 and to determine what was necessary to produce a consistently good weld, the history of previous welding procedures was reviewed, and a new series of carefully monitored test weld procedures was evaluated.

1. HISTORY OF BEND TEST WELDS

A review of the previous welding techniques suggests that the W-4 welds had higher heat inputs, since this welder required more total weld time which included cleaning and grinding between passes. The W-4 and W-2 welds were made with separate machines but the same make and model number (Miller #330A/BP). Both used about 20 cubic feet per hour of argon purge gas and both used about 145 amps for the first pass (with 0.050" weld filler rod) and 155 amps for subsequent passes (3/32" filler rod). Both welders used a small carbide cone-shaped burning tool followed by a power driven stainless wire brush to clean between passes. Tempil sticks were used to insure the interpass temperatures did not exceed 350°F.

2. TEST WELD PROGRAM

An additional objective of the test welds was to determine if the Laves phase formation could be minimized by controlling those weld parameters which affect cooling rates.

To investigate the possibilities, a series of six V type weld joints were prepared by different heat inputs, (1) minimum current, minimum interpass temperature (2) maximum current, maximum interpass temperature and (3) medium current and medium interpass temperature, each with two different cover gases (1) 100% argon and (2) 75% helium, 25% argon. The weld parameters for the twelve welds were monitored and are tabulated in Tables III and IV. Pre-weld and post-weld temperatures were monitored using Tempil Sticks just prior to arc ignition and within 15 seconds after completion of a pass. On the "minimum" and "medium" welds the interpass temperature was controlled to maintain a pre-weld temperature at or below 150°F and 200°F respectively. On the maximum welds no interpass temperature control was used and the temperatures varied from 280°F to 400°F. In every case but one, the W-4 welder who produced the low ductility welds had lower heat input than the W-2 welder, which is contrary to expectations.

Observations of the two welders during these tests revealed some differences in welding technique. While the W-4 welder tended to maintain a steady, unwavering welding arc, W-2 welded with a slight circular oscillation which agitated or lightly stirred the molten puddle. The W-4 welder held a closer arc-to-work distance than did W-2. The W-4 welder fed considerably more wire into the arc than did W-2 producing a higher crown on the weld beads.

It was also observed that the selection of shielding gases had an effect on such welding characteristics as penetration, pattern, and bead contour. Changes in arc-length created smaller voltage changes with argon than with the argon-helium combination, an advantage for manual tungsten-arc welding. Since argon does not have as high a conductivity as helium, the all argon cover gas produced a hot spot directly below the arc giving rise to a "U" shaped penetration bottom compared to a parabolic shape for the helium containing cover gas. The helium additions improved the weldability or flow characteristics of the weld metal making higher weld speeds possible. The resultant heat inputs were in the range of 20,000 to 40,000 joules per in. with either cover gas.

3. METALLURGICAL EXAMINATION OF TEST WELDS

Figures 18 through 23 show low magnification photomicrograph composites of the twelve welds and Figures 24 and 25 show typical high magnification photographs, all in the as-welded condition. It was found for the range of heat inputs tried in this study that the formation of Laves phase could not be suppressed. Long stringers of interdendritic Laves phase were formed during each welding procedure and by each welder. No one procedure could be selected as having less Laves phase.

Microcracking was observed in the heat affected zone in every "minimum" and "maximum" current argon cover gas weld, produced by each of the welders. Typical microcracking is shown in Figure 26. Microcracking was not observed in any of the "medium" current argon or any of the helium cover gas welds. The parent metal for all of these test welds was from the same heat of material and in the same metallurgical condition, so grain size was not a factor(5).

TABLE III

GROUP I 718 MANUAL WELD SPECIMENS
100% ARGON TORCH SHIELDING GAS

Direct Current Welding Condition	No. 1 - Min. Current Min. Inter-Pass Temp		No. 2 - Max. Current Max. Inter-Pass Temp		No. 3 - Med. Current Med. Inter-Pass Temp	
	W-4	W-2	W-4	W-2	W-4	W-2
Amperage	94	106	153	153	146	140
Arc Voltage (v)	12	11.8	13.6	14.0	13.7	14.2
Weld Pass Length (in.)	5½"	5½"	5½"	5½"	5½"	5½"
Arc Time per Pass (Sec)	89	115	108	124	96	94
Weld Travel Speed (ipm)	3.54	3.28	2.91	2.54	3.28	3.36
Weld Heat In-Put (joules per in.)	19118	27392	42971	46661	39942	34011
Weld Rod Used per Pass (in.) Weld Rod Dia = 3/32 (in.)	7.0	4 7/8	13 7/8	12 3/4	9 11/16	8 3/8
Pre-Weld Part Temp. (°F)	150	150	400	400	200	200
Post-Weld Part Temp (°F)	443	537	800	812	458	470
Shielding Gas (argon) Flow Rate (cfh)	20	20	20	20	20	20

TABLE IV

GROUP II 718 MANUAL WELD SPECIMENS
75% Helium - 25% ARGON TORCH SHIELDING GAS

Direct Current Welding Condition	No. 1 - Min. Current Min. Inter-Pass Temp		No. 2 - Max. Current Max Inter-Pass Temp		No. 3 - Med. Current Med. Inter-Pass Temp	
	W-4	W-2	W-4	W-2	W-4	W-2
Amperage	55	55	85	83	123	127
Voltage (v)	12.5	14.2	10.5	14.7	15.1	15.4
Weld Pass Length (in.)	5½	5½	5½	5½	5½	5½
Arc Time Per Pass (Sec)	257	236	149	110	89	101
Weld Travel Speed (ipm)	1.28	1.39	2.22	3.0	3.71	3.27
Weld Heat In-Put (joules per in.)	32230	33712	24121	24402	30037	36192
Weld Rod Used Per Pass (in.)	5 5/8	3 7/16	14 11/16	5 3/8	11 7/8	7 7/16
Weld Rod Dia = 3/32 (in.)						
Pre-Weld Part Temp (°F)	150	150	283	300	200	200
Post-Weld Part Temp (°F)	526	580	700	675	456	550
Shielding Gas He 75% A 25%	He 30	He 30	He 30	He 30	He 30	He 30
Flow Rate (cfh)	A 10	A 10	A 10	A 10	A 10	A 10

HCl-Methanol Etch

Welder W-2

50X

HCl-Methanol Etch

Welder W-4

50X

Figure 18 - Photomicrographs of weldments made by each welder using minimum current and argon cover gas.

HCl-Methanol Etch

Welder W-2

50X

HCl-Methanol Etch

Welder W-4

50X

Figure 19 - Photomicrographs of weldments made by each welder using maximum current and argon cover gas.

HCl-Methanol Etch

Welder W-2

50X

HCl-Methanol Etch

Welder W-4

50X

Figure 20 - Photomicrographs of weldments made by each welder using medium current and argon cover gas.

HCl-Methanol Etch

Welder W-2

50X

HCl-Methanol Etch

Welder W-4

50X

Figure 21 - Photomicrographs of weldments made by each welder using minimum current and helium cover gas.

HCl-Methanol Etch

Welder W-2

50X

HCl-Methanol Etch

Welder W-4

50X

Figure 22 - Photomicrographs of weldments made by each welder using maximum current and helium cover g.

HCl-Methanol Etch

Welder W-2

50X

HCl-Methanol Etch

Welder W-4

50X

Figure 23- Photomicrographs of weldments made by each welder using medium current and helium cover gas.

HCl-methanol etch
Minimum

Maximum

Medium

500X

Figure 24 - Typical high magnification photomicrographs of welds produced with argon covered gas at the three current settings.

Hel-methanol etch
Minimum

Maximum

Medium

500X

Figure 25 - Typical high magnification photomicrographs of welds produced with helium cover gas at the three current settings.

HCl-Methanol Etch

Argon Minimum

100X

HCl-Methanol Etch

Argon Maximum

50X

Figure 26 - Typical microcracking observed.

IV. HEAT TREATMENTS

Samples of the W-2 medium current argon and the W-4 medium current argon weldments were given one hour solution heat treatments at five different temperatures. This totaled ten samples, one of each kind for one hour at each of the following fahrenheit temperatures: 1875 $\begin{smallmatrix} +25 \\ -0 \end{smallmatrix}$, 1925 $\begin{smallmatrix} +25 \\ -0 \end{smallmatrix}$, 1975 $\begin{smallmatrix} +25 \\ -0 \end{smallmatrix}$, 2025 $\begin{smallmatrix} +25 \\ -0 \end{smallmatrix}$, and 2100 $\begin{smallmatrix} +25 \\ -0 \end{smallmatrix}$.

1. METALLURGICAL EXAMINATION

The progress of Laves phase dissolution could be detected as the temperature was increased, Figures 27, 28, and 29. It is seen that after the 2025 + 25⁰F temperature for one hour the solution of the

Laves phase was adequate, for both weld types, throughout the weld except for a surface layer, Figure 30. It is apparent that the redistribution and solution of the Laves phase is associated with recrystallization as well as solution heat treat temperatures. A layer of surface grains which did not recrystallize retained the interdendritic stringered Laves phase, while in the body of the weld where recrystallization was complete the Laves phase was no longer stringered and was greatly reduced in amount.

It was observed that there was no dissolution of the Laves phase at the 1875 + 25°F temperature in either the W-2 or W-4 samples. It

- 0

even appeared that additional Laves phase had precipitated, increasing the width of the stringers. Curiously, the W-2 sample showed extensive recrystallization after the 1925 + 25⁰F heat treat temperature while

- 0

the W-4 sample showed very little recrystallization. The W-2 sample, therefore, exhibited extensive redistribution and solution of the Laves phase compared to the other. As with the high temperature samples, the recrystallized W-2 material also had a layer of dendritic material at the weld face surface which contained the stringered Laves phase. At 1975 + 25°F both types showed extensive recrystal-

- 0

lization and again, the dendritic surface layer with the Laves phase. The 2100 + 25⁰F samples were similar to the 2025 + 25⁰F samples but with - 0 - 0 more dissolution of the Laves phase.

The thickness of the un-recrystallized layer decreased with increased heat treating temperature. At 1925°F, the W-2 sample surface layer was 0.100", the maximum at 1975°F was also 0.100" in the W-4 sample and at 2025°F the layer in the W-4 sample was down to 0.050".

2. DISCUSSIONS

The observations made during this study have contributed to our understanding of the erratic mechanical behavior experienced in the bend tests. The facts that: (1) Laves phase redistribution and solution is related to recrystallization, (2) the W-4 sample after the 1925°F solution treatment retained extensive areas of un-crystallized material, and (3) the surface layer grains in the W-2 sample after the 1925°F treatment did not recrystallize, explain why the W-4 bend samples could fracture and separate completely along the Laves phase stringers of the un-recrystallized areas and the W-2 bend samples could have incipient cracks in the un-recrystallized surface areas which would arrest in underlying recrystallized material.

In order to insure recrystallization and therefore break-up of interdendritic Laves phase stringering, solution heat treat temperatures somewhat above 1975 +25°F are desirable.

-0

Solution heat treating in the range of 2100°F or above makes the material susceptible to microcracking if weld repairs are needed and even at the 2100°F treatment an un-recrystallized surface layer contains stringered Laves phase. Solution heat treatments below 1900°F produce material which has reduced Charpy impact strength.

Because of these considerations, a localized solution heat treatment of the weld at 2025°F followed by a 1925°F heat treatment of the entire assembly, followed by double aging appeared to be a way to insure adequate ductility in the weld (8%) and adequate strength in the main tube (150 ksi yield).

HCl-Methanol Etch

W-2

50X

HCl-Methanol Etch

W-4

50X

Figure 27 - Photomicrographs of weldments made by each welder using the medium current procedure after heat treating 1 hour at 1925°F.

HCl-Methanol Etch

W-2

50X

HCl-Methanol Etch

W-4

50X

Figure 28 - Photomicrographs of weldments made by each welder using the medium current procedure after heat treating 1 hour at 1975°F.

HCl-Methanol Etch

W-2

50X

HCl-Methanol Etch

W-4

50X

Figure 29 - Photomicrographs of weldments made by each welder using the medium current procedure after heat treating 1 hour at 2025°F.

HCl-Methanol Etch

Un-recrystallized surface layer

500X

HCl-Methanol Etch

Recrystallized area

500X

Figure 30- Laves phase solution and redistribution after the 2025 + 250°F solution heat treatment.

- 0

V. FINAL QUALIFICATION OF WELDING PROCEDURES AND HEAT TREATMENTS

In order to determine if the weld could be successfully given a localized heat treatment at 2025°F prior to the 1925°F plus double age treatments to overcome the Laves phase embrittlement, a mock-up weld was made. Transverse tensile tests and impact tests in this simulated nozzle weld as well as tensile tests on parent metal from the in-pile tube forging with one solution heat treatment, the 1925°F , followed by the double age are reported herein.

Figure 31 is a diagram of the simulated nozzle weld heat treating mock-up. Ring 1 and ring 2 are the Inconel 718 material joined by a "medium" Argon test weld. Ring 3 and a base plate are stainless steel extensions to provide a heat sink. The test weld and adjacent parent material were heated to $2025 \pm 25^{\circ}\text{F}$ for an hour by induction heating and air cooled. The entire simulation was then solution heat treated at $1925 \pm 25^{\circ}\text{F}$ for one hour and air cooled. It was then aged at $1325 \pm 15^{\circ}\text{F}$ for eight hours, furnace cooled at the rate of 100°F per hour and aged for eight hours at $1150 \pm 15^{\circ}\text{F}$.

Material removed from the surface of the in-pile tube forging in the bulk head near the nozzle region was used to determine properties of the in-pile tube. Material from this location was solution heat treated at $1925 \pm 25^{\circ}\text{F}$ for one hour and aged for eight hours at the $1325 \pm 15^{\circ}\text{F}$ and eight hours at $1150 \pm 15^{\circ}\text{F}$ temperatures with the furnace cool between aging temperatures.

The tensile and impact data for the simulated nozzle weld samples are tabulated in Table V. All of the samples were produced transverse to the weld. All of the tensile samples except one, or 12 out of the 13 tests, had more than the required 127.0 ksi yield strength. The one outlier had 121.0 ksi yield strength. All of the tensile samples had more than the required 8% elongation. Three of the samples 2-4, 4-4, and 6-3 had reduced ductility associated with an acceptable sized inclusion in the weld. Grain size in the weld metal after heat treating ranged from ASTM 2 to 4 and 1 to 2 in the parent metal adjacent to the weld. The impact values were better than expected.

The tensile values for the in-pile tube material are shown in Table VI. All samples were above the required 150 ksi yield and 8% elongation. The grain size after heat treating ranged from ASTM 3 to 5.

Figure 31 - Local anneal nozzle weld mock-up.

TABLE V

RESULTS OF MECHANICAL TESTS FOR DOUBLE SOLUTION AND
DOUBLE AGE HEAT TREATED WELDMENTS

<u>Sample Designation</u>	<u>0.2% Offset Yield Strength ksi</u>	<u>Ultimate Strength ksi</u>	<u>% Elongation in 1 in.</u>	<u>Charpy Values ft-lb</u>
1-4	153.0	185.5	21.3	--
2-4	156.0	187.0	14.6	--
3-4	159.0	190.5	24.7	--
4-4	154.0	179.0	9.7	--
5-3	166.0	194.5	21.0	--
6-3	169.0	196.0	8.5	--
7-3	168.5	195.5	20.0	--
8-M	162.0	194.0	23.0	--
9-M	163.5	193.5	20.2	--
10-M	121.0	170.5	14.2	--
T-1	162.0	192.0	23.3	--
T-2	151.0	186.5	24.8	--
T-3	130.0	171.0	16.0	--
C-1	----	----	----	51
C-2	----	----	----	40

TABLE VI

TENSILE TEST RESULTS FOR SINGLE SOLUTION
AND DOUBLE AGE HEAT TREATED IN-PILE TUBE PARENT METAL

<u>Sample Designation</u>	<u>0.2% Offset Yield Strength ksi</u>	<u>Ultimate Strength ksi</u>	<u>% Elongation in 1 in.</u>
I-1	154.6	185.6	26.9
I-2	151.0	183.0	26.2
I-3	155.6	183.0	24.2
I-4	158.4	184.0	23.8

VI. HEAT TREATING AND VERIFICATION OF THE ACTUAL IN-PILE TUBE

1. PLANNED HEAT TREATING PROCEDURE

The in-pile tube was heat-treated in the manner which was planned. The nozzle welds were given a local solution heat treatment at $2000^{\circ}\text{F} + 50^{\circ}\text{F}$. The entire assembly was solution heat treated at $1900 + 50^{\circ}\text{F} - 0$ and then aged at $1300 + 50^{\circ}\text{F}$ for eight hours, furnace cooled at $100^{\circ}\text{F} - 0$ per hour to $1125 + 50^{\circ}\text{F} - 0$ and held for eight hours.

Tensile samples were attached to the tube after the localized heat treatment and remained with the assembly during its solution and aging treatment at the locations designated in Figure 32. Material for the tensile samples was removed from the in-pile tube forging adjacent to the actual in-pile tube wall and from the approximate axial location. In addition to the 13 locations shown, material removed from the top head was designated as location 14. Samples from each axial (number) location were taken at four azimuthal points 90 degrees apart and these points were designated A, B, C, and D. The eight nozzle samples designated 10 and 11 contained a transverse weld. These eight were first given a furnace solution heat treatment at 2025°F to simulate the localized treatment of the weld area before being attached to the tube.

It was intended to test all the samples upon completion of the heat treatments. However, it was found that the strength requirements of the main tube, 150 ksi minimum yield, was not achieved. After thirteen of the parent metal samples were tested and only one met the minimum it was decided to discontinue testing until the problem could be solved. The data obtained at this time, which includes the values for the eight welded specimens, are shown in Table VII. The yield strength values ranged from 136.2 ksi to 151.2 ksi which was adequate for the nozzle requirement (127 ksi) only. In all cases, the elongation was more than the required 8%.

2. RE-AGE

It was of considerable interest at this point to try to understand why the parent metal samples attached to the tube did not respond to heat treatment the same way that the previous qualification samples did. But more than this, it was desirable to use this knowledge to decide what could be done to bring the tube properties up to where they should be.

Three questions that were considered are: (1) Since the parent metal used for qualification came from the surface of the forging was the material truly representative of the tube wall material?, (2) Did the greater temperature range (50°F) experienced during heat treating of the tube compared to (25°F) for the qualification samples affect the properties?, (3) In either case, what minimum heat treating effort could one utilize to increase the yield strength to 150 ksi or above?

There were, of course, a number of approaches one could take. The least effort would be to re-age or extend the aging time⁽⁶⁾. This would minimize the possibility of distortion and the amount of scaling and produce the least divergence between the history of qualification samples and the actual tube. To investigate this possibility, the butt ends of the tested tensile samples were hardness checked (at room temperature) as a function of re-aging time, Table VIII, Figure 33. It was found that after two hours at 1325°F the Rockwell hardness (R_C) unexpectedly dropped two points. After four hours the two points were recovered, but then the hardness remained essentially the same as the starting hardness for the remainder of the 1325°F part of the re-age cycle. The hardness increased rapidly after cooling to 1150°F. After 26 hours of total aging time, samples showed as much as three points hardness increase and ranged from R_C 41 to 42.5. It appeared that the increased hardness was achieved at the 1150°F temperature and that the 1325°F age was contributing nothing. To check this observation, a sample from the A-12 tensile specimen was re-aged at 1150°F only. But after 28 hours it showed only one point increase in hardness.

On the basis of these hardness increases, it was decided to re-age some of the tensile specimens that had not yet been tested; to determine if the hardness increases represented sufficient strength increases. Three specimens C-13, B-3, and D-2 were re-aged at 1150°F for a total of 48 hours. Four other specimens B-5, B-7, C-6 and B-14 were re-aged at 1325°F for 8 hours, furnace cooled for two hours to 1150°F and held at 1150°F for 14 hours giving a total re-aging time of 24 hours. The results, Table IX, show that the 1325-1150°F re-age combination produced the desired results while the straight 1150°F re-age did not.

The remaining tensile samples were then re-distributed over the length of the tube assembly which was re-aged according to the 24 hour 1325-1150°F combination. These final tensile samples which had been with the tube throughout all of its heat treatments confirmed the final desirable results, Table X, with a minimum yield strength of 160.0 ksi and a minimum elongation of 16%.

Figure 32 - Tensile sample location designations.

TABLE VII

TENSILE TEST RESULTS OBTAINED FROM SAMPLES ATTACHED TO THE
IN-PILE TUBE DURING PLANNED HEAT TREAT PROCEDURES

Sample No.	Nominal Heat Treatment	Type Sample	0.2% Offset Yield Strength	Ultimate Strength	Elongation % in 2"	Reduction of Area %	R _c Av.
A-1	1925 ^o F-1 hr. 1325 ^o F-8 hr. 1150 ^o F-8 hr.	Parent Metal	140.0	165.5	27.5	46.9	38.3 37.5 37.5 38.0
B-1		" "	142.0	168.0	27.5	45.8	
C-1		" "	151.2	174.5	22.5	45.1	
D-1		" "	146.5	172.5	27.0	44.5	
A-2		" "	138.8	166.1	30.0	46.5	
B-2		" "	137.8	164.1	28.0	46.5	
C-2		" "	136.2	164.0	31.5	46.9	
D-3		" "	139.0	163.9	25.5	34.5	
B-4		" "	138.7	164.0	24.0	36.0	
D-4		" "	137.5	162.5	24.0	35.4	
A-12		" "	148.7	174.0	26.5	44.4	
C-12		" "	146.8	172.9	26.5	44.9	
A-13		" "	148.7	173.5	24.5	46.3	
A-10	2025 ^o F-1 hr. 1925 ^o F-1 hr. 1325 ^o F-8 hr. 1150 ^o F-8 hr.	Transverse Weld	150.7	162.2	15.0	34.5	34.8 34.4 43.2 35.2 37.0 39.8 34.0
B-10		" "	140.3	162.7	12.7	34.8	
C-10		" "	141.9	165.4	14.7	34.4	
D-10		" "	147.3	171.7	18.0	43.2	
A-11		" "	140.6	164.9	12.2	35.2	
B-11		" "	139.3	164.7	14.0	37.0	
C-11		" "	147.3	170.6	13.0	39.8	
D-11		" "	141.3	164.6	12.5	34.0	

TABLE VIII

HARDNESS AS A FUNCTION OF TIME IN RE-AGING OVEN

Sample No.	Temperature of Oven °F	Hardness Before Re-aging R _c	Hardness After Re-Aging R _c	Hardness R _c 44 Test Block	Time in Oven hours
A-1	1325	39.0	37.0	44.0	2
B-1	1325	37.5	37.5	44.2	4
D-3	1325	38.0	37.5	44.5	6
D-4	1325	38.5	38.5	43.9	7.5
A-2	1325 to 1150	38.0	38.5	44.0	9
C-2	1150	38.0	40.0	44.0	11
B-2	1150	37.0	40.5	44.5	13
B-1	1150	38.0	41.0	44.0	15
C-1	1150	40.0	41.0	44.5	17
A-13	1150	39.5	42.5	44.5	26.5
D-1	1150	40.0	42.0	44.5	28.5
A-12	1150	39.5	42.0	44.5	28.5
C-12	1150	39.5	41.5	44.5	28.5

Figure 33 - Rockwell "C" hardness change as a Function of re-age time at temperature.

TABLE IX

TENSILE TEST RESULTS OF SAMPLES (PREVIOUSLY ATTACHED TO THE TUBE DURING ITS
PLANNED HEAT TREATMENTS) AFTER RE-AGING

Sample No.	Re-Age Nominal	Type Sample	0.2% Offset Yield Strength	Ultimate Strength	Elongation % in 2"	R _c Av.
C-13 } B-3 } D-2 }	1150 - 48 hrs	Parent Metal " " " "	150.8 141.9 142.8	---- ---- ----	26.5 27.0 29.5	41.0 40.5 41.5
B-5 } B-7 } C-6 } B-14 }	1325 - 16 hrs 1150 - 14 hrs	" " " " " " " "	162.8 164.5 163.4 162.9	184.8 185.3 185.4 182.9	17.0 21.5 16.2 21.2	42.5 42.5 42.5 41.0

TABLE X

TENSILE TEST RESULTS OBTAINED FROM SAMPLES ATTACHED TO THE IN-PILE TUBE
DURING BOTH, THE PLANNED AND RE-AGE HEAT TREATMENTS

Sample No.	Nominal Heat Treatment	Type Sample	0.2% Offset Yield Strength	Ultimate Strength	Elongation % in 2"	Reduction of Area %	R _c
A-5	1925°F-1 hr. 1325°F-16 hr. 1150°F-22 hr.	Parent Metal	161.1	182.7	17.2	27.5	41
C-5		" "	162.4	182.4	17.2	25.9	41.5
D-5		" "	160.6	182.4	17.5	28.7	41.5
A-6		" "	161.6	181.4	16.0	30.7	42
B-6		" "	163.1	182.9	17.0	29.7	41.5
D-6		" "	165.5	185.2	21.0	41.7	41.5
A-7		" "	165.4	183.4	13.7	27.8	42
C-7 to A-4		" "	162.4	182.4	16.7	29.9	40.5
D-7		" "	164.4	185.4	15.2	29.3	40.5
A-14 to B-1		" "	161.0	183.6	23.0	41.5	40
C-14 to C-2		" "	160.0	181.1	23.2	43.0	41.5
D-14 to A-3		" "	160.0	180.6	21.5	42.2	41.0

VII. FURTHER DISCUSSION

Additional work prior to fabrication could have been done to determine optimum properties of the particular heats of Inconel 718(4) used for the PBF in-pile tube. It has been shown, DMIC 217, page 4, that some strength increase, which depends on aluminum content, can be derived by using a 1400°F initial age temperature rather than 1325°F. The recent trend is to use a 1400-1200°F age cycle if the material is solution heat treated in the 1900 to 1950°F range(7). However, at the time when the initial qualification tests were performed considerably more data were available for the 1325 - 1150°F aging cycle to show that this would produce a good compromise between strength, toughness, and ductility (1,8). If more work had been done, (1) to determine how to promote weld recrystallization at lower solution heat treat temperatures, (2) to determine what strengths could be achieved for various solution heat treat temperatures above the recrystallization temperature, and (3) to determine the effects of longer age times for the various solution heat treat temperatures, a single solution heat treat and age procedure might have been developed which would have eliminated the need for localized heat treating of the welds. The optimum welding and single heat treating procedure for this application would (1) eliminate localized Laves phase embrittlement so that elongation in all parts of the weld would be 8% or greater, (2) produce a fully recrystallized weld, (3) keep grain size in the parent metal small, ASTM size 4-6, (4) have a yield strength above 150 ksi, and (5) prevent the formation of micro cracks. Although it is not known whether optimum properties were attained, the minimum 160 ksi yield and 16% elongation finally achieved were more than adequate.

The Charpy impact values for weld metal solution heat treated at 1925°F and double aged were 19.8 ft-lbs for a un-recrystallized area and 27 ft-lbs for a recrystallized area, once again, illustrating the benefit of recrystallization. Samples that had been double solution heat treated at 2025 and 1925°F before aging gave surprisingly high values of 40 and 51 ft-lbs which would indicate that extra time at temperature might be beneficial to the toughness of alloy 718 welds.

The weld studies showed that one has greater weld parameter latitude considering microcracking tendencies if 75% helium, 25% argon, rather than 100% argon is used for cover gas. However, the welding procedure used for the actual nozzle weldments was the medium current 100% argon cover gas procedure because much of qualification work utilized this procedure and the welders were more experienced with it. The welds were crowned purposely so that, in grinding flush, the un-recrystallized layer containing Laves phase stringer would be removed.

VIII. CONCLUSIONS

1. Localized Laves phase embrittlement can exist in Inconel 718 weldments which have been solution heat treated at 1925°F. This embrittlement is associated with un-recrystallized areas in the weldments.
2. Laves phase solution and redistribution is related to recrystallization of the weld metal as well as to solution heat treatment temperatures.
3. Non-restrained surface weld beads do not recrystallize even upon heating to 2100°F. The un-recrystallized areas continue to show stringered Laves phase after heat treating up to this temperature.
4. The recrystallization threshold temperature for the restrained portion of the weld (all but the surface bead) is slightly below 1975°F but above 1925°F.
5. Additional strengthening of an assembly that has been solution heat treated at elevated temperature to reduce Laves phase, can be achieved by lengthening the aging time. Care should be taken to avoid excessive aging times at the higher aging temperatures to prevent over-aging.

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APPENDIX A

This appendix contains the results of qualification test data generated to support a proposed single solution heat treatment followed by a standard double age of the entire PBF in-pile tube assembly to be used after the nozzle weldments were made. During this test series, tensile test results were satisfactory but bend tests indicated areas of localized low ductility. The heat treatment procedure for all of these samples and that originally proposed for the assembled tube was 1 hour at 1925°F air cool, 8 hours at 1325°F furnace cool to 1150°F at a rate of 100°F per hour, hold at 1150°F for eight hours and air cool. See Tables XI through XV.

TABLE XI

RESULTS OF TENSILE TEST CONTAINING TRANSVERSE WELDS

Specimen Identification	UTS ksi	YS ¹	% Elongation in 1 in.	RA ²
		0.2% Offset ksi		(%)
W2-4	177.8	155.2	13.5	35.3
W2-5	178.0	153.0	11.7	34.0
W2-6	180.2	153.4	10.8	31.4
W4-2	182.6	153.8	13.5	33.9
W4-3	181.6	152.6	12.8	33.9
W4-4	179.4	151.0	12.4	35.3
W4-5	182.2	153.0	13.1	32.4
W4-6	180.4	155.2	12.8	34.8

Note: All specimens fractured in base metal.

¹Yield strength

²Reduction of area

TABLE XII

IPT BASE METAL TENSILE TESTS

<u>Specimen Identification</u>	<u>UTS ksi</u>	<u>YS 0.2% Offset ksi</u>	<u>% Elongation in 1 in.</u>	<u>RA (%)</u>
I-1	185.6	154.6	26.9	47.0
I-2	183.0	151.0	26.2	47.0
I-3	183.0	155.6	24.2	49.8
I-4	184.0	158.4	23.8	49.8

TABLE XIII

ALL WELD TENSILE TESTS

<u>Specimen Identification</u>	<u>UTS ksi</u>	<u>YS 0.2% Offset ksi</u>	<u>% Elongation in 1 in.</u>	<u>RA (%)</u>
W2-1	188.6	150.4	21.5	34.6
W2-2	189.4	152.8	21.4	35.7
W4-1	187.0	155.0	23.7	31.4
W4-2	187.2	150.0	18.0	32.6

TABLE XIV

IPT BEND TEST RESULTS

<u>Welder</u>	<u>Type Bend</u>	<u>Force to Bend Specimens. - ksi</u>	<u>Bend Results</u>
W-2 Test #1	Face	25	Bend 180°
W-2 Test #2	Root	23	Bent 180°
W-4 Test #1	Face	13	Broke in center of specimen at 30°
W-4 Test #2	Face	24	Broke approx. 1/2 way between center and end of specimen at 120°
W-4 Test #3	Root	21	Bent 180°
W-4 Test #4	Root	23	Bent 180°

TABLE XV

COMPARISON OF PROPERTIES FROM 30° FAILED BEND
TEST SPECIMENS MATERIAL TO PROPERTIES OF UNFAILED
BEND TEST SPECIMEN MATERIAL

<u>Property</u>	<u>W-2 Welder Unfailed Bend Test Specimen Material</u>	<u>W-4 Welder 30° Failed Bend Test Specimen Material</u>	
		<u>Left One-Half</u>	<u>Right One-Half</u>
Oxygen content	4 to 6 ppm	8 to 18 ppm	8 to 18 ppm
Tensile Specimens	UTS = 197 ksi	UTS = 188 ksi	UTS = 173 ksi
All Weld Metal	YS = 165 ksi	YS = 153 ksi	YS = 153 ksi
	Elong. = 22%	Elong. = 20%	Elong. = 4.5%
	RA = 33%	RA = 26%	RA = 8%
Charpy Specimen	27 ft-lb	22.2 ft-lb	19.8 ft-lb
Columbium Content	4.90% to 5.15%	4.90% to 5.15%	4.9 to 5.15%