OPTIMIZED SOLUTIONING TECHNIQUES

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Summary

The effect of several different solution treatments upon the tensile, stress- and creep-rupture properties of DS CM 247 LC is reviewed in comparison to the properties of the previously standard treatment. It is shown that the optimized solution treatment results in complete solutioning of the as-cast, coarse γ' along with considerable eutectic γ - γ' solutioning, without incipient melting. The resultant increase in fine γ' volume fraction, compared to the baseline, is shown to effect significantly improved mechanical property response. Microstructural features resulting from the various treatments investigated are presented with respective correlation to strength capability. High temperature, long duration soak and stress-rupture tested specimens (optimized solution treatment conditions) are used to illustrate the relatively stable (no sigma, mu or alpha W formation; some minor $M_6 C$) nature of the DS CM 247 LC alloy.

Introduction

Ni-based superalloys are referred to as "superalloys" due to their ability to exhibit outstanding strength at temperatures as great as 85% of their melting points (0.85 T_{M}). Such alloys consist of an austenitic FCC matrix (γ), dispersed intermetallic FCC gamma prime (γ) precipitate that is coherent with the matrix, plus carbides, borides and other phases which are distributed throughout the matrix and along the grain boundaries. Property attainment with superalloys is principally a function of a) the amount and morphology of the γ ', b) grain size and shape, and c) carbide distribution.

Superalloys have evolved over the last 50 years with most development work stimulated by demands of the advancing gas turbine engine technology. Cast, equiaxe superalloy development was fast-moving and highly rewarding up through the 1960's, with such activity resulting from the introduction of commercial vacuum induction melting and vacuum investment casting in the early 1950's (Fig. 1). However, once the developing alloys became "super-saturated" in terms of γ volume fraction, more attention was placed on casting process development, e.g., directional solidification.

Substantiating these efforts, directionally solidified superalloy turbine airfoils appeared military application during mid-1960's. Commercial engine application of such followed in the late 1960's and early 1970's, with turbine engine builders having utilized than 1 million DS airfoils to Significant mechanical date. property advantages were attained directionally solidified turbine airfoils, superalloy relative to those conventionally specifically, greater cast, (8X), rupture fatigue life (2X), and rupture ductility (4X) (Ref. 2).

PROGRESS IN TURBINE BLADE MATERIALS

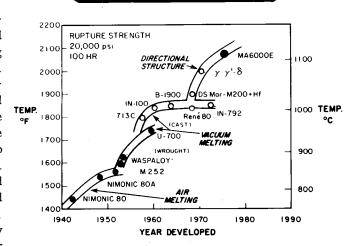


Figure 1. Progress in the Temperature Capability of Superalloys in the Last 40 Years (Ref. 1).

Although the strength advantages attained with DS processing were well established and many DS production castings were produced during the last 10-15 years, relatively few alloys realize production application. The fact that relatively few, really good DS superalloys exist, again, was a function of industry R&D emphasis being placed more on process rather than alloy development during this period. Of course, some of the well established equiaxe superalloys were modified slightly (Hf introduction) to facilitate improved DS castability, and were utilized in DS application. However, most can exhibit grain boundary cracking problems or metal/core reaction tendency (due to relatively high Hf level) when cast in today's complex-cored, thin wall airfoils.

In an attempt to develop an alloy which would overcome these problems, Cannon-Muskegon derived the DS CM 247 LC alloy from the base Mar M 247 composition. The primary alloying modification was the reduction of C by approximately one-half to improve carbide microstructure, stability,

Table I.

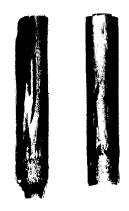
NOMINAL COMPOSITION (Wt. %)

	C	Cr	Co	W	Мо	Ta	A1	Ti	Нf	В	Zr	Ni
Mar M 247	0.15	8.4	10.0	10.0	0.7	3.0	5.5	1.0	1.5	0.015	0.05	BAL
CM 247 LC	0.07	8.1	9.2	9.5	0.5	3.2	5.6	0.7	1.4	0.015	0.015	BAL

and alloy ductility, plus the modification of the Zr and Ti contents to improve DS grain boundary cracking resistance without sacrificing strength (Table I). Additionally, the alloy's W and Mo levels were slightly reduced to compensate for the lower C and Ti levels to thereby minimize the formation of deleterious secondary M_6C platelets, μ phase and/or alpha W platelets or needles due to the degeneration of primary carbides with high temperature exposure. This degeneration or breakdown of the low parameter MC-1 (Ti rich) and MC-2 (Ta rich) carbides to form the higher parameter MC-3's (Hf rich) results in the release of Ta and some Ti to the solid solution, thereby changing the solubility of the W and Mo in the basic gamma solid solution; the result of such being the possible formation of μ phase, alpha W and/or M₆C, depending on temperature (Ref. 3). More detail regarding the early (1978) DS CM 247 LC development activity was presented in Reference 4. The alloy was also evaluated in integral turbine wheel application due to its desirable carbide morphology characteristics (LCF considerations), excellent ductility and creep strength with such having been reviewed in Reference 5 and 6. Additionally, further DS production data and review was presented in Reference 7.

In terms of the alloy's DS process castability, the same evaluation test, developed by the General Electric Company during their René 150 development work (Ref. 8), showed DS CM 247 LC to be similar to René 150 in grain boundary cracking resistance, with both being much superior to Mar M 247. The test essentially produces a thin-walled DS tube cast about an alumina core with the resultant solidification strain said to be approximately 2%. Grain-boundary cracking occurs in crack-susceptible alloys, with ratings ranging from A (no cracking) to F (gross grain-boundary cracking). Figure 2 illustrates the results of such a test undertaken with DS CM 247 LC and Mar M 247 with no cracks apparent in the DS CM 247 LC tube (A-rating) and considerable grain-boundary cracking in the Mar M 247 tube (E-rating).

So, with the DS CM 247 LC alloy possessing excellent DS castability, the thrust of the current program was to maximize the alloy's mechanical properties response through optimized solutioning and aging procedures. Program detail, along with pertinent results, follow. Note that the program is still in process, and as such, this writing represents an interim report.



Mar M 247 vs. CM 247 LC

Figure 2.
DS Grain Boundary Cracking
Test Results

Experimental Procedure

DS CM 247 LC VIM bar stock was produced at Cannon-Muskegon exercising the VIM technology detailed in Reference 9. Bar stock from two different CM, 8000 lb. master heats, i.e., V6550 and V6692, was supplied to two major precision investment casters, as well as a turbine engine producer captive foundry. Both DS bar and slab product (respective sizes as outlined in Table II) were obtained and utilized in chemistry overchecks and the heat treat investigation.

Single step solutioning was performed in a Lindberg tube furnace (Model 59545, accurate ± 3°F) at 2200°F, 2230°F, 2240°F and 2250°F for 2 hours at each condition, followed by rapid air cooling (air-blast assisted). Microstructural review (Nikon Epiphot instrument) the single-step solutioned specimens, in conjunction with knowledge that the material's incipient melting point (when single-step solutioned) is near 2290°F, resulted in the subsequent solutioning trials being undertaken with a "pre-homogenization" treatment (essentially homogenizing interdendritic areas) to effect increased incipient melting point.

Table II.

DS CM 247 LC SOLUTIONING CONDITION MATRIX

Single-Step Solution (°F/2 Hrs./A.C.)
· 2200°F

· 2220°F · 2230°F

· 2240°F

· 2250°F

Two-Step Solution (°F/2 Hrs. + °F/2 Hrs./A.C.)

· 2200°F + · 2220°F + · 2230°F + · 2230°F + 2270°F; 2280°F; 2290°F; or 2300°F

· 2240°F +

· 2250°F + 2270°F; 2280°F; 2290°F; 2300°F; 2310°F; or 2320°F

The two-step solutioning study followed with a DS slab casting being sectioned in a transverse manner to create five (5) each 2-1/2" x 5/8" x 5/8" test pieces with one of each being solutioned for 2 hours at 2200°F, 2220°F, 2230°F, 2240°F and 2250°F. The "pre-homogenized" specimens were then sectioned into fourths, apart from the 2250°F/2 hour specimen, which was sliced into six pieces.

The final step of the two-step solution treatment was undertaken with the sectioned specimens, being further treated at 2270°F, 2280°F, 2290°F and 2300°F, plus two of the 2250°F pre-homogenized specimens being treated at 2310°F and 2320°F for 2 hours, respectively. Table II details the solution treatment conditions in tabular form.

Microstructural characterization of the two-step solutioned specimens was undertaken and an optimum solutioning procedure selected for the subsequent mechanical test, alloy stability, DS casting process and aging treatment investigations.

Both slab and bar specimens were heat treated according to the optimized conditions $(2250^{\circ}\text{F/2 hrs.} + 2300^{\circ}\text{F/2 hrs.}/\text{RAC} + \text{pseudo coat}$ and aging) and subjected to RT and 1600°F tensile, plus $1400^{\circ}\text{F}-2000^{\circ}\text{F}$ stressand creep-rupture testing. Failed stress- and creep-rupture samples were reviewed for signs of unstability, along with specimens which were soaked at 1900°F for 500 and 1000 hours each, respectively. Additionally, identically solutioned slabs were used to study the effects of various aging treatments, e.g., 1975°F/4 hrs., 1800°F/5 hrs., 1922°F/16 hrs., 1600°F/20 hrs., and 1650°F/16 hrs.

Results and Discussion

Chemistry Overchecks

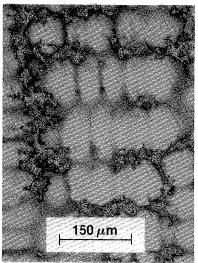
The DS CM 247 LC aim chemistry was balanced to provide excellent DS process castability and strength potential. The alloy was also designed to exhibit a relatively wide heat treatment "window" (difference between the alloy's γ ' solvus and incipient melting temperatures), which is quite unique relative to other DS and equiaxe alloys.

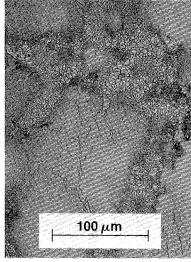
Features such as DS grain boundary cracking resistance and heat treatment "window" are extremely chemistry sensitive, with the severity dependent upon the alloy. Maintaining proper chemistry balance through precision investment casting application is crucial. Table III shows some of the elements which must be tightly controlled in the CM 247 LC alloy to maintain its performance capability. Note that pick-up of Si and Zr occurred as a function of molten metal contact time with the investment casters melt crucible and/or shell system. Additionally, Hf loss occurred as a relation of the same. Gas levels were maintained quite satisfactorily, indicating excellent mold pre-heat/out-gassing (inherent with DS) and vacuum furnace leak-tightness. Al + Ti levels were maintained with the exception of one set of specimens.

Severe Si and Zr pick-up could lead to increased DS grain-boundary cracking tendency during casting production -- the level of pick-up required for such not investigated herein. Inclusions associated with high gas level are also found to affect DS grain yield, therefore making tight control of [N] and [O] and overall alloy cleanliness extremely important. Of course, Al + Ti fade results in inclusion formation and mechanical property deterioration. Sulfur, tending to migrate to grain boundaries, can decrease hot ductility and promote cracking during component application (Ref. 10), thereby suggesting it be closely controlled as well. Minimization of shell or core system reactivity with Hf containing alloys must be achieved, otherwise hafnium oxide formation may occur at the expense of overall alloy Hf content potentially causing variation in terms of component cleanliness, grain yield, carbide morphology and eutectic γ - γ ' characteristics. Noteworthy was the fact that the severity of Hf loss and Si plus Zr pick-up increased in the top of the specimen, an effect of the longer molten alloy contact time in the mold.

Single-Step Solutioning

Figure shows typical as-cast, DS CM 247 LC slab microstruc-Comparison Figures 4 and 5 suggest that coarse some solutioned the 2220°F/2 hr. condition, and that the amount γ' solutioning creased as the soak temperature was raised from 2220°F to 2250°F. Significant eutectic γ - γ' solutioning was





realized except Figure 3. DS CM 247 LC as-cast microstructure.

Section size: 5/8"

Table III.

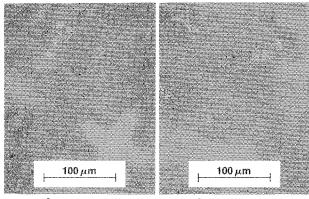
DS CM 247 LC SELECTED CHEMISTRY DETAIL
MASTER HEAT VS. INVESTMENT CAST SPECIMENS

Element		Master Heat	TRW DS S	Slab *	Rolls Royce D	S Slab **	
		V6692	Bottom	Top	Bottom	Top	
С		.070	.070	.067	.070	.070	
Si		.003	.005	.014	.009	.010	
Zr		.018	.022	.023	.021	.018	
A1		5.65	5.72	5.70	5.68	5.66	
Ti		.69	.68	.68	.70	.68	
Нf		1.4	1.39	1.36	1.49	1.22	
[N]	ppm	3	2	2	3	2	
[0]	ррщ	3 5	2	2	3	3 7	
S	ppm	5	6	6	6	7	
Element		Master Heat	AETC DS	Slab †	TRW DS Bar ††		
		V6550	Bottom	Top	Bottom	Top	
С		.071	.075	.076	.072	.070	
Si		√, .007	.004	.006	.008	.009	
Zr		*.020	.024	.023	.025	.023	
Al		5.55	5.48	5.50	5.58	5.60	
Ti		.70	.63	.63	.68	.66	
Нf		1.4	1.26	1.06	1.41	1.23	
	ppm	2	2	3	2	2	
	ppm	1	1	1	2	2 7	
_	ppm	6	8	8	6	7	

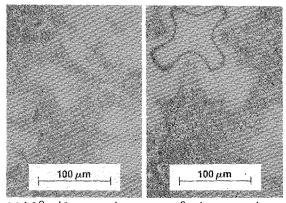
at the $2250\,^{\circ}\text{F/2}$ hr. condition, that of which was later realized to be much below the alloy's capability.

The increased solutioning achieved at the $2250\,^{\circ}\text{F/2}$ hr. soak condition effected improved creep-rupture life as a result of increasing the volume fraction of fine, reprecipated γ' relative to those specimens solutioned at lower temperature. The consequence that increased volume fraction fine γ' (given a fixed total amount of γ') results in enhanced creep response is well accepted, as reported for the DS Mar M 200 Hf alloy by Reference 11 (Fig. 6).

The creep-rupture benefit gained with the single-step solutioned specimens was not significant since the alloy's volume fraction of fine γ ' was only marginally changed due to inadequate solutioning. However,



2220°F/2 Hrs./RAC 2230°F/2 Hrs./RAC



2240°F/2 Hrs./RAC 2250°F/2 Hrs./RAC

Figure 4. DS CM 247 LC single-step solutioning.

Figure 5. DS CM 247 LC singlestep solutioning.

the exercise did show the alloy's γ ' solvus to be near 2220°F, as well as the capability to solution a portion of the eutectic γ - γ ' constituent without incipient melting -- a most unusual characteristic for a DS alloy where grain boundary strengthening elements such as C, B, Zr and Hf (melting point depressants) are present.

Two-Step Solutioning

The full solutioning potential of the DS CM 247 LC alloy was realized by step-solutioning to effect

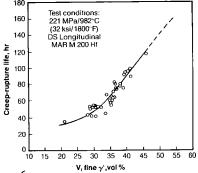


Figure 6. DS Mar M 200 Hf rupture life vs. volume fraction (V_f) fine γ' at a fixed total amount of fine and coarse γ' (Ref. 11).

homogenization of the cast, segregated structure prior to a final, high-temperature treatment. The homogenization effectively raises the alloy's incipient melting point, thereby allowing much more flexibility in the choice of a final treatment temperature. Additionally, it was found that a relatively high secondary solutioning temperature must be attained to result in the most effective γ - γ ' eutectic solutioning.

Due to the alloy's inherently high incipient melting point and relatively wide temperature range between its γ' solvus and incipient melting point (not common for DS alloys), effective homogenization of the segregated structure occurs in a reasonable time frame and without incipient melting. Most γ' super-saturated DS alloys possess a relatively narrow heat treatment "window" making extensive γ' solutioning (apart from the dendrite cores) difficult within a reasonable period of time, with incipient melting often occurring in the segregated interdendritic regions. These other DS Ni-based alloys are not known to exhibit eutectic γ - γ' solutioning without incipient melting, with some not even able to achieve coarse γ' solutioning.

With DS CM 247 LC, though, complete coarse γ' solutioning occurs, along with considerable eutectic γ - γ' solutioning, also. The alloy's extensive eutectic γ - γ' solutioning capability (as shown in Fig. 7

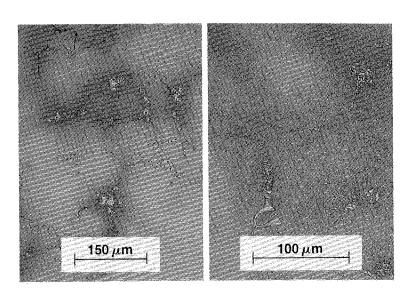


Figure 7. DS CM 247 LC solution treated at $2250^{\circ}F/2$ Hrs. + $2300^{\circ}F/2$ Hrs./RAC.

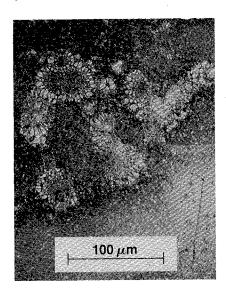


Figure 8. DS Mar M 247 solution treated at 2230°F/2 Hrs./GFQ. Section size: 1/8"