STATISTICAL MODELS FOR HEAT TREATING RENE'220C

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ABSTRACT

Statistically designed studies on Rene'220C have indentified hardness and impact behavior with varying heat treatments. The Central Composite Design approach, used in this effort, is a powerful tool for developing heat treating models and thereby optimize parameters. Conclusions from this evaluation are that fine-grain (FG) and conventionally cast (CC) Rene'220C not only exhibit different microstructures, but have different hardness and impact responses to the imposed heat treatments. Further, Rene'220C behaves as an "in situ" composite. The model shows an interaction between the two aging steps (AGE1 and AGE2) and hardness and reveals a "robust" design point. The impact response strongly depends on the first step (AGE1) of the two-step age cycle. A significant observation is that CC material exhibits over 2 times the impact of FG material.

INTRODUCTION AND BACKGROUND

An ever increasing demand for more efficient aircraft engines provides a drive for improved cast Ni-based superalloys. Rene'220C is a strong candidate for engine structural applications. Earlier, Rene'220C had been developed as a wrought alloy, then called CH22¹, but now is being successfully applied as a cast material on F110-129 turbine frames². Rene'220C's composition is similar to cast 718, but contains controlled amounts of strengthening alloy elements. Although the composition approximates Cast 718, the Fe has been replaced with Co, and Ta has been added.

Metallographic observations on Rene'220C have revealed similarities with cast 718. In both cases the composite ...and I emphasize the composite ...nature of this structure has been clearly identified^{3,4}. This material distinguishes itself by the precipitation of the delta (δ) phase. Recent work compares small grained castings with conventional cast and HIP materials⁵. Such high Co alloys have large liquidus-to-solidus gaps (ca. 350°F) that aid metal flow and mold fill, but display extreme cast inhomogeneity with large dendrites⁶.

The specific goal of this work is to investigate the interaction of hardness and impact properties for both conventionally cast and fine grain Rene'220C.

A powerful tool in developing and refining heat treat processes is statistics. Factorial experiments are most commonly used. Another scheme for evaluation of a process is by the Central Composite Design. An advantage of this approach is that it allows you to look at extended regions, or what are called "star" points, for enriched data analysis and modeling. In this effort, impact (as it relates to crack resistance) and hardness (as it depends on strength) are evaluated. Previous work evaluated the HIP temperatures, the delta precipitation temperature, and the initial step in the age cycle⁴. Here the focus is on the delta precipitation temperature and the two age cycles.

MATERIAL AND PREPARATION

Two types of material were evaluated in this study. One was conventionally cast (CC) R'220C, from a PCC cast turbine rear frame flange (#015), as part of lot AL V0737. The second material type was fine-grain (FG), sometimes referred to as "Micro-Grain", cast R'220C, from Howmet lot VKK0002. These two materials had similar chemistries. The conventionally cast flange was given the 2125°F/4 hours HIP cycle, while the fine-grained received a 2050°F/4 hours HIP cycle. Both materials were solution treated at the 2000°F/1 hour condition. These materials were then heat treated in a statistically designed manner, shown in Table I and II.

APPROACH

CENTRAL COMPOSITE DESIGN HEAT TREATMENT

All specimens were vacuum heat treated to evaluate three post-solution treatments. First, the DELT treatment, precipitates the delta (δ) phase around the interdendritic regions, in a temperature range between 1615°F to 1785°F centered about 1700°F, all for 4 hours. Second and third, the age cycles precipitates both gamma prime (γ') and gamma double prime (γ''). The initial age cycle, designated AGE1, specimens were heated between 1355°F and 1445°F centered about 1400°F, all for a time of 4 hours. The second part of the age cycle, designated AGE2, specimens were heated between 1280°F and 1370°F centered about 1325°F, all for a time of 8 hours. Again the summary of the three-block, 18 specimen, test matrix is shown in Table II. All sections were machined into Charpy specimen, hardness tested, impacted, and metallographically evaluated.

Table I Hardness, Impact, & Percent Delta Results For Fine Grain (FG)

Rene'220C.

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Row	STA_NO	HIP_T	DEL_T	AGE1_T	AGE2_T	HARD_DELRc	HARD_AG1Rc	HARD_AG2Rc	Impact_Flb	per_DELTA
1	1.	2050.	1650.	1375.	1300.	24.2	35.2	39.6	7.59	15.
2	2.	2050.	1650.	1425.	1350.	24.0	38.3	40.6	6.51	15.
3	3.	2050.	1750.	1375.	1350.	20.5	35.3	40.4	7.23	25.
4	4.	2050.	1750.	1425.	1300.	21.2	38.1	42.2	6.15	25.
5	5.	2050.	1700.	1400.	1325.	23.4	36.2	41.4	6.44	20.
6	6.	2050.	1700.	1400.	1325.	22.4	36.7	41.1	6.73	25.
7	7.	2050.	1650.	1375.	1350.	22.1	35.7	39.9	7.23	10.
8	8.	2050.	1650.	1425.	1300.	23.5	37.0	42.6	6.36	15.
9	9.	2050.	1750.	1375.	1300.	22.7	36.5	40.5	8.10	30.
10	10.	2050.	1750.	1425.	1350.	21.2	38.3	41.6	6.58	25.
11	11.	2050.	1700.	1400.	1325.	23.5	38.0	41.5	6.51	15.
12	12.	2050.	1700.	1400.	1325.	22.6	37.0	41.6	6.36	20.
13	13.	2050.	1785.	1400.	1325.	18.5	37.8	40.5	7.16	30.
14	14.	2050.	1615.	1400.	1325.	24.8	37.7	39.5	6.58	5.
15	15.	2050.	1700.	1445.	1325.	22.0	36.5	42.3	5.71	15.
16	16.	2050.	1700.	1355.	1325.	22.4	35.0	39.5	7.96	20.
17	17.	2050.	1700.	1400.	1370.	24.6	38.3	41.2	7.09	15.
18	18.	2050.	1700.	1400.	1280.	22.4	38.9	41.3	6.58	10.
19	19.	2050	1700/8	1425.	1300.	22.8	38.3	41.5	6.58	25
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Table II Hardness, Impact, & Percent Delta Results For Conventionally

Cast (CG) Rene'220C.

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Row	STA_NO	HIP_T	DEL_T	AGE1_T	AGE2_T	DELT_Rc	AGE1_Rc	AGE2_Rc	Impact_flb	pct_DELTA	
1	21.	2125.	1650.	1375.	1300.	21.0	36.4	41.1	21.62	15.	
2	22.	2125.	1650.	1425.	1350.	19.4	37.7	40.9	15.40	20.	
3	23.	2125.	1750.	1375.	1350.	17.0	34.4	40.0	14.90	30.	
4	24.	2125.	1750.	1425.	1300.	17.6	38.8	43.0	11.57	40.	
5	25.	2125.	1700.	1400.	1325.	19.6	37.5	41.6	13.89	35.	
6	26.	2125.	1700.	1400.	1325.	19.1	36.9	42.1	13.89	40.	
7	27.	2125.	1650.	1375.	1350.	20.3	36.3	41.3	13.02	20.	
8	28.	2125.	1650.	1425.	1300.	19.7	37.2	42.8	10.49	20.	
9	29.	2125.	1750.	1375.	1300.	18.1	35.3	39.9	19.45	50.	
10	30.	2125.	1750.	1425.	1350.	16.1	39.3	43.3	14.83	40.	
11	31.	2125.	1700.	1400.	1325.	20.7	37.7	41.9	13.31	20.	
12	32.	2125.	1700.	1400.	1325.	18.8	37.3	41.6	13.16	20.	
13	33.	2125.	1785.	1400.	1325.	14.4	39.6	43.7	13.67	60.	
14	34.	2125.	1615.	1400.	1325.	20.5	34.5	38.6	19.45	10.	
15	35.	2125.	1700.	1445.	1325.	14.5	34.0	40.9	13.67	20.	
16	36.	2125.	1700.	1355.	1325.	19.7	34.8	39.2	22.27	30.	
17	37.	2125.	1700.	1400.	1370.	17.6	35.6	39.9	14.90	40.	
18	38.	2125.	1700.	1400.	1280.	14.6	36.6	38.7	15.69	25.	
19	39.	2125.	1700/8	1425	1300	21.6	38.2	43.0	12.15	40	
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TESTING

Charpy specimens were machined with the length along the periphery of the flange and along the plate length, and the machined impact surface on the flange's and plate's outer surfaces.

Evaluations of the dependent variables of hardness, Charpy impact, and metallographic structure were made. All measurements were performed on the Charpy specimens. Rockwell C hardness tests, a minimum of three per specimen, were made in identical locations on each specimen away from the impact area. Charpy testings were performed on an Amsler-Schnadt impact machine. One-half of each fractured Charpy bar was mounted for optical metallography, polished and etched electrolytically at 2-3 volts in Rife's etchant (62.5% H_2O , 22.5% HCl , 7.5% HNO_3 , 7.5% HF , and 5 gms $CuCl_2$). Scanning electron microscopy (SEM) was performed in the Cambridge instrument and dispersive x-ray analysis (EDAX) was made at 20 KV. The grain size and volume percent of delta (δ) were both determined by a lineal analysis technique.

RESULTS

All hardness and impact data are recorded in Table I for the fine-grain material and in Table II for the conventionally cast material.

HARDNESS

There are three types of hardness data. The first was taken after the delta treatment (DELT); the second after the initial age treatment (AGE1), and the third was after the second part of the double-age cycle (AGE2). The data after the AGE2 cycle was considered to represent the full heat treatment, and most of the statistical evaluation was made on these data.

After the DELT heat treat cycle, the hardness levels for both the FG and CC materials are about 5 Rc points higher than the starting hardness of the as-solutioned material, i.e., 18.5 (FG) vs. 13.5 (CC). The lower temperature reaction probably forms a finer δ phase in the grain boundaries, and thereby δ age-hardens the material by about 5 Rc points above the as-solutioned material.

Following the AGE1, the first part of the dual age, there appears to be a maxima at slightly over 1400°F. This results from the precipitation of fine, coherent γ'' particles. Both materials now exhibit the same hardness level, ca. 37 Rc.

After the AGE2, the second part of the dual age, the materials are considered to be representative of the full heat treatment, and in general indicate a hardness peak at about 1325°F. Although the CC material is slightly harder, the FG material displays less scatter. These hardness values result from both the γ' and γ'' precipitations. The contour plots for both FG and CC under these conditions are shown in Figures 1, 2, 3, and 4. Interesting to note is that the maximum hardness for the FG material is more influenced by the DELT temperatures, while the CC material is controlled more by the AGE2 temperature (Figure 1 and 2). Further, the CC material reveals a distinct hardness maxima for the AGE1 and AGE2 temperatures at 1440°F and 1315°F (Figure 4), which also is shown in Figure 4A as a doom-shaped area on the surface plot.

Figure 1. Hardness(Full HT) Vs. DELT & AGE2 Temperatures (Fine Grain Rene'220C).

Figure 2. Hardness(Full HT) Vs. DELT & AGE2 Temperatures (Conventionally Cast Rene'220C).

Figure 3. Hardness(Full HT) Vs. AGE1 & AGE2 Temperatures (Fine Grain Rene'220C).

Figure 4. Hardness(Full HT) Vs. AGE1 & AGE2 Temperatures (Conventionally Cast Rene'220C).

Figure 4 A. Hardness(Full HT) Vs. AGE1 & AGE2 Temperatures
(Conventionally Cast Rene'220C).

IMPACT

The Charpy impact results are recorded in Table I and II. These impact energy relationships with the independent processing variables are shown in contour plots, Figure 5, and 6. Clearly noted is that the impact strengths for the CC material are over twice that of the FG material. For both FG and CC materials, the AGE1 temperature produces an inverse relationship (more pronounced for the CC material), with the lowest AGE1 temperature generating the highest impact values. This suggests that the γ'' controls impact to a greater extent, and probably the fine γ'' distribution enhances energy absorption. This γ'' feature is described later.

Comparing the FG material impact (Figure 5) with hardness (Figure 3) it can be noted that impact can be increased, with little hardness loss, by decreasing both AGE1 and AGE2 temperatures. Likewise, for the CC material, comparing Figure 6 (impact) with Figure 4 (hardness), that by decreasing the AGE1 temperature to 1400°F increasing the AGE2 temperature to 1325°F, the hardness would increase (higher strength), and more importantly, impact could be significantly increased (almost 25%).

Figure 5. Impact Vs. AGE1 & AGE2 Temperatures
(Fine Grain Rene'220C).

Figure 6. Impact Vs. AGE1 & AGE2 Temperatures
(Conventionally Cast Rene'220C).

METALLOGRAPHY

Metallographic examinations on the etched material show distinct differences between the FG and CC materials as seen in Figures 7 and 8. The surfaces were first lightly etched to reveal the δ phase, then more heavily etched to enhance the grain boundary regions. In most cases the δ plates are etched away leaving behind the δ trenches.

The microstructures clearly show the FG's more equiaxed grain structure, and the CC's dendrite nature. These structures reveal the varying amounts of needle-like δ phase. The δ phase is a complexed $\text{Ni}_3\text{Cb}(\text{Ta})$ structure, usually orthorhombic, with a number of different features: δ can appear as globular eutectic, needle-like (most commonly noted form), "blocky", or fine. The δ needles appear to deform extensively, even at room temperature, but at higher magnification this deformation is seen as a "kinking" behavior.

The 1650°F DELT treatment not only limits the needle δ formation, but also produces a "halo" effect. This "halo" effect is identified with the presence of a γ " (or a fine δ -like) precipitate in the region adjacent to the grain boundary. Also noted was that γ " precipitation occurs in the previous traces of the solutioned δ needles. At first it may seem strange that γ " forms in the vestiges of the previously dissolved δ , but it is not too surprising, since both Cb and Ta have not diffused away, and δ and γ " are essentially the same $\text{Ni}_3\text{Cb}(\text{Ta})$ chemistries. This provides a "memory" for structure morphology.

Estimates of the amount of needle δ phase as a function of DELT were made. The observed percent increases monotonously with increasing DELT temperature. Again, it needs to be pointed out that the observed percent of the needle δ phase was a lineal analysis of the area and does not account for the depleted area between the δ plates. Further, these amounts depict neither the quality nor the fineness of δ phase. Higher magnifications (5000X) reveal the δ to be coarser at higher DELT temperatures.

Highly significant is the composite nature of both cast alloy. The dendrite regions are lightly alloyed matrix sprinkled with γ' and γ ", while the interdendritic regions, in addition to γ' and γ ", have extensive build-ups of Cb and Ta and contain carbides and δ . EDAX examination of the interdendritic region reveal the presence of a small amount of Cb(Ta) carbide and the coarse ("blocky") eutectic δ ; the matrix emits a normal trace for the Rene'220C matrix.

Estimates of the ASTM grain size of both the FG and CC materials were made. The average grain size for the FG was 4.2, while for the CC material it was about -4.

There is a distinct difference in the crack propagation modes for FG and CC Rene'220C. The FG cracks follow, almost exclusively, the grain boundaries, while for the CC material cracking migrates across dendrites. These dendrites appear to act as barriers and provide crack blunting. Hence, given the opportunity, cracks will propagate through the more brittle interdendritic network.

100X

500X

1000X SEM

5000X SEM

Figure 7. Optical and SEM Microviews of Specimen 13, Fine Grain(FG) R'220C, After the 1785°F/4Hour(DELT) + 1400°F/4Hour(AGE1) + 1325°F/8Hour(AGE2) Heat Treatment.

100X

500X

1000X SEM

5000X SEM

Figure 8. Optical and SEM Microviews of Specimen 33, Conventionally Cast(CC) R'220C, After the 1785°F/4Hour(DELT) + 1400°F/4Hour(AGE1) + 1325°F/8Hour(AGE2) Heat Treatment.

DISCUSSION

Both fine grain (FG) and conventionally cast (CC) Rene'220C materials are "in situ" composites, as previously noted^{3,6}. Properties of these materials are dependent on this composite-like structure.

Hardness of both FG and CC materials are inversely proportional to impact absorption. These hardness levels increase as a function of the imposed independent processing variables, DELT, AGE1, and AGE2. The baseline values, after the 2000F/1hour solution, are Rc=18.5 for the FG and Rc=13.5 for the CC.

The DELT treatment increases the hardness levels about 5 points, to Rc=22.5 for the FG and Rc=18.3 for the CC. Further noted was that the lower DELT temperatures produce higher hardness; not surprising, since this hardness increase is due to the δ precipitation, with the lower temperatures producing the finer δ precipitation.

The AGE1 treatment increases the hardness level over 15 points, to Rc=37.0 for the FG and Rc=36.7 for the CC. In both cases a peak appears at about 1425°F. Probability this hardness increase results from γ' precipitation.

The AGE2 treatment produces an additional hardness increase of about 4 points, to Rc=41 for the FG and Rc=41.4 for the CC. This added hardness increase occurs from the γ' precipitation. Undoubtedly γ' precipitation occurs in close proximity with the γ'' , in a "sandwich" γ'/γ'' structure, previously noted⁴. No hardness peaks appear, but rather the AGE2/hardness curves are relatively flat between 1300°F - 1350°F.

Interactions are noted between the AGE1 and AGE2 treatments, notably for the CC material, which reveals a hardness peak (at AGE1=1440°F and AGE2=1315°F). This peak probably results from the maximum matrix strain from the coprecipitation of γ'' and γ' , and results in the highest strength levels. In a statistical design parlance, this maxima represents a "robust" design point, since it would offer the best parameter condition to minimize the dependent property loss with variation in the independent processing variables. However, this property point must also be balanced with the other property values considered, in this case, impact.

The CC Rene'220C exhibits more than two times the impact of FG Rene'220C. The reason for this relates to structure and the mode of crack propagation. CC material contains more dendrites and these aid in crack blunting.

Impact property is found to be relatively insensitive to DELT and AGE2 treatment, i.e. the curves remain flat. However, impact is most sensitive to the AGE1 treatment. For both the FG and CC material, the lower the AGE1 temperature, the greater the impact. This is probably due to the formation of a finer γ'' within the interface between the dendrite and the interdendritic regions.

If one makes the assumption that interdendritic regions offer no resistance to crack growth (impact energy absorption), then all the energy is absorbed by the dendrites. The impact behavior is determined solely by the nature of the dendrites. Also let us assume that the impact energy has a direct relationship to the crack hold-time growth behavior. More than likely this assumption is not valid, that all the energy is absorbed by the dendrites, but rather some of the energy is absorbed by the interdendritic regions. Other studies have shown the δ distribution influences the crack growth behavior.

It should be recognized that the as-cast structure is transient. With each succeeding heat treatment, Cb and Ta tend to level-out. The fine needle-like δ provides more effective crack-blunting. When "sufficient" Cb and Ta diffuse (at temperatures above 1700°F for long times) then coarse δ can decorate the grain boundary and embrittle the material.

SUMMARY AND CONCLUSIONS

In summary, a statistically designed heat treat study of Rene'220C indentified the hardness and impact interactions as a functions of DELT, AGE1, and AGE2 treatments.

The main conclusions from this study are:

1. The fine grain (FG) Rene'220C and conventionally cast (CC) Rene'220C differ not only microstructurally, but exhibit different hardness and impact properties.
2. The CC Rene'220C has over 2 times the impact of FG Rene'220C.
3. Impact is strongly dependent on the AGE1 treatment (the first step in the double age cycle) with lower temperatures resulting in higher values.
4. The "in situ" composite nature of Rene'220C is reconfirmed.
5. The CC Rene'220C exhibits a rather unique interaction with the AGE1 and AGE2 treatments; hardness can be chosen from a "robust" design point.
6. The heat treat models aid in understanding the heat treatment responses.
7. This Central Composite Design approach is a powerful method to determine heat treat interaction, and thereby optimize parameters for desired properties.

ACKNOWLEDGEMENTS

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