Investigation of the Formation of Topologically
Close Packed Phase Instabilities in
Nickel-Base Superalloy René N6

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July 1999
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Summary

Topologically close packed (TCP) phase instability in third generation Ni-base superalloys is understood to hinder component performance when used in high-temperature jet engine applications. The detrimental effects on high temperature performance from these brittle phases includes weakening of the Ni-rich matrix through the depletion of potent solid solution strengthening elements. Thirty-four compositional variations of polycrystalline René N6 were defined from a design-of-experiments approach and then cast, homogenized, and finally aged to promote TCP formation. Our prior work reported on the results of the multiple regression modeling of these alloys in order to predict the volume fraction of TCP. This paper will present further regression modeling results on these alloys in order to predict the occurrence of TCP in third generation Ni-base superalloy microstructures. Kinetic results are also discussed.

Introduction

The development of Ni-base superalloys continues to be an area of interest for high temperature engine application. These alloys are being studied because they exhibit good properties at elevated temperatures. However, their high refractory metal content creates difficulties in alloy development because although they contribute significantly to high temperature strength, they also promote microstructural instability. The occurrence of brittle, intermetallic topologically close-packed (TCP) phases that is typical of third generation superalloy compositions has a detrimental effect on the alloy at probable service conditions.

The goal of this work is to broaden the understanding of the presence of TCP phase formation in the microstructures of third generation Ni-base superalloys. The effect of chemistry on the occurrence of TCP phases in Ni-base superalloys has been widely reported (ref. 1) and several predictive methodologies such as PHACOMP (ref. 2) and the d-electron theory (ref. 3) have been used as guides for alloy design. Several of the assumptions associated with these techniques, however, are not accurate with respect to the actual partitioning of elements between phases in the microstructure. These alloys are very complex and detailed experimental observations of the chemical effect on microstructure are useful in order to understand the formation of TCP phases. This work uses an alternative approach of statistical design of experiments to determine the propensity of individual elements for contributing to the precipitation of TCP. The resultant models should also be a good predictor of TCP occurrence in alloys that fall within the experimental design space.
Approach

A series of experimental superalloys with compositions containing Ni-Co-Al-Cr-Mo-W-Re-Ta were chosen based on René N6 because it is typical of third generation alloys (ref. 4). Third generation superalloys are used in single crystal form in turbine blade applications. However, to simplify production and the microstructures, the alloys in this study were cast in polycrystalline form and without the inclusion of C, B, and Hf, which are elements that are typically present in commercially available René N6. The polycrystalline material increases the likelihood of TCP formation in the microstructure due to the increased amount of high-energy grain boundary area and by precluding the formation of carbides, which ties up refractory elements.

All alloys were vacuum induction melted in argon starting with 100 g charges of high-purity melting stock. The inductively stirred melts were then poured into a copper mold to obtain the cylindrical castings nominally 2 cm in diameter by 4 cm long. All samples were fully homogenized at 1315 °C for 80 hr followed by an air cool. A second exposure at 1093 °C for 400 hr was given to promote TCP precipitation in the superalloy microstructures. Back-scattered electron scanning electron microscopy was performed on all aged microstructures to observe the changes caused by aging. Image analyses of the microstructures were subsequently performed using a computerized image analysis software package in order to determine total volume fractions of TCP phases. Several additional time-temperature aging conditions at 871, 1093, and 1204 °C at times between 1 and 1000 hr were investigated on several representative alloys to establish an expanded understanding of TCP precipitation kinetics.

The current work is an extension of the project first reported in reference 5 where TCP phase formation was examined in third generation Ni-base superalloys through regression modeling. The previous investigation used a 44 alloy database (34 unique compositions plus 10 repeats of one composition) to determine the regression model. Since then, seven new alloys were made to verify those results and it became apparent that remodeling using the new 51 alloy database could possibly improve the previous result and those trials are presented in this paper. The compositions of these new alloys are listed in Table I in atom percent. A design-of-experiments (DOE) was set up to quantify the linear and pairwise interactive effects of Al, Co, Cr, Mo, Re, Ta, and W in an 8-component superalloy (Ni-base René N6 without C) on the resultant amount of TCP phase. Regression equations were developed from a single temperature and time combination of 1093 °C for 400 hr in order to predict TCP precipitation in the alloys after aging and to empirically investigate specific element contributions to TCP in the microstructures. The DOE investigated a range of elements included at either their low or high limit as defined by the patent (ref. 6). The alloys had the following composition ranges in atomic percent: Co 10.61 to 16.73 percent, Mo 0.32 to 1.34 percent, W 1.85 to 2.52 percent, Re 1.80 to 2.11 percent, Ta 2.36 to 3.02 percent, Al 11.90 to 14.75 percent, and Cr 3.57 to 6.23 percent.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Al</th>
<th>Co</th>
<th>Cr</th>
<th>Mo</th>
<th>Re</th>
<th>Ta</th>
<th>W</th>
<th>Ni</th>
</tr>
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<tbody>
<tr>
<td>V1</td>
<td>14.9</td>
<td>16.7</td>
<td>6.3</td>
<td>0.3</td>
<td>2.1</td>
<td>2.4</td>
<td>1.9</td>
<td>55.5</td>
</tr>
<tr>
<td>V2</td>
<td>14.9</td>
<td>16.7</td>
<td>6.3</td>
<td>1.3</td>
<td>1.8</td>
<td>2.4</td>
<td>1.9</td>
<td>54.8</td>
</tr>
<tr>
<td>V3</td>
<td>11.9</td>
<td>16.7</td>
<td>6.3</td>
<td>0.3</td>
<td>1.8</td>
<td>3.0</td>
<td>2.5</td>
<td>57.4</td>
</tr>
<tr>
<td>V4</td>
<td>14.9</td>
<td>10.6</td>
<td>6.3</td>
<td>0.3</td>
<td>2.1</td>
<td>3.0</td>
<td>2.5</td>
<td>60.3</td>
</tr>
<tr>
<td>V5</td>
<td>14.9</td>
<td>16.7</td>
<td>6.3</td>
<td>1.3</td>
<td>1.8</td>
<td>3.0</td>
<td>2.5</td>
<td>53.4</td>
</tr>
<tr>
<td>V6</td>
<td>11.9</td>
<td>16.7</td>
<td>3.6</td>
<td>0.3</td>
<td>2.1</td>
<td>2.4</td>
<td>2.5</td>
<td>60.5</td>
</tr>
<tr>
<td>V7</td>
<td>11.9</td>
<td>10.6</td>
<td>3.6</td>
<td>1.3</td>
<td>2.1</td>
<td>3.0</td>
<td>1.9</td>
<td>65.6</td>
</tr>
</tbody>
</table>

Results and Discussion

Aged microstructures

The aging condition of 1093 °C for 400 hr was the baseline aging condition used for modeling composition effects. The microstructures of the alloys fell into three groups based on the TCP distribution. Group 1 alloys contained no TCP phase, Group 2 was comprised of alloys where TCP formed exclusively at the grain boundaries, and Group 3 alloys formed TCP at the grain boundaries as well as within grains. Group 2 alloys could be broken down into two categories: 2a where the TCP formed in a random distribution along grain boundaries and 2b where the TCP tended to form in a cellular fashion. Group 3 was subdivided into three categories based on the form of TCP phase within the grains: 3a (isolated needles), 3b (interconnected needles), and 3c (abundant-random).
Examples of these microstructures can be found in figures 1 to 3 as well as in reference 5. Figure 4 shows how the assigned categories have a general correlation with TCP volume percent, as expected. Figure 4 also indicates that there is certainly some overlap between the subtle group designations, however, categorization is still believed to be useful because a significant distinction does exist between the major groups.

Transmission electron microscopy identified the TCP present as both tetragonal \( \sigma \) and orthorhombic \( P \) phases. Although quantitative measurements were not made in sufficient detail, X-ray diffraction results indicate that \( P \) is the majority phase. Because \( \sigma \) and \( P \) have very closely related crystal structures and compositions, they could not be distinguished by backscattered electron contrast.

![Figure 1](image1.png)

Figure 1.—TCP phase occurrence in (a) Group 2a and (b) Group 3b alloys. TCP is the white phase, \( \gamma \) is light gray, and \( \gamma' \) is dark gray.

![Figure 2](image2.png)

Figure 2.—Microstructural evolution as a function of time and temperature for Group 2b (cellular grain boundary precipitate).
Certain elements or combinations of alloying elements are more potent than others in forming TCP. It is situations such as these where DOE strategies are valuable as they employ multivariable regression models to accurately quantify both the simple linear and complex interactive effects of the alloying elements. The measured volume percent of TCP in all alloy microstructures was the dependent variable while the levels of the individual elements in the alloys (in atomic percent) were the independent variables. The volume percent of TCP was transformed by a square root function to accommodate the fact that TCP response values were more reproducible at lower volume percent than they were at the higher volume fractions near 15 percent. The model would be of the form

\[ \text{TCP volume percent} = f(\text{element levels}) \]
\[(TCP\ phase)^{1/2} = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \ldots
\]
\[
\beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \ldots
\]

where \(X_1\) and \(X_2\) are primary/linear effects and \(X_1 X_2\) and \(X_1 X_3\) are interactive effects of alloying element levels on TCP formation with \(X_1 (1 = 1, \ldots, 7) = Al, Co, Cr, Mo, Re, Ta, W\). Table II summarizes the three models that have been developed in this study and contains the coefficients for all terms found to be significant. Model 1 was presented earlier (ref. 5) to describe the occurrence of TCP in the microstructure of third generation Ni-base superalloys. Since the results of Model 1 were presented, seven new alloys were made to verify Model 1. They are defined as V1 to V7 in table I. Table III shows that Model 1 predicted six out of the seven verification alloys well although it did have trouble with V5. Because of this one poor predictive occurrence, the seven new alloys were grouped with the original 44 to increase the total database of alloys to 51. A second model, denoted Model 2, was generated from this expanded database of 51 alloys and when compared to Model 1, Model 2 still had all of the primary terms included as significant with a few changes in the significance of the interactive terms. As evidenced in table III, Model 2 was able to predict slightly better than Model 1. It may seem striking, however, that the coefficient of W went from +6.46 in Model 1 to -28.55 in Model 2. Again, such a simple comparison is not valid, however, as Model 1 also has W involved in two interactions, Re*W and Ta*W, whereas Model 2 has W involved in only one interaction. Al*W, but also a quadratic term, \(W^2\). In addition, a word of caution must be issued when examining/comparing the coefficients of the simple linear effects of the alloying elements for the purposes of assessing their potency. If an alloying element is also involved in an interaction (e.g., Al*Co), then the linear effects of the variables involved in the interaction are not unique. For example, the effect of Al changes depending upon the level of Co and vice versa. A third model was then generated from the original 44 alloy database to determine a simplified relationship. In this case, a more strict statistical criteria was used than that which was chosen when arriving at Model 1. Specifically, all terms for Model 3 had to be significant at the 95 percent level instead of only at the 90 percent level. Table II shows that Model 3 is much simpler, with all of the primary effects still identified as statistically significant but only one interactive effect, the Co*Re interaction. This simplified model can still describe 91 percent of the data \((R^2 = 0.91)\). Table III indicates that Model 3 predicts the seven verification runs fairly well.

<table>
<thead>
<tr>
<th>Term</th>
<th>Model 1</th>
<th>Model 2</th>
<th>Model 3</th>
<th>Term</th>
<th>Model 1</th>
<th>Model 2</th>
<th>Model 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Co*Re</td>
<td>0.8</td>
<td>0.87</td>
<td>0.68</td>
<td>W*W</td>
<td>1.16</td>
<td>0.37</td>
<td>0.37</td>
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<tr>
<td>Cr*Mo</td>
<td>0.26</td>
<td>0.22</td>
<td></td>
<td>Ta*W</td>
<td>1.82</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cr*Re</td>
<td>0.94</td>
<td></td>
<td></td>
<td>Ta*W</td>
<td>1.82</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mo</td>
<td>0.43</td>
<td></td>
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<tr>
<td>Re</td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ta</td>
<td>-12.58</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>W</td>
<td>5.56</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Al*Co</td>
<td>0.94</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Al*Cr</td>
<td>0.96</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Al*Mo</td>
<td>0.3</td>
<td></td>
<td></td>
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<td></td>
</tr>
<tr>
<td>Al*Co</td>
<td>0.21</td>
<td>0.17</td>
<td></td>
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<td></td>
</tr>
<tr>
<td>Al*Cr</td>
<td>0.21</td>
<td>0.17</td>
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<td></td>
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<tr>
<td>Al*Mo</td>
<td>0.3</td>
<td>0.4</td>
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</tr>
<tr>
<td>BSE</td>
<td>0.3</td>
<td></td>
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</table>

TABLE III.—PREDICTIONS OF TCP VOLUME PERCENT IN VERIFICATION ALLOYS

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Model 1</th>
<th>Model 2</th>
<th>Model 3</th>
<th>Actual</th>
</tr>
</thead>
<tbody>
<tr>
<td>V1</td>
<td>9.5±3.9</td>
<td>7.4±3.2</td>
<td>5.7±3.6</td>
<td>6.7</td>
</tr>
<tr>
<td>V2</td>
<td>11.7±4.3</td>
<td>9.0±3.6</td>
<td>5.5±3.6</td>
<td>9.2</td>
</tr>
<tr>
<td>V3</td>
<td>0.5±0.9</td>
<td>0.7±1.0</td>
<td>0.1±0.5</td>
<td>0.7</td>
</tr>
<tr>
<td>V4</td>
<td>19.2±5.6</td>
<td>19.2±5.2</td>
<td>18.2±5.6</td>
<td>17.9</td>
</tr>
<tr>
<td>V5</td>
<td>38.2±7.9</td>
<td>19.3±5.2</td>
<td>14.7±5.8</td>
<td>14.4</td>
</tr>
<tr>
<td>V6</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>V7</td>
<td>0</td>
<td>0.2±0.5</td>
<td>0</td>
<td>0.3</td>
</tr>
</tbody>
</table>

NASA/TM—1999-209277 5
The predictive capabilities of each of these models changes depending on the predicted value itself. The formula for the ~95 percent confidence interval for a predicted TCP value is TCP ± K(TCP)^{1/2} where K = 1.27, 1.19, and 1.52 for Models 1, 2, and 3 respectively. Model predictions and confidence intervals on those predictions are valid within the ranges of the alloying element studied.

Figure 5 shows an example of how the three models predict TCP volume percent differently for one specific combination of elements, Cr and Al, where the elements not included in the plot were held at their midrange value. Note, however, that all three models show the same general shape of response surface and similar behavior. The response surfaces in this figure illustrate a very strong Al*Cr interaction for Models 1 and 2 where at low Cr, the effect of Al is relatively mild, however, at high Cr the effect is much stronger. It should be noted that the response surfaces are curved because the data was square root transformed. With such steep curves of Models 1 and 2 extrapolation could be problematic.

The three models were also compared to see how well they predicted TCP volume percent in several first, second, and third generation commercial superalloys. The expected amounts of TCP indicated in table IV for each alloy are estimates that were extracted from the literature for each of the alloys after exposure at 1093 °C for 400 hr. It is important to note that all three models were generated from a database of alloys based on René N6 composition so they should only be expected to successfully predict third generation superalloys accurately.

Table IV shows that all three models predicted the third generation alloys correctly. However, Model 1 did have trouble with second generation alloys CMSX-4 and René N5 which is not surprising since those alloy compositions are far outside of the experimental design space. Model 2 predicted the commercial alloys well across all three generations, most likely because of the large number of terms that are fitting the data and because the compositional design space was expanded to accommodate the seven verification alloys. Like Model 1, Model 3 gave a poor prediction for René N4, CMSX-4, and René N5 because their compositions were not close enough to a third generation superalloy composition, thereby making accurate extrapolations difficult. Model 3 does have an advantage, however, because it shows that a simple model can predict third generation alloys accurately. Although Ti is not included in any of the models, it was assumed and treated to behave similar to Ta where applicable.
**TABLE IV.—PREDICTION OF TCP VOLUME PERCENT IN COMMERCIAL ALLOYS**

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Generation</th>
<th>Model 1</th>
<th>Model 2</th>
<th>Model 3</th>
<th>Estimated TCP</th>
</tr>
</thead>
<tbody>
<tr>
<td>CMSX-10</td>
<td>3</td>
<td>0</td>
<td>0.1±0.1</td>
<td>0</td>
<td>-0</td>
</tr>
<tr>
<td>René N6</td>
<td>3</td>
<td>1.8±1.7</td>
<td>1.1±1.2</td>
<td>1.6±1.9</td>
<td>-0</td>
</tr>
<tr>
<td>René N5</td>
<td>2</td>
<td>2.4±0.3</td>
<td>2.9±0.8</td>
<td>0</td>
<td>-0</td>
</tr>
<tr>
<td>René 162</td>
<td>2</td>
<td>2.8±0.6</td>
<td>0.8±1.1</td>
<td>1.5±1.9</td>
<td>0 → 1</td>
</tr>
<tr>
<td>CMSX-4</td>
<td>2</td>
<td>7.6±3.5</td>
<td>0</td>
<td>14.9±5.9</td>
<td>0 → 1</td>
</tr>
<tr>
<td>René N4</td>
<td>1</td>
<td>0.3±0.7</td>
<td>0</td>
<td>67.8±12.5</td>
<td>0</td>
</tr>
</tbody>
</table>

CMSX-4® and CMSX-10® are trademarks of the Cannon Muskegon Corporation.

All three models represented the experimental design space well. The next modeling iteration will be a hybrid of Model 2 and Model 3 in an effort to capture the most attractive features of each. Model 4 will be generated from the 51 alloy database used to build Model 2 with the more strict statistical criteria that was used for Model 3.

**Microstructural Dependence on Time/Temperature Combinations**

Alloys from each group (1, 2a, 2b, 3a, 3b, 3c) were selected for more in-depth analysis of the kinetics of TCP precipitation. The group designations for these alloys were originally assigned based on their microstructures after aging at 1093 °C for 400 hr. Such groupings may not be accurate across all time and temperature combinations but were not changed in order to maintain consistent definitions for the individual alloys. Specimens were exposed at 871, 1093, and 1204 °C for times ranging from 1 to 1000 hr. The alloys that were originally classified as Group 1 remained stable across all time/temperature combinations. Figure 6 indicates that 871 °C induces TCP to form at the grain boundaries after long exposure for Group 2 and Group 3 alloys. Figure 2 shows the microstructural evolution over time of a Group 2b alloy (cellular grain boundary precipitate) at both 1093 °C and 1204 °C. At both temperatures TCP phase precipitation at the grain boundaries occurs after just 1 hr with an accompanying coarsening of γ prime at the boundary. After 1093 °C for 25 hr, the cellular structure has grown significantly to ~10 μm in thickness and after 400 hr continual coarsening is evident. At 1204 °C, similar trends are seen in comparison to 1093 °C although the rate of coarsening is much more rapid. Figure 3 shows the microstructural evolution over time for a Group 3c alloy (abundant/random precipitate) which exhibited the greatest propensity for TCP phase of all of the alloy group types. After only 1 hr at both 1093 and 1204 °C the cellular condition at the grain boundary is apparent. After 25 hr the cellular structure continues to grow and TCP needles start to grow within the grains. After 400 hr the TCP needles are fully connected and broken down to the abundant/random condition for both temperatures.

![Figure 6.—TCP occurs in the grain boundary at 871 °C. (a) Group 2a for 1000 hrs, (b) Group 3b for 1000 hrs.](image-url)
Based on the microstructures resulting from the kinetic experiments given in figures 2 and 3, time-temperature-transformation (TTT) diagrams were generated to show how the TCP phases were precipitating at both the grain boundaries and within the grains. These diagrams were generated for each of the microstructural group types (Groups 1, 2a, 2b, 3a, 3b, 3c). It is important to remember that the propensity for TCP phase in the microstructure increases with increasing group numbers as was shown earlier in figure 4. Figures 7a and b show the TTT diagrams for grain boundary TCP phase precipitation and TCP phase precipitation in the grain interior, respectively. Each curve was constructed from experimental data points which are omitted on the diagrams for simplicity.

Group 1 alloys were stable across all time/temperature combinations and thus are not shown in figure 7. Figure 7a shows that for grain boundary precipitation a greater propensity for TCP in the alloy pushes the knee of the curve to higher temperatures and lower time. This is consistent with the observations in reference 7 where similar trends were seen with increasing Re content. Figure 7b shows that for TCP occurrence in the grain interiors, a greater propensity for TCP in the alloy pushes the knee to shorter times. However, the temperature of the knee did not change as dramatically as what was observed for precipitation at the grain boundaries. Specifically, the temperature of the knee for the Group 2b curve was expected to be lower and align more closely with the Group 3a curve which prompts the need for more experiments to investigate this discrepancy. By comparing the TTT curves for grain boundary precipitation to that for within-grain precipitation, it can be seen that the temperature of the knee for both curves is about the same. The major difference between the two curves is that the grain boundary curve is shifted to much shorter times.

Polycrystalline Behavior as an Approximation for Single Crystal Stability

One potential criticism for this work is that polycrystals are used to study single crystal behavior. However, this is not viewed as a major problem for several reasons. First, based on phase diagram theory, the equilibrium amount of TCP in the microstructure should be independent of the presence of grain boundaries. Second, although the kinetics of precipitation are clearly faster than that for transgranular precipitation, the shapes and positions of the two TTT curves are quite similar for a given alloy. Third, the accuracies of the regression models were equally good whether the alloy was in Group 2 (grain boundary TCP only) or 3 (TCP at the grain boundaries as well as within the grains). Finally, the regression models were not strongly affected by grain boundary TCP because the volume percent of TCP at the grain boundaries was always lower than one percent.
Conclusions

An approach based on a statistical DOE was conducted to empirically describe TCP phase instability as a function of chemical content in advanced Ni-base superalloy René N6. Ni-base superalloy microstructures are highly dependent on chemical composition and even slight variations in starting alloy composition can produce a wide range in the propensity to form TCP phases. Regression modeling is a useful tool for predicting the contribution of individual elements to superalloy instability within a given design space. However, extrapolation may be problematic.

Kinetics experiments explored the conditions where TCP phases precipitated. The time and temperature data gathered showed that TCP precipitation at the grain boundaries always precedes precipitation within the grains and that the knee of the TTT diagrams tends to move to shorter times and higher temperatures in alloys with greater propensity for TCP formation.

References

Investigation of the Formation of Topologically Close Packed Phase Instabilities in Nickel-Base Superalloy René N6

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Topologically close packed (TCP) phase instability in third generation Ni-base superalloys is understood to hinder component performance when used in high-temperature jet engine applications. The detrimental effects on high temperature performance from these brittle phases includes weakening of the Ni-rich matrix through the depletion of potent solid solution strengthening elements. Thirty-four compositional variations of polycrystalline René N6 were defined from a design-of-experiments approach and then cast, homogenized, and finally aged to promote TCP formation. Our prior work reported on the results of the multiple regression modeling of these alloys in order to predict the volume fraction of TCP. This paper will present further regression modeling results on these alloys in order to predict the occurrence of TCP in third generation Ni-base superalloy microstructures. Kinetic results are also discussed.