

Fig. 1—Enlarged Zn corner of the 450 °C isotherm (Fig. 2 from Ref. 1) showing the various phase fields. δ + 5 pct Al lies within [ι + η -(Fe₂Al₅-Zn_x)], while ζ + 5 pct Al can be seen to lie within [ι + η -(Fe₂Al₅-Zn_x)], phase field. oll2

Our objective here is not to engage in discussions that are unrelated to our work. The differential scanning calorimetry (DSC) measurements carried out in our study show clearly that invariant reactions can be uncovered at any stage of thermal evolution of the milled materials. This implies therefore that one can unravel when a particular phase field is traversed. As a consequence, one can generate isotherms based on the analyses of DSC traces of carefully selected milled materials. Based on this principle, one can verify the accuracy of an existing or available isotherm if phase identifications are subsequently carried out after appropriate heat treatments, *etc.*

Concerning the indexing of our X-ray diffraction spectra, Dr. Tang remarks that we did not identify Zn peaks in the high Zn alloys. It is a usual practice among researchers to index only important or relevant peaks in a given spectrum. Therefore, series of spectra could be displayed without indexing all the observed peaks. The reason for his comments on the precision of our measurements is not clear.

We find no need to address the issue concerning FeAl₂ at this stage; this is because we have not generated additional data to warrant such discussion as of now. We will revisit this appropriately in time.

Finally, our assertion that our work can be used to test the accuracy of proposed equilibrium phase diagrams still stands. This is because materials of different compositions lying within the same phase fields will exhibit upon DSC examination similar transformation behaviors, unless they are not located within the same fields at different temperatures. We appreciate his pointing out some typographical errors in our manuscript and, again, regret their occurrence.

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Parametric Analysis of Monocrystalline CMSX-4 Creep and Rupture Data

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Time/temperature parameters have been used for nearly 50 years to display creep and rupture data on a single master plot over a wide range of test conditions. The approach may be used to achieve the following major design objectives.^[1]

- (1) It allows the representation of creep rupture (or creep) data in a compact form, allowing interpolation of results that are not experimentally determined.
- (2) It provides a simple basis for comparison and ranking of different alloys.
- (3) Extrapolation to time ranges beyond those normally reached is straightforward.

There are a number of methods available for optimizing these parametric approaches,^[2,3] although the standard Larson–Miller (LM) parameter^[4] is by far the most widely used. The original suggestion of a universal constant of 20 in the LM parameter in the form $T(20 + \log t)$, where *T* is the temperature in Kelvin and *t* the time in hours, is also widely employed, especially for comparing different alloys.

The fundamental requirement of all time/temperature parameters is that the value of the parameter at a given stress is independent of temperature and time. The constant in the parameter, *e.g.*, the value of 20 in the LM parameter, should also be independent of stress if the parameter is to be used for comparison and simple extrapolation.

This article describes an analysis of creep rupture data for fully heat-treated (solutioned and aged) CMSX-4 nickel-base monocrystalline superalloy. This alloy is a rhenium-containing second generation monocrystalline alloy used primarily in combustion turbine hot section blades. The analysis is performed in terms of the LM parameter, although similar conclusions might be expected for other parameters. The data were taken at five different stresses and a variety of temperatures, so isothermal curves over a range of stress could not be readily constructed without some parametric analysis. However, the isostress data provided an excellent basis for assessing the applicability of a unique parametric representation. This is because many of the common parameters require linear isostress lines on a plot of log time *vs T* or 1/T.^[5]

Figure 1 shows the stress vs LM parameter curves for time to 0.5 pct creep and rupture. The curves are fitted with a second- and third-order polynomial, respectively. Considerable scatter about these curves is apparent in the figure,

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Fig. 1—Stress vs LM parameter (T in K, t in h) for times to 0.5 pct creep and rupture.



Fig. 2—Reciprocal temperature (T in K) vs log time (h) to 0.5 pct creep.

especially at the lower stresses, so it was necessary to analyze the data to achieve the optimum value of the constant in the parameter. It should be noted, however, that one pair of duplicate tests at 1116C and 86.1 MPa failed in 184 and 1115 hours, indicating unusually high variability under identical conditions at low stresses and high temperatures. There are thus two issues here: repeatability of test measurements under identical conditions in monocrystalline materials, in which crystal rotation and activation of new slip systems are expected to contribute to variability; and precision of time/temperature parameters for tests at different temperatures.

Figures 2 and 3 are plots of isostress lines for 0.5 pct creep and rupture, respectively. They are reasonably linear on reciprocal temperature vs log time coordinates, but do not converge to a common value on the log *t*-axis, as is required for a unique constant in the LM parameter.^[5] The equation for each line was solved to provide the optimum value of the LM constant for each stress. For example, the value of the parameter, *P*, for time to 0.5 pct creep is given by

$$P = T (C + \log t) \text{ or}$$
$$\frac{1}{T} = \frac{C}{P} + \frac{\log t}{P}$$

At 137.8 MPa, the equation for the straight line in Figure 2 is

$$\frac{1}{T} = 0.000694 + 0.0000291 \times \log t$$



Fig. 3—Reciprocal temperature (T in K) vs log time (h) for rupture for different stresses.



Fig. 4-LM constant vs stress for 0.5 pct creep and rupture.

Comparing the two equations,

$$\frac{C}{P} = 0.000694$$
 $\frac{1}{P} = 0.0000293$

Therefore, C = 23.85.

Optimum values for C were calculated for all the isostress lines. It was found that the optimum value was stress dependent. Figure 4 shows that the value of the "constant" as a function of the stress for 0.5 pct creep ranged from 19.3 to 41.2 and varied from 13.7 to 31.0 as a function of stress for rupture.

The analysis shows that, although for the range of analyzed data a parameter can be calculated that gives a value independent of temperature and time at a fixed stress, the optimized value of the constant in the parameter is strongly stress dependent. For stresses above about 150 MPa, use of a value of 20 may be reasonable for the 0.5 pct creep data and less so for rupture, but at lower stresses, it is clearly inappropriate for both sets of data. Moreover, the greatest stress dependence of the parametric constant occurs at low stresses, where the interest in extrapolation may be greatest. However, it should be noted that these low stress data were also taken at the highest temperatures, which may be well above normal operating conditions. The effectiveness of the parameter for data extrapolation at low and intermediate stresses and intermediate temperatures cannot be determined from these data.

To show that much of the scatter in the parametric representation of creep and rupture shown in Figure 1 may



Fig. 5—Stress vs parameter with a stress sensitive constant for 0.5 pct creep and rupture.



Fig. 6-Stress vs temperature for 0.5 pct creep and rupture in 1000 h.

indeed be eliminated, the optimized stress sensitive parameters are used in Figure 5. Here, the constant is incorporated as a function of stress, which significantly reduces the range of values at each stress. However, this plot requires a unique stress dependency and is awkward to apply because the value of the constant actually goes through a minimum with decreasing stress. This leads to the odd curve shape. It also leads to lower parametric values for rupture than for 0.5 pct creep because of the lower values for the optimized constant. Although this analysis eliminates much of the scatter that is due to failure of the parameter, it cannot be used effectively as a basis for prediction at low stresses where the value of the optimized constant has to be interpolated and is so sensitive to stress. Also, the specimen-tospecimen variation for duplicate tests remains as a primary source of scatter at the lowest stress.

An open-ended parametric form based on a graphical optimization procedure^[6] could be used for this alloy. However, this also lacks generality. Perhaps the best solution is to develop individually optimized parametric analyses for every alloy and then compare all alloys on unique curves such as creep strength vs temperature constructed from the optimized parametric curves. An example for the CMSX-4 is given in Figure 6, which shows the stress vs temperature curves for 0.5 pct creep and rupture in 1000 hours. The temperatures were calculated from the linear equations fitted to the data in Figures 2 and 3 and are thus optimized using all the test data. Little or no extrapolation is involved to 1000 hours for this data set. Similar plots for longer times may be obtained by extrapolating the straight lines in Figures 2 and 3. Figure 6 clearly provides a much more accurate basis for comparison of creep and rupture with other alloys, that have been individually optimized, than does plotting against a universal time/temperature parameter.

The following conclusions may be drawn from this analysis.

- (1) The value of 20 in the LM parameter is inappropriate for this alloy except for time to 0.5 pct creep over a restricted stress and temperature range.
- (2) The optimized constant in the parameter for creep and rupture is stress dependent.
- (3) A stress-dependent constant in the LM parameter, or any common parameter, restricts the broad applicability for comparison and prediction.
- (4) A simple plot of the basic variables may be produced from separately optimized parametric correlations for different alloys.

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